PLATSOL^{TM} TREATMENT OF THE NORTHMET COPPER-NICKEL - PGM BULK CONCENTRATE - PILOT PLANT RESULTS

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ABSTRACT

PolyMet Mining is developing the NorthMet property near Duluth, Minnesota. Laboratory investigations had led to the development of the PLATSOL^{TM} process to dissolve in one step the base metals (copper and nickel) as well as the gold and PGM's. To complete the pre-feasibility study, an integrated ten-day leach pilot plant trial was undertaken, comprising of: high temperature (>220ºC) pressure oxidation of the bulk concentrate in the presence of 5-10 g/L chloride (the PLATSOL^{TM} process), followed by solid/liquid separation, gold plus PGM's recovery from solution, conventional Cu SX/EW and recycling of the copper raffinate to the autoclave. The concentrate tested was a NorthMet bulk flotation concentrate assaying 14.7% Cu, 3.05% Ni, 0.14% Co, 26.7% S, 1.4 g/t Au, 2.2 g/t Pt and 9.9 g/t Pd. Laboratory results were fully confirmed and, with minor adjustments of the laboratory PLATSOL^{TM} conditions, the following leach extractions were obtained during the pilot plant trial: Cu 99.6%, Ni 98.9%, Co 96%, Pd 94.6%, Pt 96% and Au 89.4%.

The autoclave pulp was filtered hot, and the gold plus PGM's recovered from the acidic pulp using NaHS sulphide precipitation. After partial neutralisation to pH = 2.0 and gypsum removal, the PLS was treated using standard Cu SX/EW, and good quality cathodes were produced at high current efficiency.

The treatment of the bleed solution for nickel and cobalt recovery was also demonstrated during a five-day pilot plant. The bleed was first purified from iron, then decopperized using NaHS. The purified bleed was then processed through cobalt/zinc Cyanex 272 bulk extraction, followed by selective stripping of cobalt and zinc from the loaded organic; the Cyanex 272 raffinate was further treated with Versatic 10 acid to recover the nickel, and the strip liquor electrowon to produce 99.9% Ni cathodes at 95% current efficiency.

BRIEF HISTORY OF A THIRTY YEAR PROJECT

The Cu-Ni Pt-Pd deposits located in the Duluth Complex in Minnesota have been known for at least three decades. The Duluth complex is a large series of mafic to felsic intrusive bodies that host these often large, low-grade, disseminated polymetallic deposits, some of which are enriched with platinum group metals (PGMs). These deposits represent a unique class of orebody, magmatic sulphide deposits, which are the principal concentrations of group VIII transition metals - iron (Fe), cobalt (Co), nickel (Ni), palladium (Pd), platinum (Pt), rhodium (Rh), ruthenium (Ru), iridium (Ir), and osmium (Os).

The two larger and better-known of these deposits – NorthMet (formerly called Dunka Road) and Babbitt (subsequently the Minnamax Project) – were discovered in the late 1960's by US Steel (USX) and Kennecott Copper Corporation (Bear Creek Mining Company) respectively. USX drilled 112 diamond drill holes into the NorthMet deposit along a geophysical grid on 183-meter centres. The Babbitt or Minnamax Project, as it became known in 1974 when AMAX became partners with Kennecott, was extensively drilled to a "reserve" status in the late 1970's. In addition, a 12-ft diameter shaft was sunk to the 1700-ft level in a higher-grade portion of the deposit for the purposes of determining continuity of mineralisation by drifting through the mineralisation, coupled by detailed underground fan drilling, and the collection of a bulk sample for metallurgical sampling.

In both cases, the enormous magnitude of the mineralised resource led property owners to think in terms of large, underground mining operations at

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appropriately high cut-off grades, with the sale of concentrates to a U.S. or Canadian smelter for processing. Unfortunately,neither USX nor AMAX were able to produce satisfactory concentrate for sale. A bulk concentrate was too low in grade to be marketable, and clean, high-quality copper and nickel concentrates could not be produced consistently.

Although alternative metallurgical processes were tested throughout the intervening years, none was found that made these projects economically viable. In 1976, USX performed a few analyses for PGMs, but the project remained uneconomic due to low metals prices and poor metallurgical recoveries. The Minnamax Project was abandoned in 1982 for essentially the same reasons. Unfortunately, sporadic and poorly defined metallurgical testing of various types occurred well into the 1990's with very limited success. Thus these polymetallic deposits continued to suffer from a reputation of being too complex metallurgically to ever be economic.

In 1987 the Minnesota Natural Resources Research Institute published PGM data collected from a number of the Duluth Complex deposits and noted the possibility for a large resource of PGMs along the base of the Complex. Occasional metallurgical testing to recover PGM values in subsequent years by various leaseholders proved largely unsuccessful in solving the metals recovery problems that surrounded these deposits in the Duluth Complex.

In 1989, Fleck Resources (now PolyMet Mining Corporation) leased the NorthMet Project from USX. In July 1990, Nerco Exploration Co. leased the property from PolyMet with an option to earn a 60% interest in NorthMet by spending a minimum of US$1.1 million within four years and completing a feasibility study prior to the end of the fourth year. Nerco contracted Fluor Daniel Wright to undertake a technical and economic evaluation of the property based on data from 102 drill holes and 4,200 assayed samples. Fluor determined a new open pittable resource of 808 Mt averaging 0.432% Cu, 0.109% Ni, 0.006% Co, 0.061 g/t Au, 1.5 g/t Ag, - /437 g/t Pd and -.16 g/t Pt, contained within a mineral resource of 1,450 Mt at similar metal grades.

Nerco Minerals undertook a comprehensive metallurgical program and more particularly examined the viability of applying the Cuprex process to Dunka Road bulk concentrates. Detailed results have been published (1). Base metals recovery was certainly satisfactory, but the PGM's were left in a very low grade residue, with little hope of an economic recovery process.

Financial difficulties forced Nerco to allow their option to lapse and three years later, Argosy Mining Corp. leased the property from PolyMet, contracting with the NRRI to conduct GPS controlled geological mapping and continued metallurgical investigations. Argosy's lease expired on the NorthMet project in 1996.

In 1997-98, PolyMet's management came to believe that technological advances made during the last 6-8 years in the general areas of hydrometallurgy offered the opportunity to solve previous metallurgical recovery problems associated with these types of deposits and thus make its NorthMet deposit economic. The Company placed into action a plan to test these beliefs and to provide a solution to previous metallurgical recovery problems. Constraints placed on the testing program by PolyMet's management resulted from its desires not to attempt to permit a smelter in Northern Minnesota, not to sell concentrate to a toll smelter, and to produce metals on site to the maximum extent possible.

The metallurgical test program again examined the feasibility of producing clean copper and nickel concentrates, but unsuccessfully. On the hydrometallurgical side, PolyMet launched an extensive metallurgical test program to examine the potential for pressure oxidation (POX) and bacterial oxidation (BIOX®) to solve the project technical challenges. Various organisations were approached, such as Process Research Associates (Vancouver), Dynatec (Fort Saskatchewan) and SGS Lakefield (Ontario) for the pressure oxidation test programs, and Research Productivity Council (New Brunswick) and Bactech (Australia) for the bacterial oxidation test programs.

Results of these various investigations have been presented (2). The overall conclusion was again that several alternatives would recover the base metals, but the PGM's recovery remained a challenge.

In 1999, the PLATSOL™ process, a high temperature pressure oxidation process was developed at SGS Lakefield by International PGM Technologies Ltd. to treat NorthMet bulk copper-nickel-PGM concentrate. Several papers have been published describing the development and application of the PLATSOL™ to the NorthMet bulk concentrate and various other feedstocks (3, 4, 5). Suffice to say here that the PLATSOL™ process allows one step dissolution of base and precious metals. Its success depends on the addition of a small amount of chloride into a high temperature (>200°C) pressure vessel. Typical results for the NorthMet bulk concentrate are presented in Figure 1.

At that stage, all metallurgical testing had been conducted at the bench, and pilot testwork was required to confirm the process in a continuous operation, with all recycle solutions.

This paper describes the results of a mini pilot plant demonstration of the application of the PLATSOL™ process to treat NorthMet bulk concentrate.

PILOT PLANT DEMONSTRATION

Based on extensive bench scale testwork and previous experience, the recommended flowsheet for the main circuit of the NorthMet concentrate treatment plant is illustrated in Figure 2.

This is the process that was used during the pilot plant. The main circuit (Figure 2) was tested in July 2000, while the bleed treatment (that will be discussed later) was piloted in November-December of the same year. Some pilot plant results have already been published (5).

Each unit process of the pilot plant will be discussed in sequence.

DESCRIPTION OF FEED SAMPLES

The NorthMet concentrates used during the hydrometallurgical pilot plant trial were produced during two flotation pilot plants conducted at SGS Lakefield in 1999 and 2000. Assays of the two concentrates are presented in Table I. Table 2 includes the chemical composition of the Michigan limestone used throughout the pilot plant test.

DESCRIPTION OF PILOT PLANT EQUIPMENT AND RESULTS

Overview

It is well known that high temperature pressure oxidation (T ³200ºC) could dissolve copper and nickel. The fundamental difference between the PLATSOL™ process and conventional high temperature pressure oxidation is the addition of chloride ions to the autoclave feed. It was discovered that the addition of chloride at concentrations as low as ~3 g/L (5 g/L NaCl) resulted in the dissolution of most of the PGM's in the autoclave together with copper and nickel. The PGM's were easily recovered by NaHS precipitation while copper and nickel could be recovered using conventional techniques.

Typical laboratory results with the year 1999 concentrate are presented in Figure 1. The overall process, as developed during the bench scale program, is illustrated in Figure 2.

Grinding

The concentrates were ground to a P_{80} of 15 micrometers using a ceramic ball mill. The limestone was ground to a P_{80} of 12 micrometers.

PRESSURE OXIDATION - THE PLATSOL™ PROCESS

Description of the Autoclave

Lakefield's continuous autoclave, constructed of Grade 12 Titanium, is 172.7 cm in length with an inside diameter of 25.0 cm, and is divided into six compartments by means of weir plates. Each weir has an upcomer assembly to prevent short – circuit overflow of slurry.

The autoclave is heated by means of externally wrapped ceramic mat heaters controlled by a centralised power supply. Thermocouples mounted on the skin of the autoclave shell provide partial feedback information to the Bailey DCS. The power supply also interacts with the Bailey DCS. The Bailey DCS receives input signals from the RTD thermocouples mounted in thermowells, which extend into the slurry zone of each autoclave compartment.

The operating temperature is principally attained through the exothermic reaction between the sulphides and oxygen. Supplementary heat is provided from the heaters. The exothermic heat generated from the reaction is controlled by dilution of the feed with water, thereby circumventing the need for any additional cooling water.

Each compartment has an independent drive mechanism, with variable speed control to the impeller shaft.

Off-gas is chilled through a water-cooled condenser to drop out any steam. The noncondensable gases such as H_2S are captured in a NaOH scrubber system. Slurry from the autoclave feed tank (at 11% solids) flowed by gravity (tank bottom discharge) into a pump that directed the flow into the first compartment of the autoclave.

Feed slurry flow to the autoclave was monitored by a magnetic flow meter. Measurements were logged every minute in the historical database of the Bailey DCS. Slurry flow was controlled at the desired set point by a PID block within the DCS.

Oxygen gas was sparged into all compartments. The oxygen was distributed with greater than 80% of the total flow directed into the first two compartments.

A photograph showing the continuous autoclave is presented in Figure 3.

Pressure oxidation preliminary results

A series of short pilot plant runs were initially conducted to examine the effects of the main variables and confirm laboratory results under full recycle conditions. Temperature was varied between 220 and 225ºC and retention time between 120 and 180 minutes, with salt NaCl addition maintained at 6 g/L Cl.

Copper extractions ranged between 98.8 and 99.3% and nickel extractions ranged from 97.5 to 98.9%. However, best palladium extractions were only 86%, 10% lower than bench extractions under similar conditions with limited recycle.

A few batch autoclaves were then conducted with full recycle; the results pointed towards a significant deleterious effect of Mg (5 g/L) and Al (2.7 g/L) in the recycle stream. The bleed ratio was modified to bring Mg and Al levels down to 1.8 and 1.2 g/L respectively. The following pilot plant short run (PP-3) under these modified conditions produced the following extractions: Cu-99.6%, Ni-98.6%, Co-98%, Pt-96.1% and Pd-92-94%. These conditions were selected for the long run.

Pressure oxidation long run results
An integrated continuous pilot plant (Figure 2) was conducted, starting with short run PP-3 conditions, and modified as needed during the run. Temperature was 225°C; pulp density was 11%; retention time was 120 minutes and oxygen overpressure was 100 psi (689 kPa). Only the 2000 bulk concentrate (reground at a P_{80} of 15 micrometer) was used during the long run.

Mechanically, the autoclave performed well at 96% availability; minor problems were experienced with oxygen lines plugging.

From a chemistry point of view, problems were encountered during the central part of the run, after Cu SX raffinate was recycled, when the concentrate fed to the autoclave was extremely flocculated, making control of the pulp density inside the autoclave difficult. Figure 4 shows that the concentrate formed a thick persistent foam difficult to break mechanically and floating at the surface of the liquor. It is believed the flocculation of the concentrate resulted from interactions between residual flotation reagents and entrained organic in the recycle. Once identified, the situation was rectified and the last 72 hours of the long run were very smooth.

Results of the first and last days of the long run are presented in Table 3. With deflocculation of the feed to the autoclave, results were as expected for copper, cobalt, platinum and palladium. Ni extractions were 3-4% higher than in the laboratory.

Gold extraction was lower than in the laboratory due to the significant lower gold feed grade in the pilot plant run as compared to the batch testwork (1.4 g/t vs. 2.2 g/t).

Periodic samples were removed from each of the compartments to examine process kinetics. Typical results are presented in Figure 5 and 6.

Results indicated a fast oxidation of the sulphides of Cu and Ni and a slower attack of the PGM elements. Analyses of the autoclave discharges during the last days are presented in Figure 7 and Figure 8 for Pd, Fe and Cu, Ni, respectively.

TREATMENT OF THE PLATSOL™ PROCESS LEACH LIQUOR TO RECOVER BASE AND PRECIOUS METALS

A typical pregnant liquor composition is presented in Table 4.

Recovery of the Gold and PGM's
Bench scale testwork had indicated that gold and the PGM's could be easily recovered from the PLATSOL™ liquor using NaHS.

Laboratory results had indicated that, without seed, one hour was sufficient to precipitate all the gold, platinum and palladium from the acidic PLS. This process is considered to be well known. Bench results also indicated that gold and PGM's could be loaded on activated carbon and stripped using the conventional Zadra method. This process, however was considered to require further testing and it was decided to test it thoroughly during the pilot plant. Therefore, NaHS precipitation was briefly tested at the start of the campaign, and activated carbon for most of the program. Bench scale NaHS treatment of the pilot plant liquor produced the results indicated in the following Table 5.

With low NaHS addition (Condition B, 0.25 g/L NaHS), the precipitate grade was high (~6200 g/t Au + Pt + Pd), the copper low (3.4% Cu) but some of the Pt did not precipitate. With higher NaHS addition (Condition A, 0.5 g/L NaHS), all the PGM's precipitated, but also too much Cu (46.7% Cu), and therefore the overall precipitate was low grade (~700 g/t Au + Pt + Pd). Clearly, the optimum condition lies between A and B. Initial pilot plant results are illustrated in Figure 9.

In 20 minutes, all gold and palladium were precipitated, but only half of platinum.

Rather than optimise the process, as mentioned earlier, the circuit was modified and activated carbon used to recover PGM throughout the remainder of the trial.

Activated carbon was tested in pulp (CIP) and in column (CIC). To summarise the overall results, it can be said that the loading step on fresh carbon was acceptable, but it was not possible to regenerate it properly to the same performance as fresh carbon, and as a consequence, activated carbon was deemed unsuitable for that service and abandoned for the present time.

Recovery of copper by conventional SX/EW
After PGM's recovery, the liquor was neutralised to pH @2.0 in a 4-tank cascading reactor using Michigan limestone. This local limestone (P_{80} = 12 μm) displayed an excellent reactivity; the pH was progressively raised through the cascading reactors, and easily filtrable gypsum was produced (Figure 10).

After proper filtration and washing, the gypsum cake assayed 0.004-0.04% Cu and 0.002-0.036% Ni.

A small Cu SX/EW pilot plant was operated in line with the PGM recovery and PLS neutralisation.

The solvent extraction circuit operated at 40°C with a 30% (vol) LIX 984 in Orform SX-7 diluent. It included 2 extraction stages, 1 scrubbing stage and 2 stripping stages. The SX circuit was operated in series with 2 electrowinning cells operating at 40°C and 290 A/m2, each with 3 stainless steel cathodes and 4 Pb-Ca-Sn anodes.

Some crud was observed in the extraction stage, but did not cause operational problems. Phase separation was rapid and clean, despite the high concentration of extractant used. Organic losses in the raffinate were consistently below 20 mg/L TOC after a few days of operation. This level was easily lowered to less than 5 mg/L TOC using activated carbon. The O/A ratio during extraction varied between 1.4 and 2.0 to account for variations in the feed copper grade and to generate the target 0.5 g/L Cu in raffinate. Figure 11 shows the pilot plant solvent extraction circuit.

A photograph of the Cu EW circuit is presented in Figure 12.

Current efficiency was excellent (98-99%) and cathode quality was good (Table 6).

Bleed Purification

About 25% of the copper solvent extraction circuit raffinate was bled off for nickel and cobalt recovery. The average composition of the bleed stream is presented in Table 7.

There are several alternatives to process such solution for nickel and cobalt recovery, as examplified by the numerous options commercially used or proposed for laterite liquors. Options considered and thoroughly tested for NorthMet bleed stream include intermediate precipitation (hydroxides or carbonates) and various all solvent extraction options.

The final choice for the NorthMet project is illustrated in Figure 13.

Prior to cobalt-zinc solvent extraction using Cyanex 272, copper, iron, aluminium and acid had to be removed. This was done in sequence, as illustrated in Figure 14.

Neutralisation - Iron Oxidation/ Precipitation

The bleed stream was partially neutralised to about pH = 2.0 using local limestone (Michigan limestone); the iron was then oxidised using SO2/O2 mixtures, and precipitated in two stages, as indicated in Figure 15.

Such a process intends to provide a purified solution where all iron and aluminium have been removed, while producing a gypsum iron residue with minimum valuable metals losses.

The process was piloted during 72 hours of continuous operation, and bench scale results were fully confirmed:
the partially purified solution contained less than 10 mg/L Fe and less than 5 mg/L Al.

- retention time in the whole circuit was about 5 hours, including neutralisation
- the gypsum-iron residue normally assayed <0.1% Ni, Co

Decopperization

Copper was removed by sulphide precipitation using NaHS. Details of the process are presented in Figure 16.

No surprises were found. Copper was easily precipitated in 20 minutes to a residual less than 2 mg/L Cu into a precipitate assaying about 40%Cu. Some Ni co-precipitated with the Cu, but this was of minor concern since the precipitate was to be recycled to the autoclave.

Under the conditions tested, no zinc was removed with the copper.

Bleed Treatment for the Recovery of Ni + Co

After purification, the bleed solution had a typical composition as shown in Table 8.
The process used to recover cobalt and nickel consisted in the following steps:

- bulk extraction of zinc and cobalt, using Cyanex 272
- selective stripping of cobalt from loaded Cyanex using pH = 4.0, followed by selective stripping of zinc from loaded Cyanex, using pH 2.0
- extraction of nickel from Cyanex circuit raffinate using Versatic 10 extraction at a pH ~6.8
- electrowinning of nickel from sulphate-rich electrolyte in a bagged anode cell

The process tested is presented in Figure 17. A photograph of the nickel cobalt solvent extraction pilot set up is presented in Figure 18.

The circuit was operated for a total of about 136 hours; final treatment of the cobalt and zinc strip liquors was batch.

Zinc-Cobalt Bulk Extraction using Cyanex 272

The main parameters in this stage were as follows:

- Extractant – Cyanex 272 10% (v/v) with 5% TBP modifier and 85% (v/v) Orform SX7.
- Temperature – 55°C
- O/A ratio – 1/1
- Extraction mixing time – 3 minutes
- pH – controlled by NaOH addition between 5.2 and 5.5
- Number of extraction stages – 4
- Scrubbing stage – 1 stage at pH 5 and 50°C

Pilot plant results are presented graphically in Figures 19 and 20.
Initial results indicated some high cobalt and zinc values in the raffinate. These were due to difficult pH control in the extraction. After modification of the strength of the caustic solution used to control the pH and modification of the addition point, control was much better (with one single exception). In general, raffinates with less than 2 mg/L Co and Zn can be expected.

Some crud was observed in the extraction circuit.

Zinc and Cobalt Selective Stripping

The main parameters in this stage were as follows:

- Cobalt stripping – pH = 4.0, 40oC
- Co strip number of stages – 3 stages
- Co stripping O/A – 20/1
- Zn stripping – 40oC, pH 2.0
- Zn strip number of stages – 2 stages
- Zn stripping O/A – 20/1

Results are illustrated in Figure 21 and Figure 22 for the cobalt strip, and Figure 23 and Figure 24 for the zinc strip.
Results indicated that at the end of the run, the cobalt strip liquor had stabilised at about 7.5 g/L Co, 7.5 g/L Mg, 0.5 g/L Mn and <20 mg/L Zn. Since the method to recover the cobalt had been selected as sulphide precipitation, the presence of large quantities of magnesium and manganese was not deemed to be of major concern.

The zinc strip liquor at the end of the trial averaged about 50 g/L Zn; magnesium was low (<20 mg/L in general); nickel was present at around 50 mg/L.

Unfortunately, there was a significant contamination by cobalt (an average of around 400 mg/L), which would constitute a loss of cobalt values and a penalty for the zinc concentrate.

We believe this cobalt contamination could be significantly reduced, if not eliminated, by a slight modification of the stripping process. The stripping process used during the pilot plant is illustrated in Figure 25.

Nickel Extraction using Versatic 10

Nickel extraction from sulphate solutions using Versatic 10 is commercially applied in Bulong.

The main parameters of this stage were as follows:

- Extractant – Versatic 10, 20% (v/v), Orform SX7 80% (v/v)
- Temperature – 50°C
- O/A ratio – 1/1
- Extraction mixing time – 3 minutes
- pH – controlled with NaOH addition between 6.6 and 6.8
- Extraction stages – 4
- Scrubbing stage – 1 at pH 7.0, 40°C
- Versatic acid recovery from raffinate – 1 stage at pH = 2, 35°C, O/A = 1/1

Results are presented in Figure 26 and Figure 27.

At the end of the trial period, nickel extraction was excellent, with less than 5 mg/L Ni in the final raffinate. The raffinate also contained about 1.5 g/L Mg.

The Versatic 10 raffinate contained between 1 and 3 g/L of total organic carbon (TOC), indicating the high solubility of carboxylic acids at high pH's. After the Versatic 10 recovery stage at a pH = 2, the TOC was reduced to around 20 mg/L.

Similarly to what has happened in the commercial plant at Bulong, a significant precipitate had formed in the Versatic 10 circuit, most particularly in the third extraction stage. X-ray diffraction confirmed the precipitate to be gypsum. The formation of gypsum has to be reckoned with, and allowance has to be made for physical removal of gypsum (pumping out and filtering) and/or dilution of the nickel circuit feed with fresh water to lower the calcium content below its saturation point, with important implications on the plant water balance.

Nickel Stripping and Electrowinning

The main parameters for the nickel stripping were as follows:

- Temperature – 35°C
- O/A ratio – 2/1
- pH – from <1 to 3-3.5 (no external control)
- Number of stages – 3 stages

The main parameters for the nickel electrowinning were as follows:

- Anodes – Pb/Ca/Sn anodes, in a bag
- Cathode – SS, sanded, no bag
- Current density – 200-240 A/m^2
- Number of anodes – 2
- Number of cathodes – 1
- Anode, cathode unit area ~1500 cm^2/side
- Catholyte pH – 3-3.5 (no control)
- Electrolyte composition – 5 g/L H_3BO_4, 120 g/L Na_2SO_4

Results are presented graphically in Figure 28 through Figure 30.

Samples of the final cathode were submitted for assays. Results are presented in Table 9.

Cathode quality was good (>99.9% Ni), with the main impurities being cobalt, iron, lead, copper and zinc.

We believe that these impurities could be reduced significantly, as indicated below:

- high cobalt and zinc resulted mostly from the problems encountered with pH control during the Cyanex 272 extraction stage. These should decrease significantly with a better pH control. Moreover, as a safety measure, it is suggested to add one ion exchange stage in the electrolyte circuit to remove cobalt and zinc (and Cu), in case of operation spikes.
- high lead: freshly prepared anodes were used during the pilot plant, and this always results in accelerated dissolution of lead, and subsequent contamination of the cathode, until a passive layer of PbO_2 is formed at the surface of the anode.

Precipitation of Cobalt Sulphide

A sample of the cobalt strip liquor was batch treated to assess the feasibility of producing a marketable cobalt concentrate. The solution sample was contacted at 80°C with a 20% NaHS solution while the pH was maintained at 6.5 using a 30% NaOH solution. Results are presented in Table 10.

Under the conditions used, for all practical purposes, all cobalt, nickel and zinc in the strip liquor were precipitated in a bulk concentrate assaying ~30% Co, 2.6% Ni and 0.4% Zn.

Precipitation of Zinc from the Zinc Strip Liquor

The amount of zinc present in the NorthMet deposit is not sufficient at the planned throughput to warrant its recovery as zinc metal. Rather its removal was more important from an environmental point of view than as a revenue generating product. The sale of a zinc product was to cover its removal costs.

Several methods are available to recover zinc from the zinc strip liquor. The method selected at this stage was carbonate precipitation. A sample of zinc strip liquor was first neutralised with limestone, then contacted at 80°C with a 25% Na_2CO_3 solution to pH = 4.0. The zinc concentrate was then filtered off, and the solution treated further with carbonate to remove the remaining zinc for final discharge of the liquor, producing a lower grade concentrate for recycling at the first stage. Results are summarised in Table 11 below.

The moisture of the zinc concentrate being quite high, the product will have to be dried, at least partially.

ENVIRONMENTAL ASPECTS

As in any other mining project, and maybe even more so in Minnesota, the environmental impacts of a process must be very thoroughly examined. This important aspect of the project is presently on-going and is as yet not fully finalised.

A few key points can already be stressed, however. From the start, it had been decided that:

- All the hydrometallurgical plant solid residues would be kept separate from the flotation residues, which have been assessed to be non acid-generating
- The three solid residues from the hydrometallurgical process, namely the PLATSOL^{TM} leach residue, the gypsum

residue and the gypsum iron residue would be mixed and discharged to a separate lined pond, with enough limestone to neutralise all the non-gypsum sulphate in the blended residue.
- The plant would be a zero-discharge to the environment, with all the liquid effluents treated and recycled as required.

CONCLUSION

A ten-day integrated pilot plant was conducted on the year 2000 NorthMet bulk concentrate, with the main objective being to confirm the applicability of the PLATSOL™ process under full solution recycle.

With minor modifications, the process as developed in the laboratory was fully confirmed; the pilot plant results indicated the need for controlling the build-up of impurities (Al, Mg) into the raffinate recycle and ensure that the concentrate is well dispersed before being fed to the autoclave. Under those conditions, excellent leach recoveries were produced, such as: 99.6% for Cu, 98.9% for Ni, 96% for Co, 94.6% for Pd, 96% for Pt, and 89.4% for Au.

NaHS precipitation was used to recover Au + PGM's from the acidic liquor. Without seed recycle, one hour retention time was required to recover all precious metals. The sulphide concentrate will likely be upgraded prior to sale by dissolving the copper sulphide.

Copper recovery from partially neutralised solution using conventional SX/EW was confirmed to be problem-free; high current efficiency and good cathode quality were indicated. Based on the pilot plant results, the process flowsheet to treat the NorthMet concentrate has been finalised as illustrated in Figure 31.

The process developed to recover the nickel and the cobalt from the NorthMet bulk concentrate was also piloted. The bleed stream was easily purified using NaHS precipitation for copper and an unusual but efficient process to remove the iron. Zinc and cobalt were extracted using Cyanex 272, and then selectively stripped into separate streams suitable for the production of high grade cobalt sulphide and zinc carbonate concentrates.

Good quality nickel cathode (99.9% Ni) was produced at high current efficiency (95%). The pilot plant was conducted for a total of 14 days for the main circuit (PLATSOL™ and Cu recovery) and 5 days for the Ni/Co recovery circuit. Although longer duration would further increase one's confidence, it was long enough to confirm the main chemistry of the process, and also pointed out the (few) areas where modifications are required.

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REFERENCES

1. C. J. Ferron, R. G. Williamson, A. D. Zunkel, "Metal Recovery from a Complex Sulphide Concentrate Using a Ferric Chloride Leach Process," EPD Congress, TMS Annual Meeting, Anaheim, California, February 4-6, 1996.
2. P. T. O'Kane, C. L. Brierley, D. B. Dreisinger, D. W. Gentry, "Hydrometallurgical Process Development at Polymet Mining Corporation's Northmet Project," CIM 2000, Toronto.
3. C. A. Fleming, C. J. Ferron, D. B. Dreisinger, P. T. O'Kane, "A Process for the Simultaneous Leaching and Recovery of Gold, Platinum Group Metals and Base Metals from Ores and Concentrates," TMS - EPD Congress, 2000. March 12-16, Nashville Tennessee.
4. C. J. Ferron, C. A. Fleming, D. B. Dreisinger, P. T. O'Kane, 2000, "Single-step Pressure Leaching of Base and Precious Metals (Gold and PGM's) using the PLATSOL™ Process," ALTA 2000 Nickel/Cobalt Conference.
5. C. J. Ferron, C. A. Fleming, D. B. Dreisinger, P.T. O'Kane, "Pilot Plant demonstration of the PLATSOL™ process for the treatment of the Northmet Copper-nickel - PGM deposit", 2001 SME Annual Meeting & Exhibit, Denver Colorado, February, 2001.

ANNEX (FIGURES)

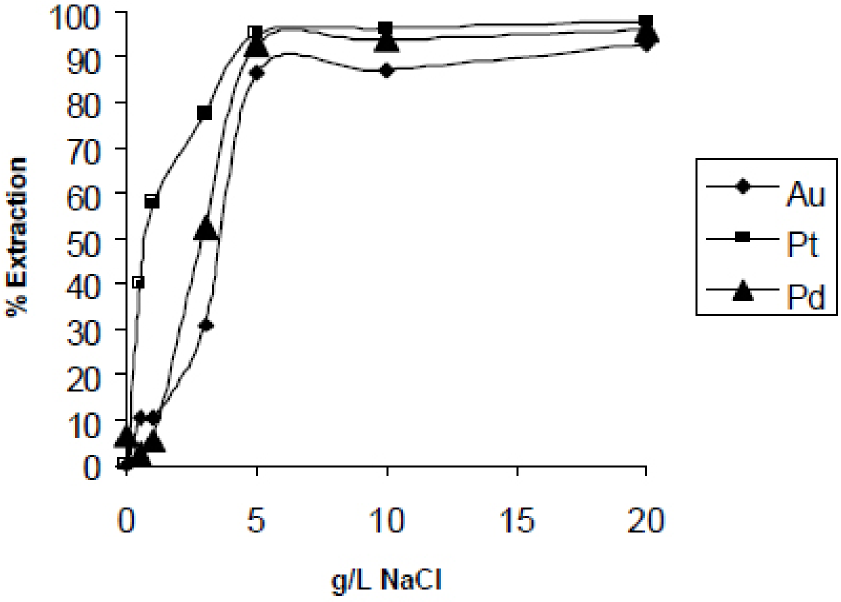

Figure 1: Effect of NaCl Addition on Metal Extraction - The PLATSOL^{IM} Process, 220ºC - 2 hours

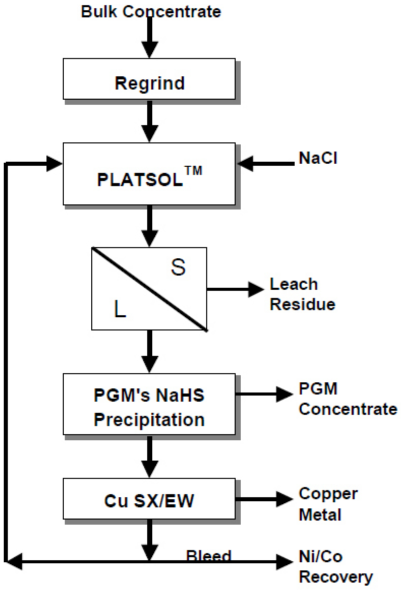

Figure 2: Application of the PLATSOLä Process to NorthMet Bulk Concentrate. Copper and PGM's Recovery Flowsheet

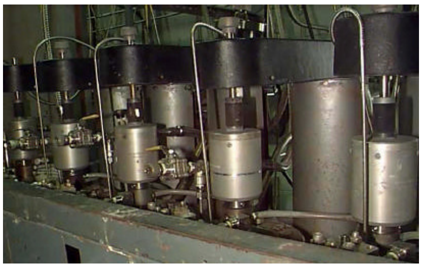

Figure 3: Continuous Autoclave Agitator Assemblies

Figure 4: Autoclave Feed after Several Hours

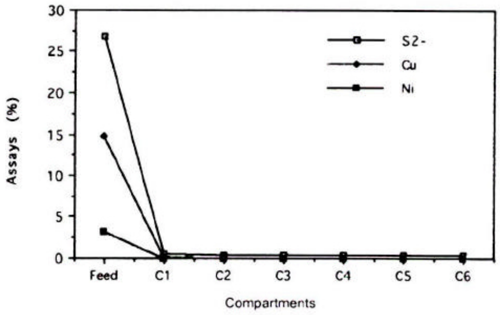

Figure 5: Process Kinetics for Cu, Ni, and S^{2-}

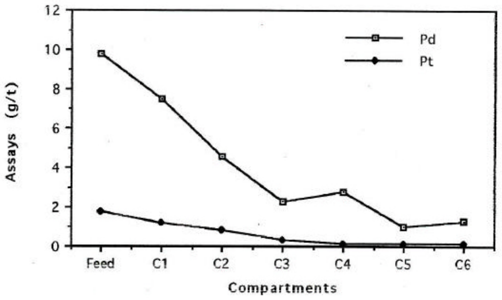

Figure 6: Process Kinetics for Pt and Pd

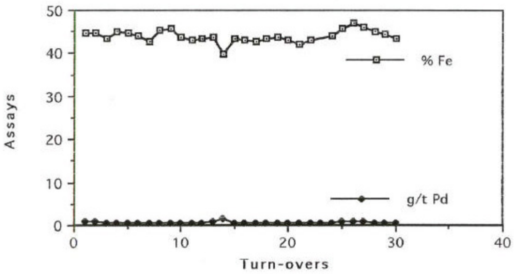

Figure 7: Analyses of the Autoclave Discharge - Last 3 days of Operation - Fe, Pd; 1 Turn-over = 2 hours

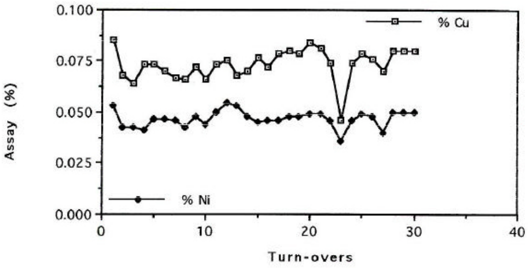

Figure 8: Analyses of the Autoclave Discharge - Last 3 days of Operation - Cu, Ni; 1 Turn-over = 2 hours

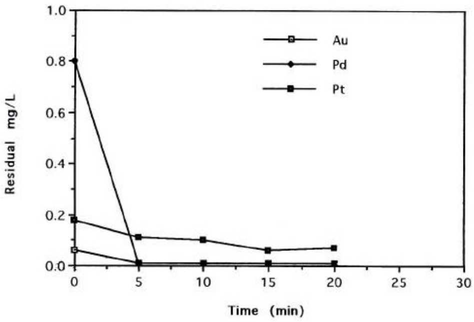

Figure 9: NaHS Precipitation of Au + PGM's - Pilot Plant - No Seed

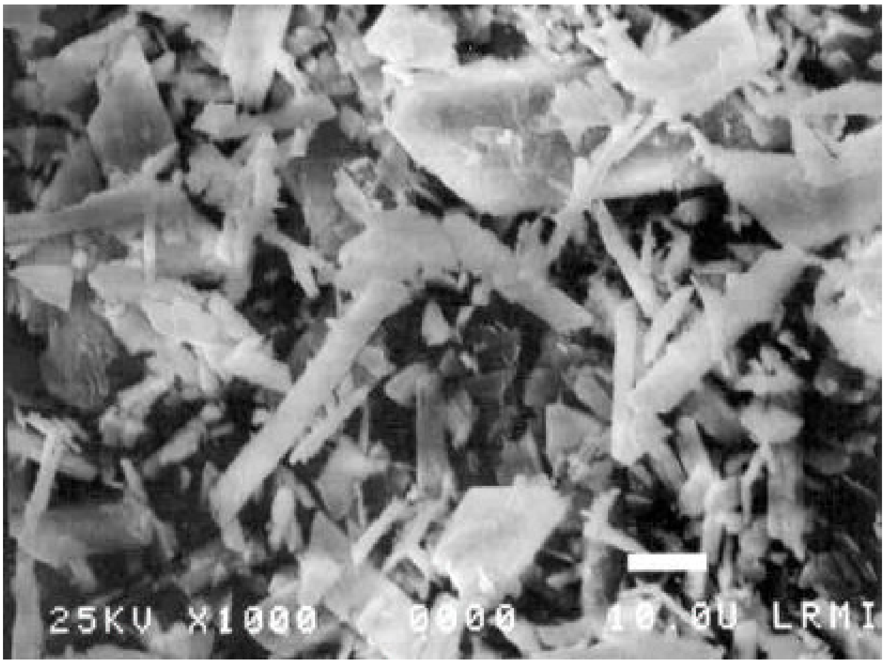

Figure 10: Gypsum produced during the NorthMet Pilot Plant

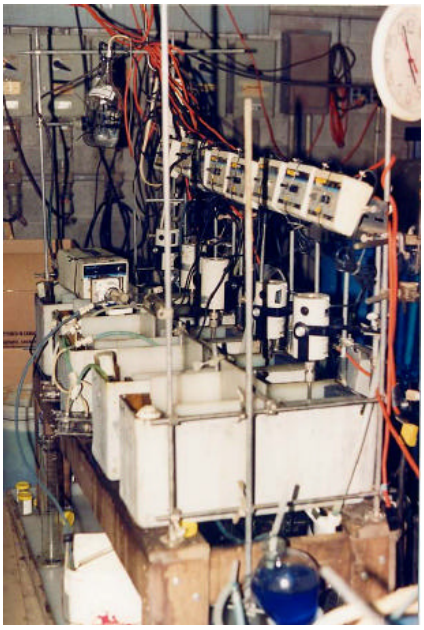

Figure 11: Cu solvent extraction circuit

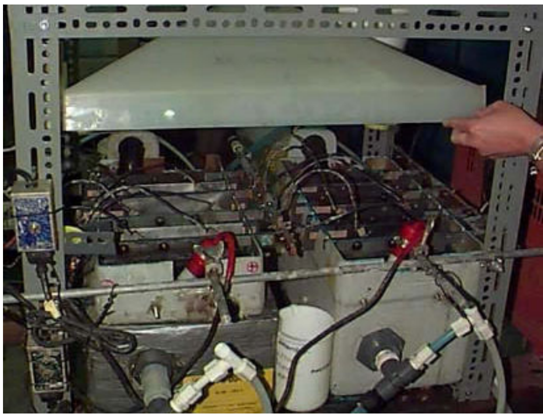

Figure 12: Cu Electrowinning Pilot Plant

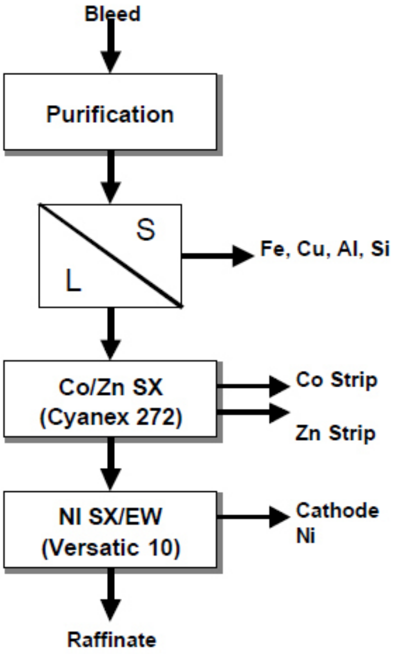

Figure 13: Flowsheet selected for the Treatment of NorthMet Bleed

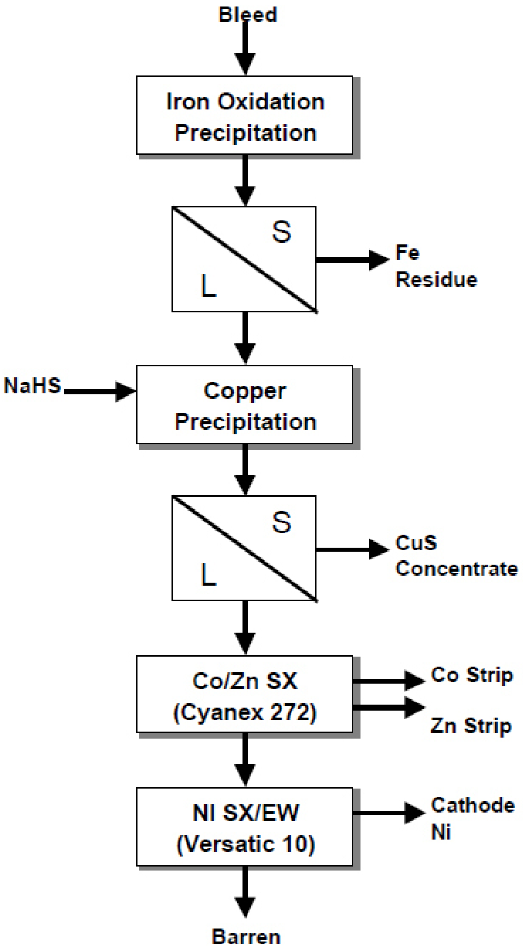

Figure 14: Purification of Bleed Stream

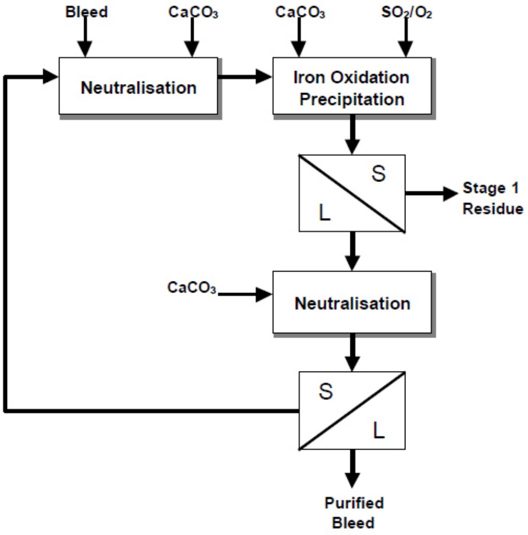

Figure 15: Neutralisation - Iron Removal

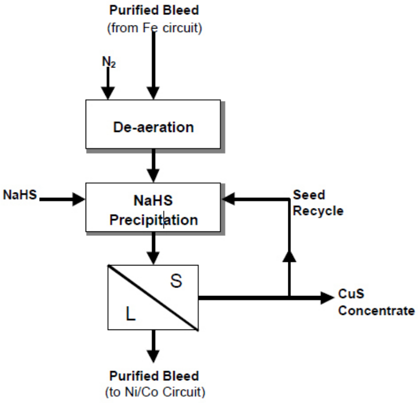

Figure 16: Copper Removal by Sulphidization

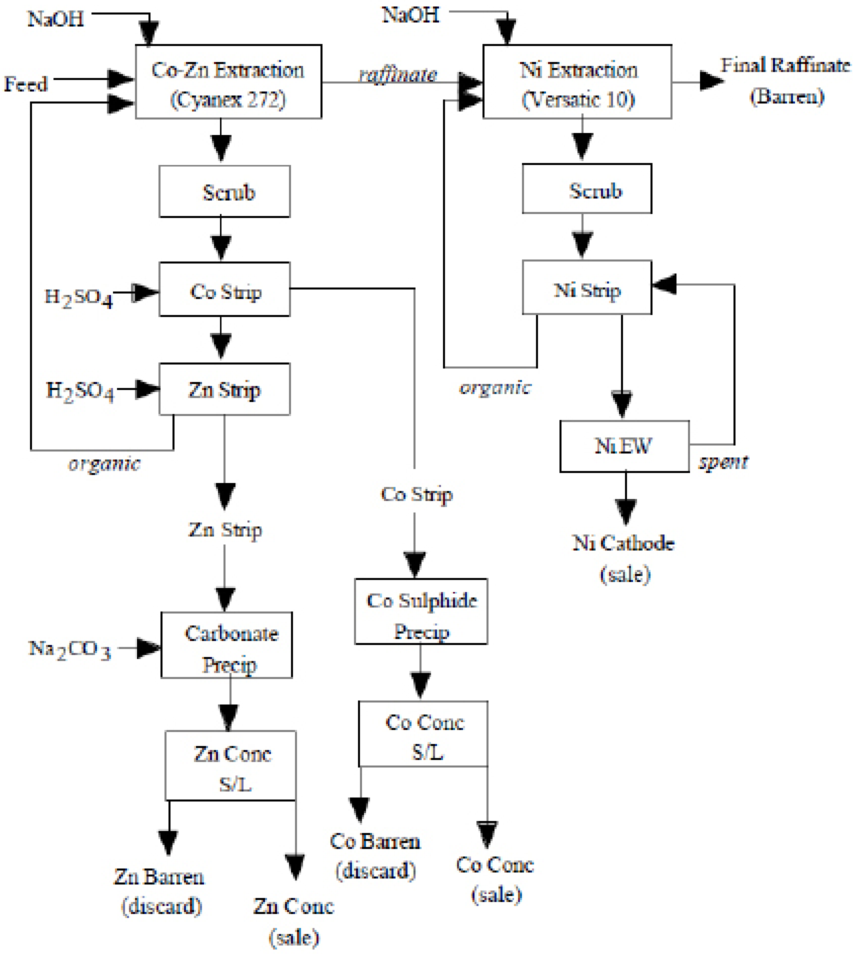

Figure 17: Process Description - Schematics

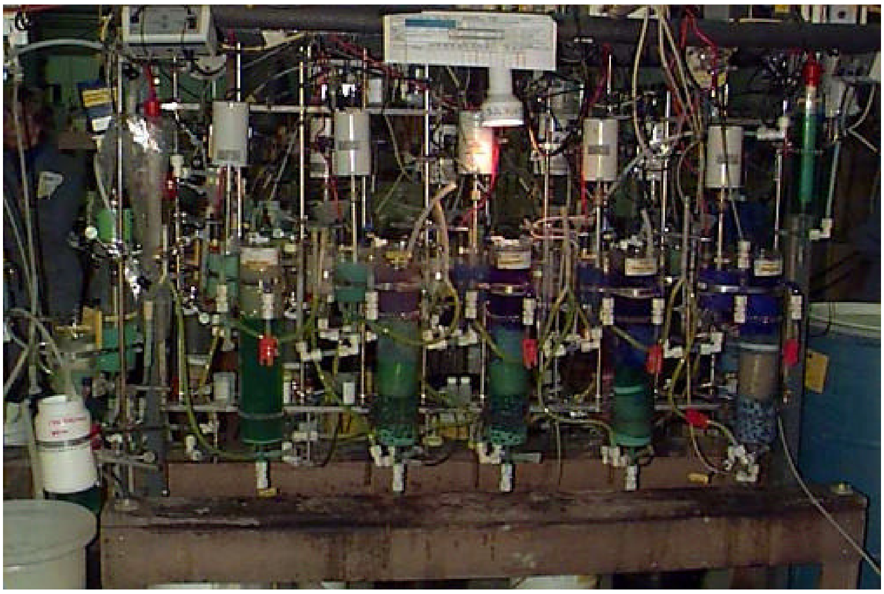

Figure 18: Ni/Co SX Circuit View

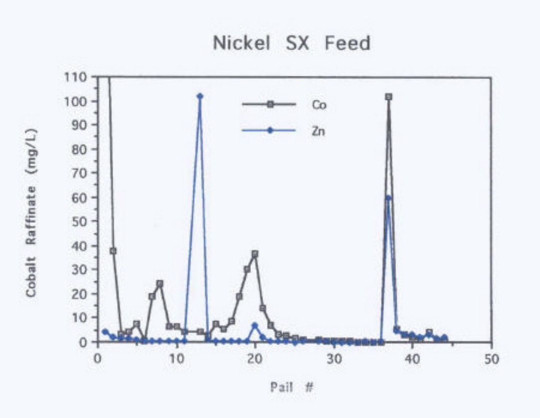

Figure 19: Co and Zn Assays of the Cyanex 272 Raffinate

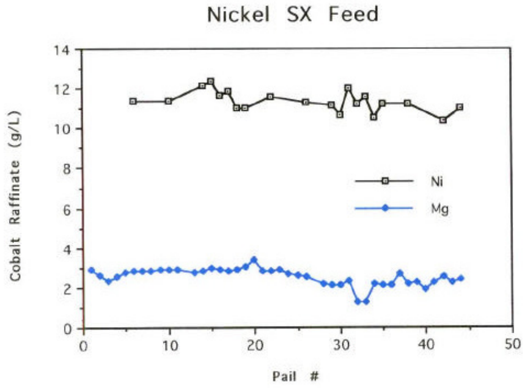

Figure 20: Ni and Mg Assays of the Cyanex 272 Raffinate

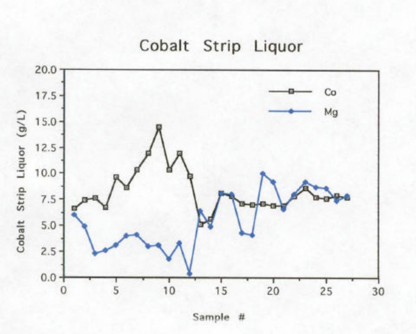

Figure 21: Co and Mg Analyses of Co Strip Liquor

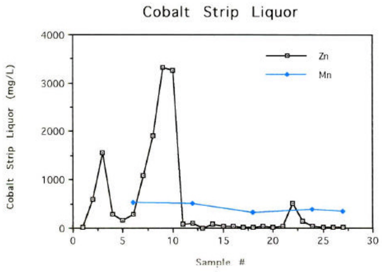

Figure 22: Zn, Mn Analyses of Co Strip Liquor

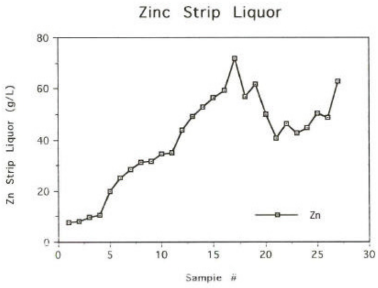

Figure 23: Zn Analyses of Zn Strip Liquor

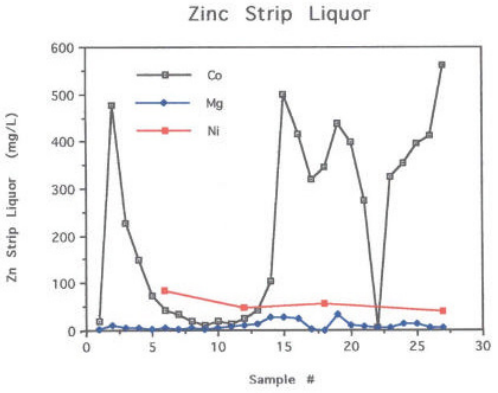

Figure 24: Co, Mg, Ni Analyses of Zn Strip Liquor

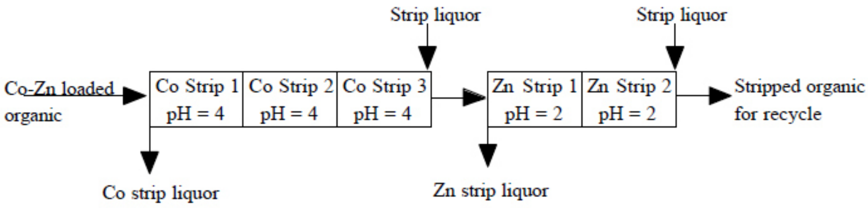

Figure 25: Co/Zn Selective Stripping Procedure Applied during the Pilot Plant

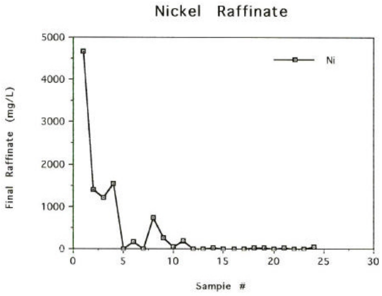

Figure 26: Ni Analyses of Final Raffinate

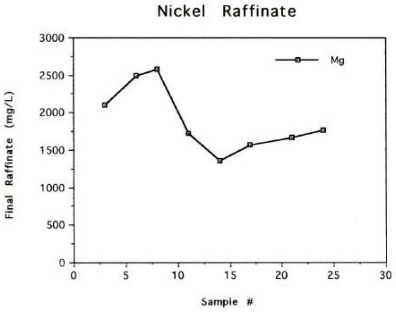

Figure 27: Mg Analyses of Final Raffinate

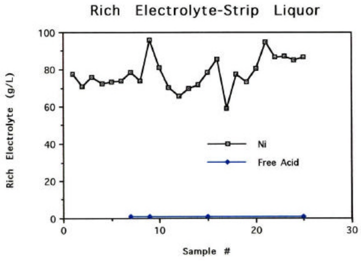

Figure 28: Ni and Free Acid Analyses of Rich Electrolyte

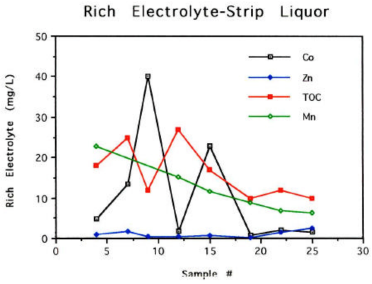

Figure 29: Co, Zn, Mn, TOC Analyses of Rich Electrolyte

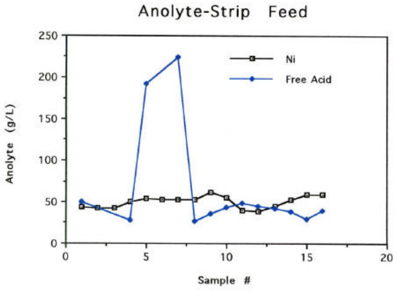

Figure 30: Ni and Free Acid Analyses of Spent Electrolyte

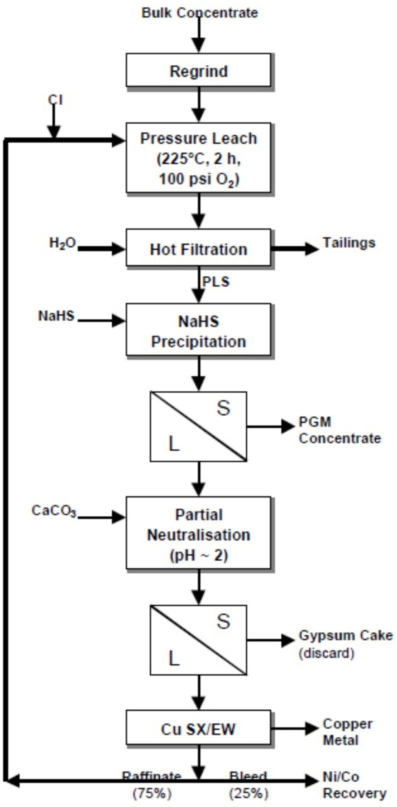

Figure 31: Recommended Process Flowsheet to Treat NorthMet Bulk Concentrate

ANNEX (TABLES)

Table 1: Chemical Composition of the NorthMet Concentrates tested during the Pilot Plant

ELEMENT %, G/T	BULK CONCENTRATE	
	YEAR 1999	YEAR 2000
Cu %	13.8	14.7
Ni	3.52	3.05
Fe	28.7	32.9
Co	0.15	0.14
S	25.6	26.7
Si	5.12	4.63
Au, g/t	2.24	1.41
Ag	44.4	-
Pt	1.75	2.22
Pd	8.91	9.90
Al	15,000	17,000
Ca	35,000	12,000
Mg	12,700	12,000
Mn	320	290
Na	4,100	3,200
Zn	6,000	2,600

Table 2: Chemical Composition of the Limestone tested during the Pilot Plant

COMPONENT %	MICHIGAN LIMESTONE
CaO	54.9
SiO_2	0.41
Al_2O_3	0.09
Fe_2O_3	0.20
LOI	43.9

Table 3: Average Pilot Plant Results - Leaching

ELEMENT	FEED	DAYS 1-3		DAYS 7-10	
		RESIDUE	EXTRACTION%	RESIDUE	EXTRACTION %
Cu (%)	14.7	0.086	99.6	0.074	99.6
Fe (%)	32.9	45.06	-	44.07	-
Ni (%)	3.05	0.047	98.9	0.047	98.9
Co (%)	0.14	0.004	97.1	0.006	96.0
Pd (g/t)	9.90	1.33	90.2	0.72	94.6
Pt (g/t)	2.22	0.164	94.6	0.12	96.0
Au (g/t)	1.41	0.22	88.6	0.20	89.4

Table 4: Composition of a Typical PLS

ELEMENT	MG/L
Ni	17,000
Cu	20,000
Fe	3,200
Cl	9,470
Al	2,300
Mg	3,600
Ca	540
Co	900
Mn	34
Na	1,400
Zn	2,200
H_2SO_4	75,000
	g/t
Au	0.11
Pt	0.23
Pd	1.03

Table 5: NaHS Batch Treatment of Pilot Plant Liquor

CONDITIONS	% EFFICIENCY			PGM CONCENTRATE	
	Au	Pt	Pd	% Cu	g/t (Au+Pt+Pd)
A	100	100	100	46.7	738
B	100	65	100	3.4	6220

Table 6: Analyses of Two Cu Cathode Samples Produced during NorthMet Pilot Plant

ELEMENT (PPM)	CU-1	CU-2	ASTM B115-93
Se	0.33	0.16	1
Te	<0.05	<0.05	0.2
Bi	<0.001	<0.001	1
Cr	0.01	0.003	0.2
Mn	<0.001	0.005	0.2
Cd	<0.01	<0.01	0.2
P	<0.001	<0.001	1
As	0.07	0.05	1
Sb	<0.005	<0.005	1
Pb	0.05	0.15	5
Si	0.12	0.09	1
Sn	<0.01	0.02	0.4
Ni	0.03	0.006	0.8
Fe	0.02	0.01	5
Zn	<0.05	<0.05	1
Co	0.02	0.007	0.2
S	2.5	0.8	10
Ag	0.16	0.15	12

Table 8: Typical Composition of Purified Bleed

ELEMENT	MG/L
Co	650-700
Ni	11-12,000
Mn	~40
Mg	~3,500
Ca	~580
Si	~38
Cu	<2
Fe	<10
Zn	~1900

Table 7: Average Composition of Bleed Stream

ELEMENT	MG/L
Cu	554
Ni	12,138
Fe	3,269
Co	643
Zn	1,747
Mn	31
Ca	565
Mg	3,086
Al	1,431
Na	1,102
Cl	5,941
H_2SO_4	17,400

Table 9: Analyses of Two Nickel Cathode Samples

ELEMENT (G/T)	NI CATHODE		
	SAMPLE 2	SAMPLE 3	SAMPLE 3 REPEAT
Si,	<50	<50	<50
As	<5.0	<5.0	<5.0
Ca	<5	<5	<5
Si	<25	<25	<25
Nat	<25	<25	<25
Se	<2.0	<2.0	<2.0
B	<10	<10	<10
Sb	<1.0	<1.0	<1.0
Ba	<1.0	<1.0	<1.0
Bi	<1.0	<1.0	<1.0
Ga	<1.0	<1.0	<1.0
V	<1.0	<1.0	<1.0
In	<0.50	<0.50	<0.50
Ag	<0.50	<0.50	<0.50
Sr	<0.50	<0.50	<0.50
Tl	<0.50	<0.50	<0.50
Ti	<0.50	<0.50	<0.50
Be	<0.5	<0.5	<0.5
ELEMENT	NI CATHODE		
	SAMPLE 2	SAMPLE 3	SAMPLE 3 REPEAT
Mg	<0.3	<0.3	<0.3
Co	420	420	420
Fe AA)	117	120	140
Pb	150	160	160
Cu	110	110	110
Zn	59	63	64
Cd	29	28	30
Sn	15	11	12
P	14	13	-
Mn	9.5	4.9	5
Na	9.0	6.4	6.7
Al	3.3	2.2	2.1
As	1.6	1.6	1.9
Te	0.61	0.62	0.63
Cr	0.42	0.34	0.33
Mo	0.34	0.33	0.33
Ge	0.3	0.32	0.32
Total	982.1	991.7	983.3

Table 10: Cobalt Precipitation using NaHS - No Seed

PRODUCT	ANALYSES (MG/L, %)			% RECOVERY		
	CO	NI	ZN	CO	NI	ZN
Start Solution	6,990	614	72.5	100	100	100
Barren	<0.5	<1.0	0.95	~0	0.2	1.0
Co Concentrate	29.9	2.64	0.40	100	99.8	99.0

Table 11: Zinc Carbonate Precipitation at 60°C

PRODUCT	ANALYSES (MG/L, %)			% RECOVERY	
	ZN	MG	CO	ZN	CO
Start Solution	61,400	15.9	385	100	100
Zn Conc 1	56.3	-	0.20	86.9	49.9
Zn Conc 2	40.4	-	1.30	13.1	50.0
Final Barren	5.6	11.5	0.2	0	0.1

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