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METALLURGICAL PROCESSING OF POLYMET MINING'S NORTHMET DEPOSIT FOR RECOVERY OF Cu-Ni-Co-Zn-Pd-Pt-Au

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INTRODUCTION

PolyMet Mining Corp. (PolyMet) is advancing the development of the NorthMet Project. The NorthMet deposit is located in northern Minnesota, adjacent to the historic Iron Range. The deposit was discovered in the 1960's and consists of a large, lower grade polymetallic sulfide deposit with values in Cu, Ni, Co, Zn, Au and Platinum Group Metals (PGM). The NorthMet resource is estimated at > 900 million tons @ >0.90 Cu Equivalent grade at a 0.15% Cu cut off grade.

PolyMet is currently completing a Definitive Feasibility Study (DFS) with Bateman Engineering (Australia). The study will include the mining, mineral processing and hydrometallurgical treatment of a bulk concentrate along with waste handling and disposal. The processing site will be the former LTV Steel Mining Company taconite iron ore processing plant which is located about 7 miles from the NorthMet deposit. The plant and associated mining operations were closed in early 2001 due to bankruptcy and at its peak, this plant processed over 90,000 long tons/ day of taconite iron ore via a four stage crushing, grinding and flotation process. PolyMet is now the owner of this plant (purchase was completed in 2005) and plans to reactivate sufficient capacity to treat 32,000 short tons per day of NorthMet ore to produce a bulk sulfide flotation concentrate for further treatment using the PLATSOL[™] process.

The DFS program has included pilot scale testwork at SGS Lakefield Research Limited in Ontario, Canada. Pilot plant tests have been performed on ore flotation to produce bulk concentrate samples and on hydrometallurgical treatment of the bulk concentrate materials. Two variants of the hydrometallurgical process were tested. The "base case" flowsheet involves high temperature chloride-assisted leaching (PLATSOL[™]), solid-liquid separation and washing, PGM/Au precipitation, neutralization, Cu SX/EW to produce copper cathode. The two variants, driven by offtake interest, include two different routes to nickel, cobalt, and zinc recovery. The first variation treats a portion of the Cu SX raffinate (remainder is recycled to the autoclave) sequentially by neutralization, first and second stage iron/aluminum removal, residual copper removal by precipitation as a sulfide, first stage Ni/Co/Zn precipitation with magnesia, second stage Ni/Co/Zn precipitation with lime and then magnesium removal with lime. The product from this first variation is a "mixed" hydroxide of nickel, cobalt and zinc.

The second variation again treats a portion of the Cu SX raffinate sequentially by neutralization, first and second stage iron/aluminum removal and residual copper sulfide precipitation. The Cu-Fe-Al free solution is then processed by Co/Zn solvent extraction (with Cyanex 272) with selective zinc and cobalt stripping, cobalt precipitation with magnesia, nickel (purified raffinate stream after Co/Zn SX) precipitation with magnesia, secondary precipitation of residual nickel with lime and finally magnesium precipitation with lime. The products from this second variation are a zinc sulfate strip solution (to a zinc offtake), a cobalt hydroxide precipitate and a nickel hydroxide precipitate.

The final choice between the mixed and purified hydroxide route for nickel and cobalt products will be based on final cost analysis and market surveys.

The following paper provides an overview of the current project and results from the pilot testing.

NORTHMET PROJECT

The NorthMet Project is located in northeastern Minnesota, near the Mesabi Iron Range and 6 miles south of the town of Babbitt. Figure 1 shows the location on a map of North America.

PolyMet has a 100% leasehold interest for the mineral rights on the 4,162 acres that make up the NorthMet property. The infrastructure is excellent. Available to the Project are low-cost power, well developed roads and railway networks, and supply-equipment centers that support the numerous operating iron ore



mines, the nearest being two miles from NorthMet. There is an abundant supply of skilled labor locally.

The NorthMet deposit is believed to be the largest undeveloped nonferrous metal project in the U.S. It is a polymetallic magmatic sulfide deposit containing platinum, palladium, gold, copper, nickel, cobalt, and silver, hosted near the base of the Duluth Mafic Complex.

Historically, the approach taken to development of NorthMet (formerly named the Dunka Road deposit), was to use open pit mining and flotation/ concentration of smelter grade nickel and copper concentrates for toll treatment. With prevailing smelter payment terms (and associated smelting and refining charges), the economics were never attractive enough for NorthMet to proceed as a "greenfields" project development using this approach. The losses of nickel and copper to tails and to the other concentrates and the relatively low levels of precious metals in the final concentrates mitigated against financial success. Starting in 1998, a new approach was taken to development of NorthMet including bulk flotation of the copper, nickel and associated base and precious metals and hydrometallurgical treatment of the bulk concentrate.

The NorthMet deposit is situated approximately 7 miles from the former LTV Steel Mining Company (LTV) taconite iron ore processing facility which prior to its closure due to bankruptcy in 2001 had capacity to treat over 90,000 long tons per day of taconite. Following closure Cliffs Erie LLC (a subsidiary of Cleveland Cliffs Inc.) acquired the mining and railroad related assets. PolyMet management recognized the opportunity that the disused taconite processing facility represented in terms of advancing NorthMet to production and in 2003 secured an option to purchase the crushing plant, concentrator, tailings impoundment and selected associated infrastructure. The option was exercised in late 2005

The significance of this acquisition is that the former LTV facilities and infrastructure now provide



Figure 1 The location of the NorthMet deposit, Minnesota, USA

the ready made comminution "front end" of the two-stage NorthMet processing route with a corresponding capital cost saving of several hundreds of millions of dollars. Given that the rate of initial treatment of NorthMet ore is 32,000 short tons per day, there is considerable scope for future expansion.



Figure 2 Overview of the "Cliff's Erie" Site Purchased by PolyMet Mining Corp. (This photograph was taken when the plant was producing taconite pellets seen in the foreground stockpiled ready for rail shipment.)

DRILL PROGRAM

During 2005 a diamond drilling program was carried out which focused on the areas that are expected to be mined in the first 20 years of the mine plan. The first phase of the program was intended to provide additional resource definition as well as a 42 short ton bulk sample for metallurgical testing at SGS Lakefield Research near Toronto, Ontario. The resultant bulk sample, which represented the envelope expected

SGS MINERALS SERVICES | TECHNICAL BULLETIN 2006-06

to be mined during the first 10 years of production, was composited as three separate blended samples representing the expected high, medium and low grade mill feed values. Subsequently, in early 2006, an additional 9 short tons of drill-derived material in two composite samples were subjected to a further phase of pilot-scale flotation and autoclave testing at Lakefield.

A final phase of DFS diamond drilling, which comprised exclusively of resource definition and in-fill drilling, was carried out during the winter of 2005-2006 and completed in February 2006.

All of the goals for the drill program have been met, including:

- Gathering geotechnical information for pit slope design – pit slope stability studies by Golder Associates are nearing completion;
- Enhancing the comprehensive geological model through in-fill drilling

 a new, detailed geological and assay model incorporating all work from 2005 has been completed and is contributing to ongoing resource evaluation and pit design work by PolyMet staff, resource consultants Hellman & Schofield and Australian Mine Development and Design (AMDAD);
- Confirming continuity of metal grades;
- Collecting data to support environmental permitting, and
- Improvement in the confidence of resource estimate categorization resulting from reduced drill hole and sample spacing.

During the 2005 program, 109 holes totaling 77,166 feet were drilled taking the cumulative drilling at the project to 310 diamond and reverse circulation holes totaling 261,227 feet. In addition to the latest drill program, PolyMet has meticulously recompiled all of the prior work started by US Steel in 1969, increasing the number of samples from nearly 12,000 to 17,194. The latest program added a further 12,806 samples, taking the total to 30,000.

In addition, drill spacing in the area to be mined in the first five years has been reduced to an average of 311 feet (from 427 feet) while the 20-year mine plan spacing has been reduced to an average of 360 feet. In Hellman & Schofield's 2005 report, a spacing of 360 feet was used to define material in the "Measured" category. Hellman & Schofield is currently finalizing the DFS Resource Estimate which includes results from all drilling done on the NorthMet deposit to date.

NORTHMET PROCESS FLOWSHEET AND PILOT PLANT STUDIES

The overall NorthMet process flowsheet involves two distinct circuits; a classical mineral processing flowsheet to produce a bulk concentrate and a hydrometallurgy flowsheet for extracting Cu-Ni-Co-Zn-Au-PGM from the bulk concentrate. The hydrometallurgy flowsheet involves chloride-assisted leaching of base and precious metals followed by a series of metal recovery steps for the base and precious metals. Within the hydrometallurgy process flowsheet, there are two options for nickel and cobalt recovery that have been carried in parallel through testwork and engineering. Final selection between

hydrometallurgy.

The pilot scale tests at SGS-Lakefield were performed in 2005 and 2006. The ore samples sent to SGS-Lakefield comprised 4 composites and a sample of assay reject material that was used as "startup" material for the flotation pilot plant. The ore samples were processed through crushing, grinding and flotation to produce a series of bulk concentrates for hydrometallurgical pilot plant testing. Table 1 below summarizes the pilot plant feed assays and weights processed.

The following is a general description of each part of the proposed plant along with selected results from the 2005 and 2006 continuous pilot testing of the process at SGS Lakefield. The pilot scale tests at SGS Lakefield form the testwork basis for the final feasibility study design for the NorthMet plant.

CRUSHING, GRINDING AND FLOTATION

The crushing, grinding and flotation circuit (Figure 3) receives ore from the mine into a coarse crusher dump pocket. The ore then proceeds through primary,

Table 1. Pilot Plant Ore Samples Processed at SGS in 2005 and 2006

SAMPLE*	WEIGHT (t)	ASSAY (%	ASSAY (% or g/t)									
		Cu	Ni	S	Pt	Pd	Au					
Composite 1	9.5	0.31	0.095	0.73	0.06	0.23	0.03					
Composite 2	19.2	0.35	0.1	0.77	0.07	0.33	0.05					
Composite 3	9.6	0.4	0.11	0.87	0.09	0.3	0.05					
Startup Material	3.6	0.31	0.09	0.87	0.04	0.31	0.05					
Composite 4	4.4	0.31	0.1	0.91	0.05	0.28	0.08					

* Composites 1-3 were processed in 2005 and the Startup Material and Composite 4 were processed in 2006

the two options will be determined by analysis of relative capital and operating costs and final discussions with metal off-take parties. The first option produces a "mixed hydroxide" which will also contain any zinc that reports to the bulk flotation concentrate. The second option utilizes solvent extraction to separate Ni, Co and Zn into separate streams for precipitation as individual metal hydroxides.

Figures 3, 4, 5 show the flowsheets in pictorial form. Figure 3 is the mineral processing flowsheet with figures 4 and 5 showing the two options for

secondary, tertiary and quaternary crushing followed by rod and ball milling to a size of 100-125 μ m P₈₀. The crushing and grinding circuits are existing circuits within the "Cliff's Erie" plant and will only utilize a small portion of the available capacity.

SGS MINERALS SERVICES | TECHNICAL BULLETIN 2006-06

The flotation circuit is designed to produce a bulk concentrate carrying pay metals and a tailing with low residual sulfur content. The float circuit has the following elements;

- Rougher conditioning with potassium amyl xanthate (PAX)
- Rougher flotation with MIBC/DF250 addition
- Scavenger flotation conditioning with copper sulfate (to activate sulfides)
- Scavenger flotation with application of PAX, MIBC and DF250.
- The rougher float concentrate (and the first scavenger float concentrate – not shown on Figure 4) is sent to conditioning followed by three

stages of cleaning to produce a final concentrate.

- The scavenger flotation concentrate and the first cleaner flotation tailings are reground to 30 μm particle size (P₈₀) and directed back to the rougher flotation circuit
- The 3rd cleaner concentrate is thickened and reground to approximately 15 μm particle size (P_{_{80}}) and sent to the autoclave feed tanks.

A series of batch and continuous pilot plant crushing/grinding/flotation tests were performed at SGSLakefield as part of the metallurgical development program. The results of a series of six test periods are summarized in Table 2. The first two sample periods were treating assay reject "startup" material followed by treatment of a composite (C4). The C4 composite sample was obtained from NorthMet deposit in the area of the first 5 years of the NorthMet mine life. On average for the 4 periods in which C4 sample was processed, the results indicate;

 3.73 % mass pull and float recoveries of 89.8% for Cu, 70.5% for Ni, 87.2% for S, 36.2% for Co, 73.3% for Au, 84.7% for Pt and 82.7% for Pd.



Figure 3 NorthMet Process Flowsheet: Crushing, Grinding and Flotation of a Bulk Cu-Ni-Co-Zn-Au-PGM Concentrate



Figure 4 NorthMet Process Flowsheet: Hydrometallurgical Treatment of Bulk Cu-Ni-Co-Zn-Au-PGM Concentrate with Production of Copper Cathode, Au/PGM Precipitate and Mixed Ni/Co Hydroxide.



Figure 5 NorthMet Process Flowsheet: Hydrometallurgical Treatment of Bulk Cu-Ni-Co-Zn-Au-PGM Concentrate with Production of Copper Cathode, Au/PGM Precipitate, Zinc Hydroxide, Nickel Hydroxide and Cobalt Hydroxide.

Table 2 Selected Flotation Pilot Plant Test Results Obtained at SGS Lakefield in 2006	

Test	Comp	Product	Wt	Grade							Distribution, %						
No.			%	Cu , %	Ni, %	S, %	Co , %	Au, g/t	Pt, g/t	Pd, g/t	Cu	Ni	S	Co	Au	Pt	Pd
1	Startup	Feed	100	0.35	0.1	0.79	0.01	0.05	0.05	0.26	100	100	100	100	100	100	100
		Tail	96.8	0.036	0.035	0.13	0.007	0.008	0.006	0.044	9.9	32.4	16.2	66	16	11.2	16.7
		Conc	3.17	10.1	2.2	20.8	0.11	1.28	1.46	6.71	90.1	67.6	83.8	34	84	88.8	83.3
2	Startup	Feed	100	0.38	0.11	0.85	0.008	0.05	0.05	0.25	100	100	100	100	100	100	100
		Tail	96.2	0.032	0.03	0.12	0.005	0.009	0.009	0.043	8.1	27	14.1	57.2	16.3	16.3	3.4
		Conc	3.8	9.22	2.04	19.3	0.095	1.17	1.17	6.47	91.9	73	85.9	42.8	83.7	83.7	85.6
3	C4	Feed	100	0.34	0.1	0.87	0.008	0.04	0.06	0.29	100	100	100	100	100	100	100
		Tail	96.4	0.034	0.03	0.12	0.005	0.014	0.008	0.053	9.6	28.7	13.6	58.6	31.2	13.3	17.7
		Conc	3.58	8.57	1.97	21.1	0.095	0.83	1.41	6.63	90.4	71.3	86.4	41.4	68.8	86.7	82.3
4	C4	Feed	100	0.34	0.01	0.93	0.008	0.04	0.07	0.29	100	100	100	100	100	100	100
		Tail	95.8	0.037	0.029	0.11	0.005	0.006	0.005	0.051	10.5	28.7	11	62.7	13.6	6.7	16.8
		Conc	4.2	7.16	1.66	19.8	0.068	0.87	1.59	5.76	89.5	71.3	89	37.3	86.4	93.3	83.2
5	C4	Feed	100	0.33	0.1	0.85	0.009	0.04	0.06	0.28	100	100	100	100	100	100	100
		Tail	96.3	0.032	0.028	0.1	0.006	0.01	0.008	0.044	9.3	27.3	11.5	63.7	25.8	13.7	15.3
		Conc	3.74	8.05	1.91	20	0.088	0.74	1.3	6.25	90.7	72.7	88.5	36.3	74.2	86.3	84.7
6	C4	Feed	100	0.32	0.1	0.86	0.01	0.03	0.06	0.26	100	100	100	100	100	100	100
		Tail	96.6	0.039	0.034	0.13	0.007	0.013	0.018	0.052	11.6	33.1	15.1	70.2	36.2	27.6	19.4
		Conc	3.41	8.4	1.97	21.5	0.084	0.65	1.34	6.12	88.4	66.9	84.9	29.8	63.8	72.4	80.6

AUTOCLAVE LEACHING

The autoclave leaching process for NorthMet utilizes a small amount of chloride (approximately 10 g/L) in solution under "total pressure oxidation" conditions to extract Cu, Ni, Co, Zn, Au, Pt, Pd from the bulk concentrate. The principles of the PLATSOL® process for NorthMet ore processing have been described elsewhere [1] and will only be briefly reviewed here. The process is regarded as a nominal "step-out" from commercial practice in total pressure oxidation for base and precious metal ore/concentrate treatment.

The autoclave oxidation process converts metal sulfide minerals into metal sulfates and iron hydrolysis products (primarily hematite but some basic ferric sulfate may also form under high acid conditions), while the precious metals are converted to chloro-complexes. The chemical reactions believed to occur in the autoclave are shown below. (Note that the mineralogy of the PGM's may be very complex, but for simplicity only the metallic species are considered.)

Chalcopyrite Oxidation/Iron Hydrolysis: $CuFeS_{2} + 4.25O_{2} + H_{2}O = CuSO_{4} +$ 0.5Fe₂O₃ + H₂SO₄ Pyrite Oxidation: $FeS_2 + 3.75O_2 + 2H_2O = 0.5Fe_2O_2 +$ 2H₂SO₄ Pyrrhotite Oxidation Fe7S_o + 16.25O_o + 8H_oO = 3.5Fe_oO_o + 8H_SO Nickel Sulfide Oxidation: $NiS + 2O_2 = NiSO_4$ Basic Ferric Sulfate Formation: $Fe_2O_2 + 2H_2SO_4 = 2Fe(OH)SO_4 + H_2O_4$ Gold Oxidation/Chlorocomplex Formation: $Au + 0.75O_2 + 4HCI = HAuCI_4 + 1.5H_2O_2$ Platinum Oxidation/Chlorocomplex Formation: $Pt + O_2 + 6HCI = H_2PtCI_6 + 2H_2O$

Palladium Oxidation/Chlorocomplex Formation: $Pd + 0.5O_2 + 4HCl = H_2PdCl_4 + H_2O$

The temperature range for total pressure oxidation is typically 220 to 230°C. The autoclave discharge from the leach process contains dissolved Cu, Ni, Co, Zn, Au, Pt and Pd. The solids are relatively "barren" of value, consisting of iron precipitates, unreacted gangue and minor amounts of residual base and precious metal minerals.

The original PLATSOL® pilot plant utilized a "straight through" design [1] in which fresh concentrate was introduced into the first compartment of a 6-compartment pilot autoclave with raffinate (for controlled cooling of the autoclave slurry) and oxygen (for oxidation). The results of the "straight through" pilot plant indicated that virtually all the copper and nickel were extracted in the first compartment of the autoclave due to the rapidity of the sulfide oxidation process. However, the platinum and palladium minerals continued to react through the entire autoclave volume, reaching ultimate extractions of about 95%. This situation was revisited in the more recent bench and pilot scale testing. It was concluded that a "recycle' design would be more efficient with respect to autoclave design. The "recycle" design involves thickening the autoclave discharge and recycling a portion of the underflow to the autoclave feed. This recycle allows for any unreacted mineral to have a chance at second-pass extraction. The greater the recycle, the longer the "average" residence time of solids in the autoclave. The limit of the recycle will be

when the solids density in the autoclave becomes unmanageable.

Figures 6 and 7 shows the impact of solids recycle on base and precious metal extraction. While there is some "noise" in the results, there is a strong indication of improvement in both base and precious metal extraction in applying the recycle system. Nickel extraction improved from 97% extraction to over 98% while platinum and palladium extraction improved from less than 90% to between 90 and 95 overall extraction. Regardless of the recycle ratio, the copper extraction was excellent at over 99%. Gold extraction was highly variable, probably due to the small amount of gold in the feed sample (0.8 to 1.3 g/t), but always around 90%. The method of reporting recycle is mass of recycle solids per mass of feed solids (t/t), expressed as a percent.



Figure 6 Copper and Nickel Extraction as a Function of Recycle of Autoclave Discharge Solids. Standard conditions: 225°C, 1.1 hour single pass residence time, 100 psig of oxygen overpressure, 10 g/L Cl, 6-compartment autoclave.

Inspection of the graphs indicates that at least 100% recycle ratio will improve overall metal extractions.

Further insight into the kinetics of the pressure oxidation process can be obtained by taking compartment samples from the autoclave at steadystate. Figure 8 shows the precious metal solution and solids assays in the compartment samples obtained at 74% recycle ratio. Figure 9 gives the comparable information for the base metals. Note that the feed grades in each case have been diluted with the barren solids recycled from the autoclave discharge.

The results show clearly that even with recycle and a relatively short autoclave residence time of 1.1 hours, the oxidation and extraction of the bulk of the base and precious metals is largely complete within the first two compartments of the autoclave (approximately 22 minutes of the 66 minute residence time). This result is consistent with all other reported information on total pressure oxidation of sulfide concentrates including the developments of Placer Dome and Phelps Dodge on copper concentrate total oxidation [2, 3].

The full plant design at NorthMet will allow for up to 300% recycle capacity to maximize extraction. Significantly, the use of recycle has allowed the specific throughput of the installed autoclaves to approximately double with respect to fresh feed (tonnes of fresh feed per m³ of autoclave volume). There may well be scope, once the plant has started, to make further increases in intensification of the process.

AU AND PGM PRECIPITATION

The autoclave discharge slurry is partly recycled (after thickening) and partly filtered to advance the metal bearing solution to the downstream recovery steps in the process. The gold and platinum group metals in solution are the first target for recovery using reductive precipitation.

In the first PLATSOL® pilot plant reported in 2001 [1], a combination of sulfur dioxide reduction of ferric ion followed by sulfide precipitation was



Figure 7 Au, Pt and Pd Extraction as a Function of Recycle of Autoclave Discharge Solids. Standard conditions: 225°C, 1.1 hour single pass residence time, 100 psig of oxygen overpressure, 10 g/L Cl, 6-compartment autoclave.

used to recover the precious metals from the pregnant solution.

In the recent pilot campaign an improved method of precious metal recovery was tested. Copper sulfides produced by precipitation of copper from the bleed stream advancing to nickel and cobalt recovery were utilized for precious metal recovery. The copper sulfides "cement" the precious metals onto the solid surface, resulting in an enriched product to advance to further processing.

The improved process was proven to be robust and flexible. It is still advantageous to reduce the ferric species in the advancing solution using sulfur dioxide gas to minimize the CuS demand for Au and PGM precipitation.

Ferric Reduction $Fe_2(SO_4)_3 + SO_2 + 2H_2O = 2FeSO_4 + 2H_2SO_4$ Gold Precipitation $2HAuCI_4 + 3CuS + 3H_2SO_4 = 2Au + 3CuSO_4 + 8HCI + 3S$ Platinum Precipitation $H_2PtCI_6 + 2CuS + 2H_2SO_4 = Pt + 2CuSO_4 + 6HCI + 2S$ Palladium Precipitation $H_2PdCI_4 + CuS + H_2SO_4 = Pd + CuSO_4 + 4HCI + S$

The recovery of gold, platinum and palladium from the autoclave solution (after filtering and washing the solids) was accomplished by precipitation with CuS in the pilot plant. The precipitate solids are collected in a thickener/ clarifier arrangement and then filtered. Recoveries of gold, platinum and palladium into this precipitate concentrate were excellent, in excess of 99.5% in each case. Base metal losses from solution into the PGM and gold residue were negligible.

Approximately 4 kg of precipitate were collected during the 2005 pilot plant analyzing 56 g/t Au, 211 g/t Pt and 907 g/t Pd. Most of the precipitate mass was copper (35.7%) and sulfur (49%). Batch releaching of the precipitate to remove copper and sulfur was tested and resulted in an upgraded material analyzing approximately 1.6% or 16,000 g/t total contained gold, platinum and palladium. PolyMet will chose between placement of the primary precipitate or the re-leach residue with an offtake/ refinery partner for final separation into pure precious metal products.

The solution after gold, platinum and palladium recovery advances to base metal recovery. The solution is still acidic and must be neutralized prior to copper solvent extraction.

SOLUTION NEUTRALIZATION

The extraction of copper by solvent extraction is inhibited by acid in solution. It is therefore important to neutralize the excess acid from the autoclave process prior to advancing to copper recovery.



Figure 8 Compartment Sample Assays for 74% Recycle Ratio for Au, Pt, Pd at Steady State. (a) Solution Assays and (b) Solid Assays.



Figure 9 Compartment Sample Assays for 74% Recycle Ratio for Ni, Cu, Fe at Steady State. (a) Solution Assays and (b) Solid Assays.

The neutralization process was piloted in a three stage neutralization circuit with limestone slurry for neutralization. The gypsum product is thickened. A 300% recycle of gypsum as "seed" for precipitation was used to grow coarse, clean, crystals of gypsum.

The chemistry of neutralization is shown below.

Gypsum Precipitation $H_2SO_4 + CaCO_3 + 2H_2O = CaSO_{4,2}H_2O$

The utilization of carbonate exceeded 99% in the pilot tests due to the use of the 300% recycle. The gypsum product assay is shown below. The chemical quality, physical nature and colour of this synthetic gypsum produced in the pilot plant appear to be suitable for entry into the gypsum wallboard market in the U.S. If an "off-take" for synthetic gypsum can be finalized, the gypsum residue will not fill space in the lined tailings facility planned for "hydromet" tailings. The value of the gypsum will partially offset the cost of oxygen for oxidation of sulfur in the concentrate to sulfate and the cost of purchasing limestone for neutralization.

COPPER SX/EW

The recovery of copper by SX/EW is conventional technology. The presence of chloride in the feed solution to copper solvent extraction necessitates the application of a wash or scrub step to displace any entrained chloride from the loaded organic solution.

Ni %	Co %	Cu %	Fe %	Zn %	SiO ₂ %	Al ₂ 0 ₃ %
0.05	<0.002	0.005	<0.05	0.003	0.19	<0.01
Fe₂0 ₃ %	Mg0 %	Ca0 %	Na ₂ 0 %	K ₂ 0 %	TiO ₂ %	P ₂ 0 ₅ %
0.06	0.2	31.7	M0.05	<0.01	<0.01	<0.01
MnO %	Cr₂0 ₃ %	V ₂ 0 ₅ %	LOI %	SO ₄ %	CO ₃ %	Cl g/t
<0.01	<0.01	<0.01	21.3	51	0.07	24

Table 3 Gypsum Solids Assay from Neutralization of Autoclave Acid

Copper Extraction

 $CuSO_4 + 2HR(org) = CuR_2(org) + H_2SO_4$ Copper Stripping $CuR_2(org) + H_2SO_4 = CuSO_4 + 2HR(org)$ Copper Electrowinning $CuSO_4 + H_2O = Cu + O_2(gas) + H_2SO_4$

In the pilot plant, copper was extracted in three counter-current stages, scrubbed in one and stripped in two stages with spent electrolyte from the electrowinning cell. Two extractants were evaluated, 35% Acorga M5640 from Cytec in Orfom SX 80CT and 35% LIX 973NS LV from Cognis in the same diluent. Both extractants performed well in the pilot plant operation. Extractions averaged about 95 % from a starting solution concentration of 17.4 g/L of Cu (average). A total of 68 kg of copper were extracted and electrolyzed during the 2005 pilot plant run at a current density of +270 A/m². The copper metal cathode was analyzed and found to meet LME grade A copper purity specifications. This product can be sold directly to copper consumers. Table 4 summarizes the assay for copper for two samples taken from the second strip cycle in the pilot plant.

RAFFINATE NEUTRALIZATION

The purpose of raffinate neutralization is to trim the acid level in the copper raffinate prior to splitting the raffinate flow between the nickel and cobalt recovery circuit and returning the raffinate to the autoclave circuit as a cooling solution. Acid must be neutralized prior to the nickel and cobalt recovery process steps and excess acid is undesirable in the feed to the autoclave as excess acid will increase the formation of basic ferric sulfate in the autoclave solids.

The chemistry of raffinate neutralization is the same as the primary neutralization discussed above.

Fe AND AI REMOVAL

The removal of iron and aluminum prior to recovery of nickel and cobalt is necessary to prevent product contamination.

The process of iron oxy-hydrolysis is well known. Iron is oxidized from the ferrous

Table 4. Copper Assays for Samples Taken from the Second Pilot Plant Strip Cycle¹.

	Analysis (g/t)									
Sample	Fe	S	Pb	Ag						
Cathode 1	1.02	10.5	0.52	<1						
Cathode 2	1.31	6	3.63	<1						
LME Grade A	10	15	5	25						

¹ The first strip cathode had higher levels of contaminants. This was attributed to the "startup" conditions used and the fact that the lead anodes in electrowinning were not conditioned prior to commencement of the pilot plant. The purity of the second strip cathodes is believed to be representative of the full scale chemistry of the copper cathodes to be produced at NorthMet.

to the ferric state with oxygen or air with limestone addition for neutralization and pH control. The temperature for iron removal was set to 60 $^{\circ}$ C and the pH to 3.0. The iron was removed to less than 5 mg/L by this process with negligible losses of Ni/Co to the iron precipitate residue.

Similar results were obtained in the pilot plant using limestone to pH 4.6-4.7 and 65 °C. Terminal aluminum levels were 38 mg/L on average.

The iron precipitate is washed and disposed to tailings. The aluminum precipitate is thickened and recycled to the iron removal step.

COPPER REMOVAL

Residual copper is removed before nickel, cobalt and zinc recovery using NaSH precipitation. The copper sulfide product (synthetic covellite) is then recycled internally to precipitate gold and PGM from the autoclave discharge solution.

The chemistry is straightforward;

 $CuSO_4 + NaSH = CuS + 0.5Na_2SO_4 + 0.5H_2SO_4$

In the 2005 pilot plant, the copper level in solution advancing to copper removal was reduced to less than 50 mg/L.

NICKEL, COBALT AND ZINC RECOVERY

There were two options for nickel, cobalt and zinc recovery that were continuously piloted at SGS – Lakefield in 2005. The first option utilized a classic "mixed hydroxide" precipitation route for recovery of the pay metals in a single product. The second option was to separate cobalt and zinc away from nickel using solvent extraction, followed by selective stripping of cobalt and zinc. The purified nickel, cobalt and zinc streams can then all be treated with magnesia to recover purified hydroxides (or in the case of zinc, be sold directly into the zinc chemicals industry).

Mixed Hydroxide Results

The mixed hydroxide process uses two-stage precipitation for nickel and cobalt recovery. In the first stage, approximately 85% of the nickel and cobalt are precipitated with magnesia (MgO). This precipitate is then thickened, filtered and washed and sent to a nickel off-take party. The balance of the nickel and cobalt are then precipitated with lime to form mixed gypsum – metal hydroxide precipitate. This precipitate is recycled to solution neutralization to re-dissolve the precipitated nickel and cobalt.

Zinc in solution will report quantitatively to the final mixed hydroxide precipitate. Nickel Precipitation with Magnesia NiSO₄ + MgO + H₂O = Ni(OH)₂ + MgSO₄

Cobalt Precipitation with Magnesia $CoSO_4 + MgO + H_2O = Co(OH)_2 + MgSO_4$ Zinc Precipitation with Magnesia $ZnSO_4 + MgO + H_2O = Zn(OH)_2 + MgSO_4$

SGS MINERALS SERVICES | TECHNICAL BULLETIN 2006-06

Sample	Moist. %	Ni %	Co %	Cu %	Fe %	Zn %	AI %	Mg %	Ca %	Si %	Mn %
1	51.2	36.3	1.92	0.37	0.59	4.84	0.07	1.04	0.02	0.05	0.03
2	41.2	31.5	1.67	0.31	0.51	4.31	0.04	0.62	0.04	0.03	0.02
		31.3	1.67	0.32	0.54	4.27	0.04	0.62	0.04	0.03	0.02
3	56.3	40.6	2.17	0.41	0.68	0.56	0.05	0.76	<0.08	0.04	0.03

Table 5. Analysis of the Mixed Hydroxide Product from the Pilot Plant

Residual Nickel Precipitation with Lime NiSO₄ + CaO + $3H_2O = Ni(OH)_2 + CaSO_{4.2}H_2O$ Residual Cobalt Precipitation with Lime CoSO₄ + CaO + $3H_2O = Co(OH)_2 + CaSO_{4.2}H_2O$ Residual Nickel Precipitation with Lime ZnSO₄ + CaO + $3H_2O = Zn(OH)_2 + CaSO_{4.2}H_2O$

The mixed hydroxide material is high quality and may be placed with an off-take/ refinery partner for final separation into pure nickel and cobalt and by-products.

Purified Hydroxide Results

A second method of nickel and cobalt recovery was tested as part of the pilot plant. In the second method, a purified nickel hydroxide was recovered using conventional solvent extraction removal of cobalt and zinc followed by precipitation of nickel hydroxide using magnesia. The reason for testing this second method was to allow for the option of producing a higher purity and higher value materials for off-take.

The solvent extraction system tested in the pilot plant used 5% Cyanex 272 in Orfom SX 80CT diluent. The circuit used 4 stages of extraction at pH's of 4.8, 5.1, 5.2 and 5.4. The loaded organic was then scrubbed in two stages with diluted zinc strip liquor to displace co-extracted magnesium.

The cobalt was then stripped in 3 stages at pH of 3.1, 3.2 and 3.4 with sulfuric acid solution. The zinc stripping was then performed in 2 stages at pH of 1.0 using sulfuric acid.

The solvent extraction circuit performed very well with an average of 97.1% cobalt removal and 100% zinc removal from the feed solution. Practically this means that the purified nickel hydroxide product (produced from the raffinate) should be very low in these key impurities.

The stripped cobalt and zinc can be neutralized with magnesium hydroxide to form separate hydroxide products. Alternatively, the zinc sulfate strip solution can be passed directly into the zinc sulfate chemical market.

Table 6 shows the analysis of the purified nickel hydroxide product recovered from the pilot plant. There was some cobalt and zinc contamination due to change over of the thickener from the "mixed" product to the purified product. The feed solution to the nickel hydroxide precipitation step (raffinate from Co/Zn SX) easily exceeded 1000:1 Ni/ Co ratio and hence cobalt content of the purified nickel hydroxide would be expected to decrease to less than 0.04% in practice. Given that zinc extraction is virtually 100% with the Cyanex 272 reagent, the zinc content in the purified product should also drop to close to zero content.

The cobalt removed by solvent extraction was precipitated using magnesia to form

Table 6. Analysis of the Purified Nickel Hydroxide Product from the Pilot Plant

a precipitate containing 35.1% cobalt, 0.42% zinc, 1.44% copper, 1.23% manganese, 0.016% nickel and 9.11% magnesium.

During the pilot plant study, the zinc was recovered as a separate solution. No attempt has been made to precipitate this zinc as a hydroxide as it is currently anticipated that the zinc solution will go directly to the zinc chemical market in an "over the fence" arrangement and railed by tank car to market.

MAGNESIUM REMOVAL

The NorthMet ore and concentrate contains some magnesium silicate mineralization. During high temperature autoclave leaching of the concentrate, some magnesium is leached. In addition, magnesia is used around the circuit for nickel and cobalt precipitation which also results in an increase in the magnesium content of recirculating solutions. In order to control the build-up of magnesium in solution, a magnesium removal step (with lime) was introduced into the circuit. Magnesium sulfate reacts to form magnesium hydroxide and gypsum with lime.

Magnesium Precipitation with Lime $MgSO_4 + CaO + 3H_2O = Mg(OH)_2 + CaSO_{4,2}H_2O$

The removal of magnesium is a control for the build-up of magnesium across the circuit. In the pilot plant approximately 50% of the magnesium was precipitated per "pass" with the balance of the magnesium allowed to recirculate in the overall circuit.

Moist. %	S.G .	Ni %	Co %	Cu %	Fe %	Zn %	AI %	Mg %	Ca %	Zi %	Mn %	S %	Cl g/t
43	2.47	43.7	0.37	0.01	0.09	0.17	0.03	1.91	0.04	0.21	0.01	2.53	1314

ENVIRONMENTAL TESTING AND SPECIALIST CONSULTANT TESTING

Barr Engineering of Minnesota, PolyMet's principal environmental review and permitting firm, was present throughout the pilot plant operation to collect environmental data for emission and waste characterization purposes. The pilot plant operation is essentially a scaled down version of the full scale plant under design and therefore represents an ideal opportunity to obtain key environmental data. Environmental data collected during piloting are being used by an independent contractor to prepare the mandated Environmental Impact Statement and to define methods of mitigation and environmental management.

Specialist consultants from reagent suppliers and solid-liquid separation vendors were also present to test applicability of reagents and measure properties of slurries for final design specification.

CONCLUSIONS

The NorthMet project of PolyMet Mining is well advanced in the Definitive Feasibility Study stage of project execution. The final feasibility document from Bateman Engineering is due in mid-2006.

The metallurgical approach to development of the NorthMet deposit is to float a bulk concentrate carrying the Cu-Ni-Co-Au-Pt-Pd values and then use the PLATSOL[™] process for the treatment of the concentrate. The PLATSOL[™] process will dissolve all the metals of value to high levels of extraction in the main autoclave, followed by a series of precipitation and SX/EW processes for recovery of final metal products.

The final products from the pilot plant will include:

- Copper Cathode of LME Grade A Quality;
- Au and PGM Precipitate for Toll Processing;
- Mixed Hydroxide Product Containing Ni-Co-Zn, or;
- Separate Purified Nickel and Cobalt Hydroxide Products and either a Zinc Hydroxide or a Zinc Strip Solution
- Synthetic Gypsum

The current design basis is for an initial 32,000 short tons of ore per day to be processed through the refurbished Cliff's Erie plant and a new hydrometallurgical refinery for metal extraction.

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13