

Solvent extraction design consideration for the Tati Activox® plant

by E. Robles*, I. Cronje*, and G. Nel†

Synopsis

The Tati Activox® Project (TA®P) is the first step towards the implementation of the Norilsk Nickel Activox® process. The ultra fine grinding (UFG) and autoclave pressure leach process conditions are the heart of the Activox® patent. Low grade base metal sulphide concentrates are leached in the process to recover the base metals and to produce LME grade nickel and copper cathodes and cobalt carbonates. The downstream processing of the metal-containing liquor produced by this process includes solvent extraction circuits to extract and concentrate metal-rich solutions, before the various final products are obtained.

This paper gives an overview of many of the relevant design aspects taken into consideration in the design of the Tati Activox® solvent extraction circuits. It addresses key design elements related to leach process conditions such as scaling, materials selection for corrosive solutions, pH control, crud formation and treatment, multiple SX trains designed to prevent organic cross-contamination, and design aspects related to the mitigation of fire risks.

Introduction

The Activox® process is a hydrometallurgical process route developed by Norilsk Process Technology to treat a wide variety of metal sulphide concentrates[1]. A combination of ultra fine grinding, which activates the sulphide minerals and oxidation at a relatively low temperature and pressure, form the core of the patented technology, which is expected to provide a competitive alternative to the traditional pyrometallurgical process routes. Work on the Tati Activox® Project (TA®P) started in 1998 with laboratory test work. The development towards commercialization has advanced systematically from laboratory-scale testing in Australia through various studies to the design, construction and successful operation of a 1:170 scale demonstration plant on the Phoenix mine site in the North Eastern district of Botswana, Southern Africa. In 2006 Hatch was awarded the contract for the execution of the Botswana Metal Refinery (BMR) for TA®P. This hydrometallurgical refinery is designed to produce 25 000 tpa nickel and 22 000 tpa copper as LME grade cathodes and 630 tpa cobalt as a cobalt carbonate.

The general flow sheet for the TA®P process is shown in Figure 1.

The Tati concentrate is trucked from the Tati Phoenix mine complex to the BMR where the concentrate is repulped in attritioners to 50% solids with recycled copper raffinate. The repulped concentrate is ground to 80% passing 10 microns using two-stage vertically stirred detritor ultra fine mills. The mill product is fed to two parallel autoclaves where the slurry reacts with oxygen at moderately high pressures (10 bar gauge) and moderate temperatures (105 degrees Celsius) to produce copper, cobalt and nickel sulphates in solution for downstream recovery. The pregnant leach solution (PLS) reports to a solid-liquid separation section, which includes a multi-stage countercurrent decantation (CCD) washing circuit where the leach residue solids are washed with recycled process water. Washed leach residue solids are pumped to the PGE recovery circuit. The overflow solution from the CCD train is clarified prior to reporting to a copper PLS pond.

Copper PLS is contacted with an organic solvent in the copper SX circuit that selectively extracts copper from the copper PLS solution. Copper is stripped from the organic using spent electrolyte from the copper EW circuit (Cu EW). The rich electrolyte is treated to remove trace organic before being pumped to the Cu EW circuit. The bulk of the copper SX raffinate is recirculated back to the concentrate repulp and the remainder is advanced to the cobalt and nickel recovery circuits. The Cu-rich electrolyte is sent to the Cu EW circuit to produce copper as LME grade copper cathode. Spent electrolyte is recirculated back to copper SX for enrichment via stripping of the copper-loaded organic phase.

* *Hatch Africa (Pty) Ltd, South Africa.*

† *Norilsk Nickel Africa (Pty) Ltd, South Africa.*

© *The Southern African Institute of Mining and Metallurgy, 2009.* *SA ISSN 0038–223X/3.00 + 0.00. This paper was first published at the SAIMM Conference,* Hydrometallurgy 2009, *24–26 February 2009.*

Solvent extraction design consideration for the Tati Activox® plant

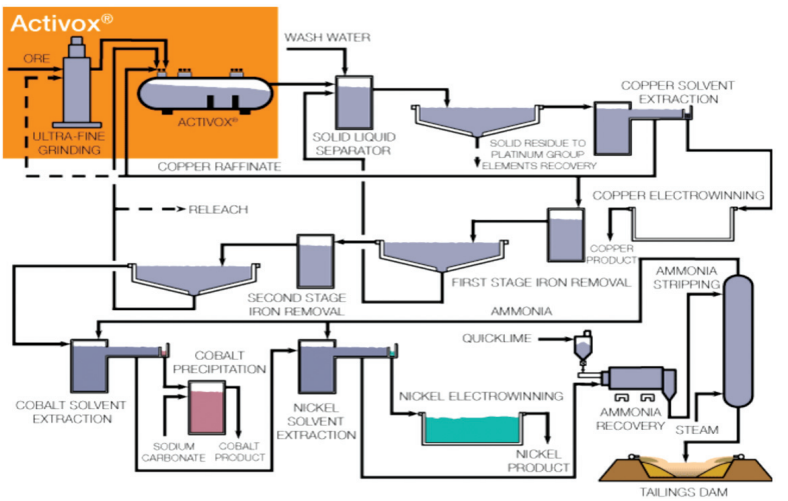


Figure 1—Tati Activox® flowsheet

Iron in the copper SX raffinate is removed to less than 10 ppm in solution using limestone, in a two-stage iron removal circuit. Cobalt PLS is contacted with an organic solvent that selectively extracts cobalt from solution. Cobalt is stripped from the loaded organic phase with an acidic strip solution and the resultant loaded strip liquor (LSL) is filtered and pumped to the cobalt precipitation circuit. Organic is removed from the cobalt SX raffinate before proceeding to the nickel PLS storage tank. Nickel PLS is contacted with an organic solvent that extracts the nickel (selectively over calcium and magnesium) from solution. Nickel is stripped from the loaded organic phase with spent nickel electrolyte recirculated from nickel electrowinning circuit (Ni EW). The rich electrolyte is treated to remove trace organic before reporting to the Ni EW cell house. The nickel SX raffinate stream is transferred to the ammonia recovery circuit.

The Ni EW process is designed to produce LME grade nickel cathode. Recovered spent electrolyte from Ni EW is treated to remove lead prior to being recirculated to nickel SX for stripping. Ammonia is recovered from the nickel SX raffinate stream for reuse as an aqueous ammonia solution for pH control in the cobalt and nickel SX processes. The ammonia is liberated from the nickel raffinate using the Norilsk patented ammonia recovery process. The process solution from ammonia recovery is recycled back to the CCD circuit and used as wash water. The residue from the CCD circuit is treated by flotation to recover the PGEs in the leach residue. Tails from the rougher circuit are neutralized together with other process effluent streams before reporting to the tailings dam.

Description of TA®P solvent extraction plants

In TA®P the process for leach liquor purification and concentration of metals consists of three SX circuits to remove Cu, Co and Ni in sequential stages by using different selective extractants.

Description of the SX circuits

Copper solvent extraction circuit (Cu SX)

Copper is selectively transferred from the relative low grade Cu PLS into a high-purity electrolyte stream using a copper selective extractant. The extractant employed is an oxime-based copper-selective chelating reagent that is dissolved in a high flashpoint hydrocarbon diluent to make up the organic phase.

The copper solvent extraction circuit consists of three main process steps:

- *Extraction*—copper PLS is contacted with the stripped organic phase in two extraction stages to transfer copper from the aqueous to the organic phase
- *Scrubbing*—the loaded organic is scrubbed with copper electrolyte bleed to remove co-extracted iron and aqueous-entrained chloride and manganese. Although highly selective for copper over most other cationic species under extraction conditions, a small quantity of Fe^{3+} is co-extracted. The scrubbing stage minimizes entrainment of aqueous in the loaded organic phase that otherwise will result in the undesirable build-up of species such as chloride and manganese in the electrolyte
- *Stripping*—the scrubbed organic phase is contacted with spent electrolyte from EW in two stages, utilizing the acid generated during the copper deposition process in Cu EW. Copper is transferred from the organic into an aqueous phase.

Cobalt solvent extraction circuit (Co SX)

Cobalt is recovered from the Co PLS following the iron removal process, using a solvent extraction circuit. The extractant employed is the phosphinic acid-based Cyanex 272, dissolved in a high flashpoint hydrocarbon diluent. This provides selective extraction for cobalt over nickel by controlling the pH in the extraction stages. Co-extraction of iron, manganese, zinc and copper also occurs.

The cobalt solvent extraction circuit consists of four main process steps:

- *Extraction*—Co PLS is contacted with stripped organic phase and cobalt is transferred from the aqueous to the organic phase in three stages. The pH is controlled by the addition of aqueous ammonia

- *Scrubbing*—loaded organic is washed with acidified, filtered water to remove co-extracted nickel from the organic phase in a single stage
- *Stripping*—scrubbed organic is contacted with acidified cobalt strip liquor and the cobalt is transferred from the organic to the aqueous phase in two stages
- *Cyanex recovery*—raffinate is washed with fresh diluent to minimize organic cross contamination to the nickel solvent extraction circuit in a single stage.

Nickel solvent extraction circuit (Ni SX)

Nickel is recovered from Co SX raffinate using solvent extraction. The extractant employed is the carboxylic-acid based Versatic 10 that is dissolved in a high flashpoint hydrocarbon diluent. By control of the extraction pH, the solvent-extraction process is selective for nickel over such species as calcium and magnesium.

The nickel solvent extraction circuit consists of the following four main process steps:

- *Extraction*—nickel pregnant leach solution (Ni PLS) is contacted with organic phase and nickel is transferred from the aqueous to the organic phase in five stages. The pH is controlled by the addition of aqueous ammonia
- *Versatic recovery*—prior to entering the extraction circuit the stripped organic phase is contacted with nickel raffinate exiting the extraction circuit in a single stage to recover aqueous-soluble Versatic 10 from the aqueous into the organic phase. This is achieved by reducing the pH with diluted sulphuric acid solution in this stage. Recovering the soluble Versatic 10 minimizes reagent losses and downstream frothing in the ammonia recovery circuit.
- *Scrubbing*—loaded organic is scrubbed with acidified nickel rich electrolyte bleed solution to remove co-extracted calcium from the organic phase in a single stage.
- *Stripping*—scrubbed organic is contacted with spent electrolyte from Ni EW in two stages and nickel is transferred from the organic to the aqueous phase. Most of the sulphuric acid utilized in stripping is generated in the electrowinning process.

Contactor equipment

The Tati Activox® SX plant design is based on the use of mixer-settlers as contactor equipment. It makes use of the countercurrent Side-Feed™ mixer-settler concept of Miller Metallurgical Services (MMS)[2]. Each mixer-settler consists of a two-stage mixer and a settler. A dual mixing system provides high overall stage efficiency by minimizing back-mixing, minimizing short circuiting, and by ensuring sufficient retention time in the mixers. The primary mixing vessel is fitted with a pump-mix type of impeller, which provides the head for the inter-stage pumping of solutions as well as mixing of the two phases. The primary mixing vessel incorporates a false bottom into which the organic, aqueous and recycle streams are introduced. Mixed phases enter the secondary mixing vessel via a connecting pipe. This vessel is fitted with a mixing impeller designed for maximum mixing efficiency with minimum shear. The dispersion overflows into a shallow gravity settler where sufficient area is provided for disengagement of the aqueous and organic phases. Cross-sectional picket fences maintain a band of dispersed phase through which finer droplet coalescence is promoted. A weir arrangement at the discharge end of the settler allows for separate discharge of the organic and aqueous phases. The aqueous/organic interface level in the settler is set during commissioning by manual adjustment of the physical height of the aqueous overflow weir.

The design allows all the mixer-settlers to run with organic or aqueous continuity by adjusting the internal recycles to the primary mixer from the settler. The general philosophy followed in all three circuits is that the extraction stages be operated in organic-continuous mode. This minimizes the entrained organic loss in the raffinate and assists in the compaction of crud solids. Organic continuity in the first extraction stage could result in high aqueous entrainment to the loaded organic phase. The deleterious effects of this are curtailed to some extent by the inclusion of the loaded-organic scrub stage that is present at all the circuits.

Configuration of the TA®P solvent extraction circuits

The Table I below shows a comparison between the three SX circuits for TA®P. Parameters compared are type of extractant and diluent, number of mixer-settlers per process step, existence of pH control and configuration for organic removal and recovery.

The number of extraction stages differ from circuit to circuit depending on the extraction time, type of extractant employed and the concentration of the metal present in the PLS. The propensity for scaling in the circuit may require the addition of an extra extraction stage as in the case of the nickel circuit. The likelihood of downtime is reduced by the addition of an extra extraction stage, which allows extraction operations to carry on as planned while cleaning can be carried out simultaneously.

Stripping in all three circuits comprise two stages where loaded organic is contacted with spent electrolyte or acidic strip liquor. As for extraction, the aqueous and organic phases flow countercurrently with loaded organic entering the first stripping stage and spent electrolyte or strip liquor entering the second stripping stage. The aqueous solution exiting the first stripping stage is enriched in metal, and after organic removal, becomes the feed to the metal winning circuit that follows.

Sufficient surge capacity is provided upstream of the SX circuits. In the case of copper, four-day surge capacity is provided in the PLS storage pond. This pond features two presettlement compartments to minimize solids ingress to the solvent extraction process. The Co PLS pond has two days of surge capacity. Cobalt raffinate is stored in a four-hour surge tank with sufficient capacity to buffer against operational surges upstream of Ni SX. All three SX circuits can therefore be operated independently without being affected by upstream bottlenecks. This provides the system with flexibility and continuity avoiding unnecessary shutdowns and start-ups. The surge capacities between process steps were sized based on a dynamic model.

Table I

Comparison of the TA®P SX circuits

Copper SX	Cobalt SX	Nickel SX	
Extractant	LIX 984N	Cyanex 272	Versatic 10
Extractant family	Oxime based	Phosphinic acid	Carboxylic acid
Diluent	Sasol SSX 210	Sasol SSX 210	Sasol SSX 210
Total flow rate per settler	1002 m^3/h	309 m^3/h	383 m^3/h
Operating temperature	30–40°C	40°C	40°C
PLS feed storage	Pond (4 days)	Pond (2 days)	Tank (4 hours)
No. of extraction mixer-settlers	2	3	5 (plus one spare stage for potential descaling of stage 1 or 2)
No. of scrub mixer-settlers	1	1	1
No. of stripping mixer-settlers	2	2	2
Organic recovery stage	No	Yes	Yes
pH control in extraction stages	No	Yes (ammonium hydroxide)	Yes (ammonium hydroxide)
pH control in scrub stage	No	Yes (sulphuric acid)	Yes (sulphuric acid)
pH control in stripping stages	No	Yes (sulphuric acid)	Yes (sulphuric acid)
pH control in organic recovery	No	No	Yes (sulphuric acid)
Potential scaling	No	Yes	Yes
Raffinate after-settler	Yes	Yes	Yes
Rich electrolyte/LSL after settler	No	No	Yes
Raffinate coalescing filters	No	Yes	No
Electrolyte/LSL coalescing filters	Yes	Yes	Yes
Electrolyte/LSL carbon columns	No	No	Yes
Crud treatment system	Tri-phase centrifuge	Tri-phase centrifuge	Tri-phase centrifuge

In all three circuits loaded organic flows via gravity from the respective extraction stages into a series of two organic surge tanks. Both tanks are equipped with internal weirs for the separation of entrained aqueous. Loaded organic is pumped to a single scrub stage where it is contacted with an acidified aqueous solution. The scrub raffinate flows by gravity into the raffinate after settler to minimize the entrainment of organic in the aqueous phase.

The other significant difference between these SX circuits is the strategy to remove or recover entrained organic from the aqueous products (raffinate and rich electrolyte/loaded strip liquor). The strategy for organic removal or recovery is essentially dictated by the requirements of the downstream process stage, as explained below.

For TA®P it is very important to minimize the loss of extractant from the circuit, to reduce the impact on the downstream operations. Also, organic extractant entrainment losses must be minimized from an operating-cost perspective. The Cu raffinate reports to an after settler followed by the iron removal process, which forms a buffer between Cu SX and Co SX. The Co raffinate stream first passes through an after settler, then a diluent wash stage and finally mixed-media coalescing filters before advancing to Ni SX. In the nickel circuit, the aqueous solubility of Versatic 10 increases significantly with an increase in pH. Acidification of the raffinate is required to recover the Versatic from the aqueous phase. This is achieved in a single stage where raffinate is contacted with stripped organic phase at a reduced pH ensuring that soluble Versatic is recovered into the organic phase. Similarly to Cu SX and Co SX, the raffinate is then transferred to an after settler.

Similarly to the raffinate, the rich electrolyte or loaded strip liquor needs to be treated to remove or recover entrained organic. The organic removal strategy for both copper rich electrolyte and cobalt loaded strip liquor is similar. In these two cases the metal-rich liquor is advanced to a series of mixed media coalescing filters.

The nickel EW process is particularly sensitive to the presence of SX organic phase components. Before advancing to the EW circuit the Ni rich electrolyte undergoes two organic removal steps. The first is a system of mixed media coalescing filters where the bulk of the entrained organic phase is removed. The solution exiting this stage is passed through activated carbon absorption columns to further reduce the organic content.

Crud that collects in the settlers is removed on a regular basis by suction with a peristaltic pump from just ahead of the overflow weir via a manifold collection system into the crud holding tank. The emulsion is fed into a tri-phase centrifuge that separates the solution phases (organic and aqueous) for recycle to the process and discharges a solid for disposal.

Removal of degradation products and solids from the organic phase is achieved in a similar way in the copper, cobalt and nickel SX circuits. An agitated crud treatment tank is provided where a portion of the organic phase is contacted with diatomaceous earth in the case of cobalt and nickel, while clay for the copper circuit to help remove solids and degraded organic products.

On each SX plant a remote emergency pond can receive overflows from all the sumps located in the bunded areas for the SX process and its associated tank farm.

Design issues related to TA®P process conditions

The conditions of the leaching process that are dictated by the type of ore to be processed, the nature of the leach reagents,

and the type and sequence of processes used to obtain the metal products, have an important effect in the design of the multiple SX circuits. In TA®P the most important design issues in the SX circuits that are related to the leach process conditions and operating parameters are scaling, the presence of a highly corrosive environment, and crud formation and control.

Scaling

The Tati concentrate is primarily a pyrrhotite (iron sulphide) type ore with some calcium, magnesium and manganese minerals. The dissolved iron is removed in a two-stage atmospheric precipitation process using limestone. Ground limestone slurry is introduced into the reaction tanks to increase the pH to precipitate ferric hydroxide. A small amount of nickel and cobalt will co-precipitate at the operating pH.

The most common cause of scaling in SX is the saturation of $CaSO_4.2H_2O$ due to the extraction and loading of calcium in the last few extraction stages and stripping of the calcium off the organic into the aqueous phase in the first extraction stages. Excessive scaling results in blocked pipes and reduced processing capacity. This in turn may lead to reduced production capacity, increased energy requirements (e.g. pumping and mixing) and increased downtime and maintenance cost.

The approach for the design of the areas most likely to be affected by scaling has been to make adjustment in the process parameters and to take special care to design details that could decrease downtime and maintenance cost.

Adjustment of process parameters

The TA®P design has catered for the dilution of the Co PLS liquor with 5% filtered raw water to maintain the calcium concentration in the Co PLS below Ca saturation. Although this reduces the risk of scaling, the increased flow rate requires larger processing capacity with incremental capital cost.

Solution temperature is also an important parameter that affects the scaling rate. The temperature in Co SX is maintained around the range of temperature that allows the maximum solubility of calcium sulphate. The change of pH of the solutions feeding Co SX and Ni SX, which is essentially dictated by the needs of the selective extraction of these metals, is controlled to minimize co-extraction of Ca in the back end of extraction and stripping in the front end of the extraction stages.

Piping design

Conductive fibre reinforced plastic (FRP) piping materials are selected for the TA®P SX plants to prevent static electricity build-up in the pipes. FRP piping also minimizes the adhesion of scale to the material surfaces, due to its smoother surface finish compared to stainless steel piping. The pipe spools were designed for easy removal and descaling. The plant layout was optimized to improve access to the pipes for periodical descaling.

Design for easy cleaning and maintenance

The Ni SX plant, which is particularly prone to scaling, is designed for regular descaling. One extraction mixer-settler is included as a spare unit with dedicated piping to allow descaling of any of the first two extraction mixer-settlers without operation interruption. The SX train layout is optimized to improve access to mixer tanks, settler and pipes and facilitate the removal of internal parts and pipe spools. The mixer tanks have removable lids and the settlers' roofs are designed with removable sections that include inspection hatches to monitor the accumulation of gypsum in the settler internals. Special nozzles were included in the mixer tanks to facilitate hydro-blasting of scale from tank internals.

Use of synergists

The use of synergists to improve the selectivity of certain metals is a relatively novel idea not widely implemented in the industry. A study conducted by du Preez *et al.*[3] which evaluates the use of a synergist system for nickel-calcium separation on Tati Demonstration Plant solutions shows that selectivity for nickel over calcium can be improved by adding a synergist to the Ni SX organic. This performance versus the increased operating cost has to be weighed up to establish the overall added value of the synergist.

Corrosive solutions

The TA®P design relies on the addition of chlorides to improve copper extraction in the autoclave. This creates a corrosive leach solution requiring the use of higher grade metallic materials for certain specified applications. Based on the experience at the Tati Demonstration Plant, duplex stainless steel SAF 2205 was selected for metal components in contact with all process liquors that contain chlorides. For the SX circuits, where the working temperature is below 45°C, the TA®P has chosen to use FRP as an alternative to SAF 2205, as the maximum temperature recommended for the use of FRP is 60°C.

Crud formation and control

In SX the formation of crud is generally caused by a variety of substances present in the solutions being processed. This includes fines from ultra fine milling, entrained solids/precipitates from leaching; foreign material such as dust, insects; or silica in the TA®P filtered water.

The strategy for crud control and treatment for the TA®P includes the following:

- *PLS clarification*—pin bed clarifiers are used in the TA®P design to reduce the solids content of the Cu PLS and Co PLS. The uncontrolled separation of solids from the process liquor is usually a significant contributor to crud formation
- *Settling ponds prior to SX*—settling ponds provide sufficient surface area and reduce flow rates so that ultra fine material has an opportunity to settle out prior to SX
- *Removal of degradation products*—degradation products are produced by reactions on the organic phase caused by biological, chemical and environmental factors. These degradation products tend to be surface active and promote crud formation. Degradation products are continually removed in the Cu SX circuit by clay treatment and in Ni SX by the use of carbon columns

- *Isolation of SX mixer-settlers from external environment*—roofing of the SX mixer-settlers, which provides isolation from the external environment significantly reduced the collection of dust and insects
- *Water purification*—water treatment and purification is used in the TA®P design to minimize silica concentration in the plant filtered water that is used as SX dilution water.
- *Phase continuity*—the TA®P design utilizes preferably organic phase continuity to promote compaction of crud solids
- *pH control*—the TA®P design optimizes pH control to prevent saturation of calcium in Ni SX and subsequent crud formation
- *Crud treatment*—the TA®P design includes the installation of a permanent crud collection manifold system, which removes the crud that accumulates in the settlers, using a peristaltic pump for crud suction. The crud collection manifold consists of short sections of perforated pipes, located adjacent to the organic weir, that are connected to a header. The collected crud is treated by means of a tri-phase decanter centrifuge for organic recovery. Each SX circuit has its own crud treatment system to prevent organic cross-contamination.

pH control

The TA®P design requires very tight pH control in Co SX and Ni SX to prevent co-extraction of metals. Impurities are scrubbed of the loaded organic using acidified scrub liquor in the scrubbed organic phase.

The most important challenge for a pH control system is the reliability of the pH measurement in the solution feeding the settler. Such a measurement could be hampered by organic coating or scaling of the pH electrode probe. Hence, in theTA®P design the electrode is located in a dedicated pH pot that receives aqueous solution from the feed end of the settler, through a pipe that connects the lower part of the settler's sidewall with the pot. This location allows pH measurement in clean aqueous solution and facilitates access for cleaning and calibration of the electrode.

The TA®P design allowed for a cascading control system where the flow rate of neutralizing or acidification reagent is the primary control measure and the pH of the aqueous solution is the secondary measure. The reagent flow set point is a calculated ratio of the main incoming flow (PLS, strip liquor or loaded organic) with a multiplier that is based on the output provided by the pH controller. The control logic minimizes reagent flowrate fluctuations and reliability on accurate pH measurements. The control logic allows for timely identification of pH deviations. Low and high alarms alert the operator to pH measurement faults or dosing ratios outside the set ranges.

The pH electrodes in the first three extraction stages of Ni SX were duplicated, with the additional electrodes located in the aqueous launders of these settlers to provide back-up for the pH control loops.

Design issues related to multiple SX trains

The multiple SX trains in the TA®P design present certain challenges in terms of optimizing plant layout, contactor design and minimizing possible organic cross-contamination. Organic cross-contamination occurs when organic from one SX circuit is carried over to the next SX circuit causing process upsets, reduction of efficiency, metal loss, and off spec product.

Plant layout

One of the major challenges that the SX design for the TA®P faced was to accommodate the various SX circuits in an optimal manner. The following had to be taken into account: minimization of the SX train footprint; location of storage ponds and/or connecting pipelines with upstream and downstream process facilities; and safe separation distance between SX circuits to minimize fire risks.

Norilsk selected the side-feed design of MMS for the SX settlers. This allows a smaller footprint for the SX train of mixer-settlers and better access for operational control, cleaning and maintenance. All operational and maintenance work can be carried out on one side of the settler.

Each SX plant is laid out with all equipment containing large quantities of organic solution grouped together in the SX area (SX train, organic storage tanks, after-settler(s), crud collection tank) and associated equipment including organic recovery and crud treatment systems are grouped into the SX tank farm area. A safe separation distance is allowed between these two areas to ensure asset protection in the case of a fire. For the same reason, the three SX trains are separated by a safe distance. These distances were defined after a fire plume analysis was performed for each SX plant.

Optimization of flow patterns inside the SX settler

The optimal design of the main phase separation equipment in the SX plants is critically important since excessive turbulence and insufficient coalescence and phase separation result in increased entrainment that may lead to cross-contamination. Hatch used computational fluid dynamics (CFD) models for settler optimization in the TA®P. CFD is an important design tool that allows the designer to visualize and test innovative design concepts quickly and economically[4–7].

Single and multi-phase CFD models were used for the TA®P to predict the flow behaviour and flow distribution in the entrance and early flow development regions of the side-feed settler vessels. The crossover launder that connects the secondary mixer tank with the settler and its guide vanes were modified to provide an optimum low-speed transition into the settler vessel, which is critical to its overall performance. Multiphase modelling has also provided significant guidance in the design of the TA®P mixer interconnecting pipe, picket fences and collection launders.

In reality all settlers deviate from an ideal 'plug-flow' distribution and results from visual inspection or physical modelling can be difficult to interpret and are therefore of limited use in design. Computation of the integrated volumetric-flow distributions across the width of the settler, as shown in Figure 2, has been used to evaluate and rank a number of different designs. The settler internals were modified to achieve a uniformly distributed flow across the width of the settler.

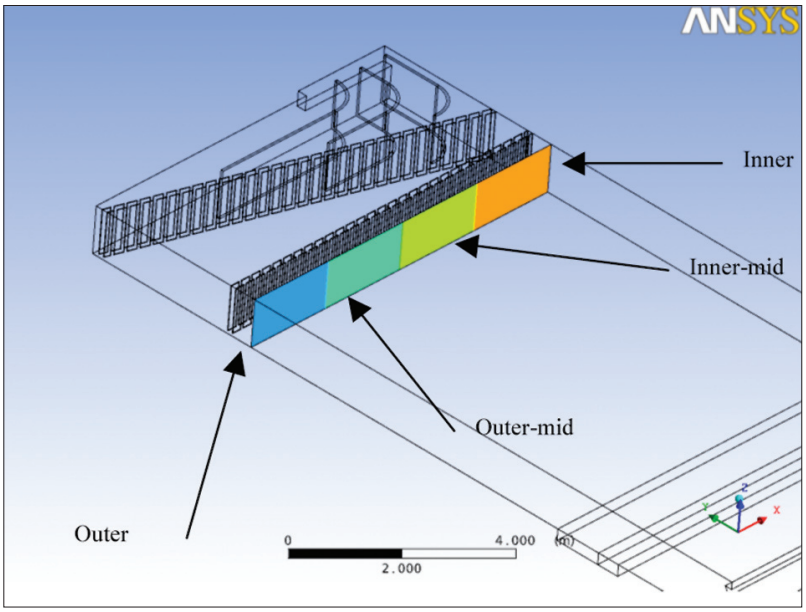


Figure 2—Post processing is used to evaluate the mass flow across each quarter of the width

Table II

Distribution of mass flow across the width of the settler for the original design and after using CFD analysis

	Integrated mass flow through lateral quarters of the settler cross-section (kg/s)			
	Inner	Inner-mid	Outer-mid	Outer
Original design	106	46	66	105
Optimized design	91	92	90	87

The computed mass flow rates through each of the lateral quarters of the settler for the original design and after using CFD analysis are listed in Table II. The optimized design provided a significantly more uniform flow distribution with a lower overall pressure drop.

Organic removal equipment

The TA®P design defined various strategies and equipment for the removal of organic phase from aqueous streams. These include physical and chemical processes depending on the state of the organic for recovery (dissolved and/or entrained). Organic recovery processes fall into four process categories:

- *Primary*—reducing organic entrainment from >200 ppm or more down to <50 ppm and recovering the organic phase
- *Secondary*—reducing organic entrainment from 100 ppm to <2 ppm and recovering the organic phase
- *Dissolved organic recovery*—reducing the soluble organic content from >200 ppm to <10 ppm
- *Tertiary*—reducing the total organic content, both entrained and soluble, from 10 ppm to <1 ppm.

Primary organic removal equipment

After-settlers are utilized in the three SX circuits as the preferred primary organic removal equipment in the TA®P design. Essentially they provide additional area for phase separation and are the first barrier to stop organic entrainment peaks should a major upset happen in the mixer-settlers. The organic collected in the after-settlers is periodically removed and returned to the extraction stages via the corresponding crud treatment system. The only exception to this is in the Co loaded strip liquor stream, where this aqueous stream is passed though a tank with coalescing media before downstream processing. The coalescing media generate large organic droplets from small entrained droplets by creating random collision opportunities. Larger droplets are separated more readily. The coalesced organic is periodically removed from the tank and returned to the extraction stages via the Co SX crud treatment system.

Non-mechanically agitated flotation cells, which are also widely used as primary organic removal equipment, were not included in the design because of the induction of high volumes of air (at ratios of about 0.8:1 v/v) may result in evaporative loss of significant proportions of the valuable organic phase or cause other process problems including oxidation.

Secondary organic removal equipment

As a secondary organic removal equipment downstream of the after-settlers, the TA®P design allowed for pressure

filtration using dual-media filters, with a combination of anthracite and garnet media, to reduce entrained organic level to <2 ppm. Organic removed from the filters is returned to the extraction stages via the corresponding crud treatment system.

The only two product streams that do not have downstream processing by pressure filtration are the raffinate streams in Cu SX and Ni SX. As they are fed into storage ponds, floating organic is removed from the ponds' surfaces using rope mop-type organic skimmers. Organic removed from the ponds is also returned to the extraction stages via the corresponding crud treatment system.

Soluble organic recovery

Highly soluble extractants need to be recovered through physicochemical and chemical means as opposed to simple physical removal. The TA®P design has two methods for bulk removal of soluble organic from the aqueous phase, depending on the nature of the extractant. One is for removal of phosphinic acid-based extractant solubilized in Co SX raffinate that is simply washed with diluent in a single stage. On the other hand, carboxylic acid-based extractant solubilized in Ni SX raffinate has to be recovered through pH adjustment with an acid solution in a single stage to reduce organic solubility in the aqueous phase[8].

Tertiary organic removal equipment

In the TA®P design, tertiary recovery equipment is employed to ensure the achievement of <1 ppm organic in the rich electrolyte going to Ni EW. The equipment selected is a package of columns filled with granular activated carbon.

The carbon columns package consists of three columns, which are connected as a lead-lag system. At any given time two of the columns are in service and one is out of service. Feed electrolyte flows into the lead column, from there to the lag column, and then leaves the system. When the granular activated carbon media in the lead column is saturated with organics, that column is taken out of service. The lag column then takes lead status and the previously out-of-service column takes lag status. The process is sequenced so that over a given period each of the columns will have been in lead status, followed by being out of service, followed by being in lag status.

Mitigation of fire risks

Recent fires in SX plants have highlighted the importance of safety in design in order to eliminate or mitigate the risk of fire and subsequent damage to facilities, injuries and fatalities. Although SX plants normally operate below the flashpoint of the organic solvent rendering the environment inherently safe, the creation of aerosols or static build-up create conditions suitable for ignition by an electrostatic charge[9].

The TA®P SX plants are designed to reduce the risk of fire initiation with the moderate operating temperature in SX, the minimization of aerosols formation in all the feeds to tanks and launders and the prevention of static charge build-up by the use of conductive FRP for all the piping systems and tanks and appropriate earthing devices along the piping systems and on tanks. These plants are also designed under an integrated fire management strategy that allows early and reliable fire detection, effective fire suppression, and fire containment and isolation.

Fire detection and alarm

The fire detection system is designed to be reliable, robust and fast. This system has detection in multiple points to prevent false alarms and subsequent damage caused by the unnecessary release of water and or foam into the processing units. The fire detectors in the TA®P SX plants, which are of the infrared flame type, are placed under the removable settler roofs, across groups of mixer-settlers and storage tanks and along pipe routes.

The purpose of the alarm system is to provide an audible warning that enables safe and timely evacuation of the plant. Manual call points and evacuation speakers are positioned around the SX areas. Fire alarm panels located in the field centralize monitoring of all the devices, which form part of the fire detection and alarm system of the area and are connected to the main fire alarm panel in the central control room.

Fire suppression system

Two options of fire suppression were analysed for the TA®P design: a water deluge system with dumping of the settler content during fire and a fixed foam-water deluge system. The two options were assessed based on minimum statutory requirements, international recognized codes and standards, insurance requirements, best industry practice, occupant life safety, asset protection, potential business interruption, site operational procedures, and capital cost.

The initial design for the fire suppression system was a water deluge with a dump system. This system was designed to prevent escalation of fire by cooling the equipment and structures adjacent to a fire to protect them from heat radiation. The system included the automatic water deluge on tank externals, structures, pipe racks and bunds and manual water hydrants along plant perimeter. The dump system was designed to allow the dumping of the contents of the mixer-settler on fire by means of actuated valves, which are activated by the fire detection and alarm systems. Sumps within the SX areas were also designed in such a way to drain the area fast and effectively to the remote fire dump pond.

This water deluge system was found to be insufficient after evaluating against the design requirements. Four main drawbacks were identified in the system. Firstly, the need to extinguish a fire in the SX mixer settler as quickly as possible was not met by this design since deluge water does not offer the extinguishing properties that foam does. Secondly, the location of the TA®P is fairly remote and the availability of trained fire crews are limited. Thirdly, the need to minimize loss of plant production leading to minimizing downtime and minimizing damage to equipment, highlighted the need for a more robust fire suppression system with lower water demand. The final drawback was the reliability of the dumping system. Regular testing of the dump valves was required to maintain the integrity of the system. False alarms would have led to the dumping of the SX inventory.

The water deluge system was then replaced by a fixed automatic foam-water deluge system. The fixed foam system consists of permanently installed piping and deluge/foam sprays tied into a fire water supply and dedicated foam concentrate supply and proportioning equipment. The deluge system provides external protection to vessels, tanks and pumps, located in each SX plant area and in the diluent storage area. Manual yard hydrants/monitors with foam making equipment are placed along the plant perimeter. The spray foam/deluge system is activated with a pilot operated water fusible plugs network. Special dedicated foam lines are provided for the internal organic tank/vessel protection. These lines are manually operated. The SX plants are divided into three to five areas with separately activated deluge foam systems. All deluge-foam nozzles are located outside the mixer settler tanks to prevent process contamination.

The foam recommended for use on the TA®P design is a low viscosity AFFF/A4P 3%. This conventional foam is suitable for extinguishing hydrocarbon liquid and polar fire.

Fire containment and isolation

A fire plume analysis was used as basis for the layout of the TA®P SX areas. The analysis takes into account the area of the fire, the volume and type of fuel that could burn, climatic and wind conditions, and the presence of other assets and public access. From the fire plume analysis, the physical layout of the assets within each SX plant is arranged in such a way to reduce impact of fire escalation and increase ease of isolation and containment. This analysis also provides information to arrange each SX plant in relation to the others.

Each SX area containing all mixer-settlers, after-settlers and crud collection and organic tanks, is separated into individual fire suppression zones. These zones facilitate fire containment within the SX area.

On each one of these zones there is a bund with a sump that allows fluids to overflow into a remote emergency pond to prevent foam/water or process liquor spillage spreading to other zones during a fire event. Fire retardant, conductive FRP piping were used inside the SX plants to prevent the spreading of a SX fire through the piping.

Conclusions

The three SX circuits of the TA®P were designed with careful consideration to the characteristics of the leach solution, the operation of multiple SX circuits in sequential mode, and the mitigation of fire risks.

The TA®P design utilizes several process steps (clarifiers, settling ponds, filters) to minimize solids entrainment into the PLS.

The design allows for a full standby mixer settler, PLS dilution and tight pH control to minimize scaling during operation.

Primary, secondary and tertiary organic removal steps are utilized to prevent organic cross contamination and rich electrolyte contamination.

A foam-water deluge system was selected for all three TA®P SX plants with emergency ponds to collect overflow form the SX bunded areas.

Acknowledgements

The authors would like to thank Norilsk Nickel Africa (Pty) Ltd. and Hatch Africa (Pty) Ltd. for their permission and support in writing this paper. We also like to acknowledge the consultancy work provided by Graeme Miller and the good work of the multidiscipline SX design team that worked out of Hatch's Brisbane office.

References

1. Palmer, C.M. and Johnson, G.D. The Activox® Process: Growing significance in the nickel industry. *Journal of Minerals, Metals and Materials Society*, vol. 57, no. 7, July 2005, pp. 40–47.

2. Miller, G. Design of Mixer Settlers to Maximise Performance, Proceedings from ALTA 2006 Copper Conference, *10th Copper Event*, ALTA Metallurgical Services, Melbourne, Australia.

3. du Preez, R., Nel, G., Kotze, M., Donegan, S., and Massina, H. Solvent Extraction test work to evaluate a Versatic 10/Nicksyn™ Synergistic system for Nickel-Calcium separation, *Proceedings of The Fourth South African Base Metals Conference, Africa's base metals resurgence*. Symposium Series S47, South African Institute of Mining and Metallurgy, 2007, pp. 193–210.

4. Gunnewiek, L.H., Oshinowo, L., Plikas, T., and Haywood, R. Using CFD in the Design and Scale-up of Hydrometallurgical Processes, *3rd International Conference on CFD in the Minerals and Process Industries*, CSIRO, Melbourne, Australia, 2003.

5. Schwarz, M.P., Lane, G., Nikolov, J., Yang, W., Bujalski, J.M., Solnordal, C.B., and Houchin, M. Development of a Design Capability for SX Contactors Using Multi-Phase CFD and Experimental Modelling, *Proceedings ALTA 2007 Copper Conference, 11th Copper Event*, ALTA Metallurgical Services, Melbourne, Australia.

6. Robles, E., Cronje, I., Poulter, S., and Haywood, R. Aspects of the Design of SX Plants for the Modern Hydrometallurgical Refinery, *Proceedings of the International Solvent Extraction Conference ISEC 2008*, Tucson, Arizona.

7. Giralico, M., Gigas, B., and Preston, M. Advanced Mixer Settler Designs that will Optimize Tomorrow's Large Flow Production Requirements, *Proceedings Hydrometallurgy 2003—Fifth International Conference in Honour of Professor Ian Ritchie*, vol. 1, Leaching and Solution Purification. TMS, 2003.

8. Bacon, G. and Mihaylov, I. Solvent Extraction as an Enabling Technology in the Nickel Industry, *Proceedings of the International Solvent Extraction Conference ISEC 2002*, K.C. Sole, P.M. Cole, J.S. Preston, and D.J. Robinson, (eds.), South African Institute of Mining and Metallurgy, Johannesburg. pp. 853–863.

9. Haig, P., Koenen, T., and Maxwell, J. Electrostatic Hazards in Solvent Extraction Plants, *ALTA SX/EW World Summit*, Perth, ALTA Metallurgical Services, Melbourne, Australia, 2003. ◆