



Pilot-plant solvent extraction of cobalt and nickel for Avmin's Nkomati project

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Synopsis

Avmin have developed a flowsheet for the refining of copper, nickel and cobalt from Nkomati concentrates. Cobalt is recovered from calcium-saturated solution using Cyanex® 272. Nickel is subsequently extracted using versatic acid. The loaded organic is stripped using spent electrolyte, producing advance electrolyte for recovery of nickel by electrowinning.

Cobalt was recovered with >99.5% extraction efficiency, reducing cobalt from 1.8 g/l to <10 mg/l. The cobalt/nickel ratio in the cobalt product solution was >1500. Nickel solvent extraction was optimized to recover 99%, reducing the nickel concentration from 32 g/l to <0.3 g/l, while minimizing calcium department to the nickel electrowinning circuit.

Process description

In the flowsheet developed by Avmin for the Nkomati base metal refinery, copper, nickel, and cobalt from a sulphide flotation concentrate are dissolved using fine grinding, followed by pressure leaching. Copper is recovered from the leach liquor by solvent extraction (SX). A bleed stream of the copper SX raffinate is treated to remove iron by oxidation/precipitation, and then to recover cobalt and nickel in sequential SX unit operations.

After precipitation of iron from the copper SX raffinate, cobalt is recovered from solution using a phosphinic acid extractant, Cyanex® 272. The cobalt concentration in the nickel solution is reduced to enable the cobalt specification in the downstream nickel electrowinning (EW) to be met. Co-extracted nickel, calcium, and magnesium are scrubbed from the loaded organic phase using a portion of the loaded strip liquor (cobalt concentration of approximately 25 g/l). The scrubbed organic phase is stripped with dilute sulphuric acid to produce a cobalt sulphate solution. The form of the final cobalt product has yet to be decided.

Nickel is extracted from the calcium-saturated solution by SX using versatic acid. Co-extracted calcium and magnesium are

scrubbed from the loaded organic phase using a portion of the nickel loaded strip liquor diluted with water to a nickel concentration of 3 g/l and a pH of 5.6. The scrubbed organic phase is stripped with spent electrolyte from the nickel EW operation.

Both SX plants were operated at ambient temperature as this was considered to be a 'worst case scenario' in terms of settling characteristics, reaction kinetics, and nickel ammonium sulphate precipitation. The circuit was continuously run for 400 hours. Nickel was recovered from the advance electrolyte by standard divided-cell EW technology. Figure 1 shows the overall process flowsheet.

Equipment and procedures

Conventional mixer-settler units, each with a mixer volume of 500 ml and settling area of 256 cm², were operated in a countercurrent configuration. Solutions were pumped into the plant using peristaltic pumps. When required, stages were operated with internal recycle to maintain aqueous phase continuity in the mixers. Adjustment of pH in both SX circuits was achieved by the addition of 400 g/l NH₄OH or 300 g/l H₂SO₄ solution directly into the mixers. The NH₄H concentration was chosen based on Avmin's estimate of the concentration attainable from the lime boil ammonia recovery planned for the full-scale circuit. Control of pH in each stage was by the use of an ABB Commander 300 PID controller linked to a peristaltic pump and Foxboro pH meter and probe.

Cyanex 272 was supplied by Cytec, Canada, and versatic acid by Shell, South

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© The South African Institute of Mining and Metallurgy, 2002. SA ISSN 0038-223X/3.00 + 0.00. This paper was first presented at: ISEC 2002, International Solvent Extraction Conference, Cape Town, South Africa, 17-21 Mar. 2002.

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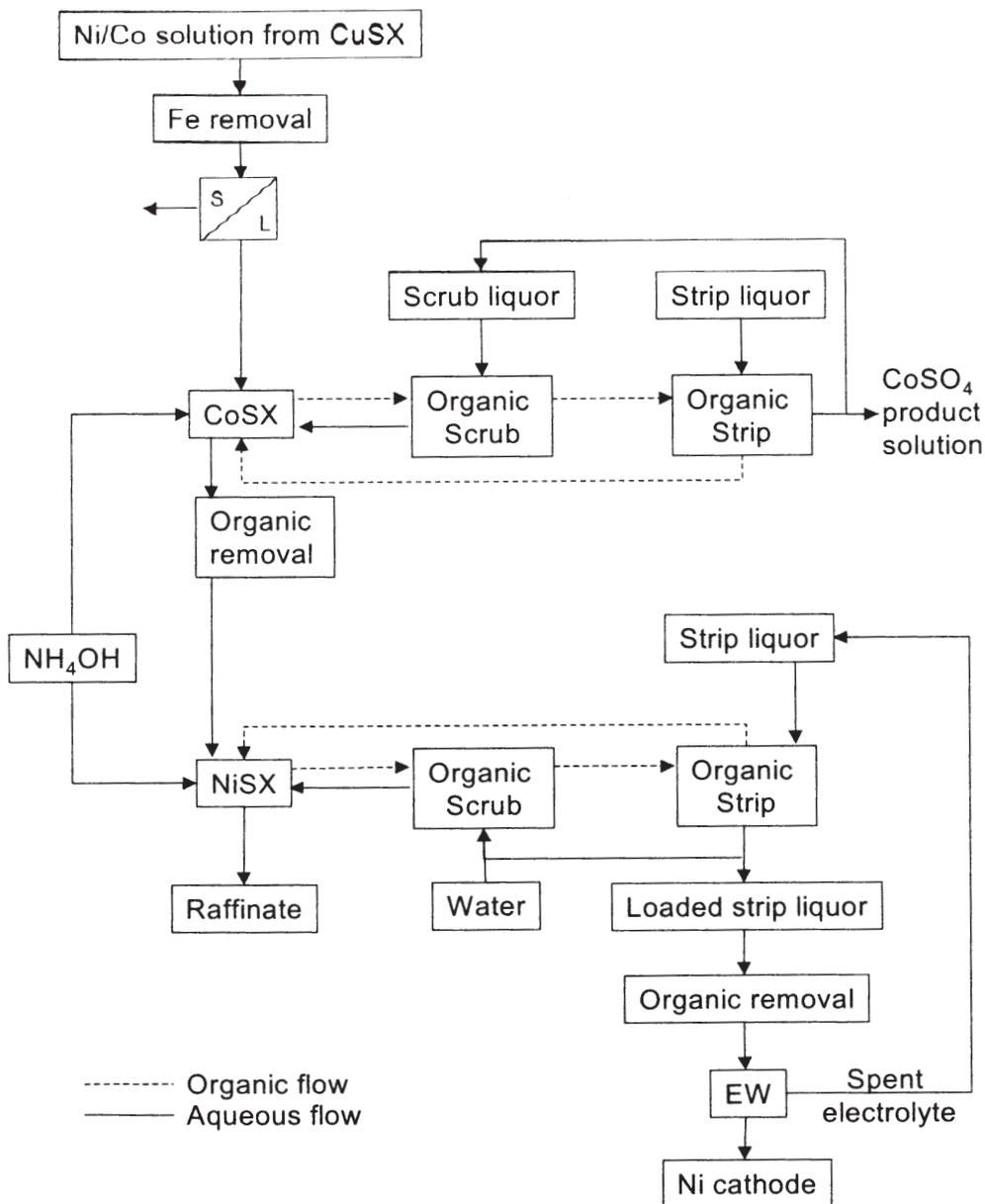


Figure 1—Process flowsheet for treatment of Nkomati copper SX raffinate

Africa. Both circuits used C₁₂-C₁₃ *n*-paraffin (SasolChem, South Africa), an aliphatic hydrocarbon diluent.

Cobalt solvent extraction

The feed solution to the cobalt SX was synthetically made up to represent Avmin's estimate of the cobalt SX feed. After 7 days operation, this was replaced by copper SX raffinate from a previous pilot campaign, which had been subjected to an iron removal step. The composition of the cobalt SX feed solution is shown in Table I and the circuit configuration in Figure 2.

Extraction

The organic phase contained 7 vol.% Cyanex 272 and, with a phase ratio of 1, enabled the cobalt concentration to be

consistently reduced to <10 mg/l. The pH was controlled in the last three stages to between 5.5 and 5.65, with the pH being slightly raised towards the end of the extraction bank to ensure low levels of cobalt in the raffinate. Some magnesium, calcium, and nickel were loaded in the last extraction stages, but progressively squeezed off the organic phase by cobalt in the incoming feed solution. Table II shows typical extraction efficiencies achieved for the various elements present in the feed solution.

The NH₄OH consumption for cobalt extraction was 0.88 kg NH₄OH per kg of cobalt extracted, which represents 75% of the stoichiometric requirement. This low consumption has been noted during previous cobalt SX campaigns¹, and is thought to be due to the strong buffering effect of the high ionic strength of the aqueous solution. The consumption includes the NH₄OH required for the extraction of impurity

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Table I
Average composition of feed solution to cobalt SX

Element	Ni	Mn	Fe	Cu	Zn	Ca	Mg	Co
Concentration (g/l)	32.7	0.31	0.001	0.007	0.121	0.576	3.58	1.87

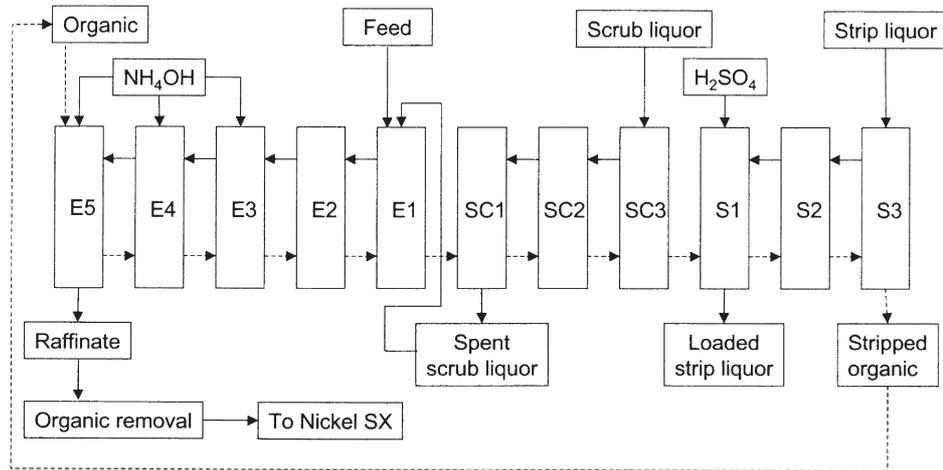


Figure 2—Cobalt SX circuit configuration

Table II
Extraction efficiencies of major elements in the cobalt SX circuit

Element	Ni	Mn	Fe	Cu	Zn	Ca	Mg	Co
Extraction (%)	1.39	99.7	>99.5	>85	>99.5	0.25	2.44	99.7

elements such as copper, manganese, and zinc, as well as cobalt that is scrubbed off the organic phase in the scrubbing section and then re-extracted in the extraction stages.

Scrubbing

The magnesium, calcium, and nickel in the loaded organic phase were scrubbed in three stages at an O/A ratio of approximately 52, using dilute cobalt (26 g/l) sulphate with a pH of 2.8. The scrub mixer-settler units were operated with internal recycle of aqueous phase, adjusted to ensure that the apparent phase ratio in the mixer was close to 1. This ensured that there was sufficient interfacial surface area for adequate mass transfer.

The optimized advance phase ratio and pH in the scrubbing section (pH 4.4 to 4.9) ensured that the following objectives were met:

- The magnesium, calcium, and nickel scrubbed from the loaded organic phase to meet the specification of the cobalt production solution
- The cobalt in the spent scrub solution to be depleted by at least 50% and
- Calcium concentration in the spent scrub solution to be below gypsum saturation level.

Stripping

The scrubbed organic phase was stripped in three counter-current stages using dilute H₂SO₄ (50 g/L). The pH in the first stage was controlled at 2 to ensure adequate stripping of impurities such as zinc, which are loaded in the extraction section and are not scrubbed off in the scrub section. Typical product solution composition is shown in Table III.

Nickel solvent extraction

The raffinate from the cobalt SX plant was passed through a column of coal-based activated carbon (Montan Chemicals GRC 22) for the removal of dissolved organic from the solution. Table IV shows the composition of a typical nickel SX feed, once the cobalt SX circuit had been optimized. The circuit configuration is shown in Figure 3.

Extraction

The objective in the extraction section was to achieve a nickel extraction efficiency of 99%, while minimizing the co-extraction of calcium into the loaded organic phase to a level that could be scrubbed into a low scrub liquor flowrate without the precipitation of gypsum. No dilution was made to the extraction circuit, other than the neutralizing base and the calcium-rich spent scrub liquor. The versatic acid concentration in the organic phase was 30 vol.%.

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Table III

Product solution from cobalt SX

Element	Concentration (g/l)	Element	Concentration (g/l)
Co	29.20	Fe	0.001
Ni	0.008	Zn	1.52
Cu	0.094	Si	0.001
Mn	3.98	Ca	0.003
Al	0.001	Mg	0.568

Table IV

Typical feed composition to nickel SX circuit

Element	Concentration (g/l)	Element	Concentration (g/l)
Ni	31.200	Zn	<0.002
Co	0.005	Si	0.039
Cu	<0.002	Ca	0.541
Mn	<0.002	Mg	3.250

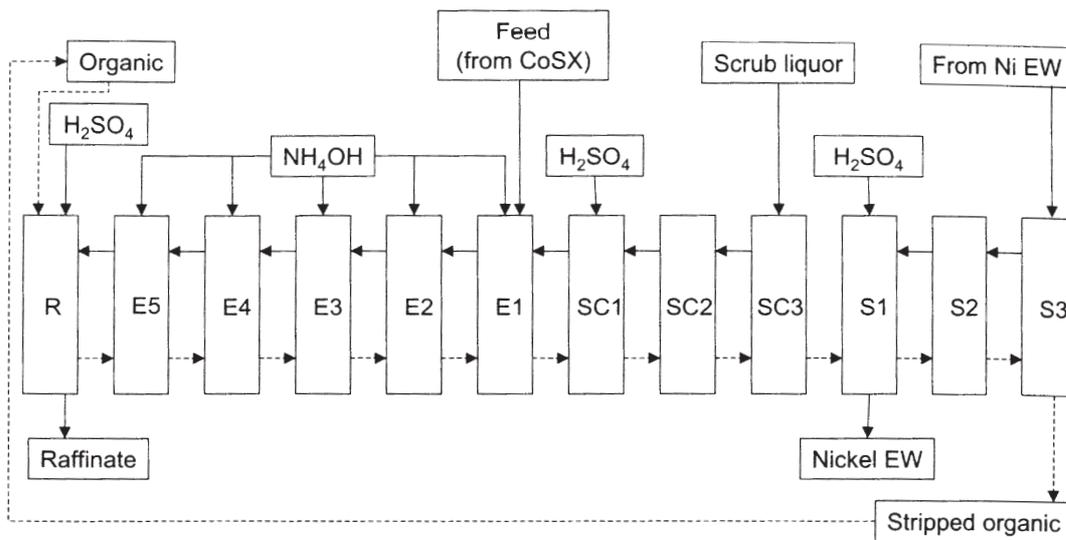


Figure 3—Nickel SX circuit configuration

The nickel SX circuit was started with four extraction stages at the chosen pH profile. To achieve 99% extraction efficiency and improve nickel/calcium separation, a fifth extraction stage was added. The plant was operated to ensure that the calcium concentration in the aqueous phase of each stage was safely below the solubility limit of gypsum.

The main control mechanism was the organic-phase calcium profile. After optimization of the pH profile, the target of 99% nickel extraction was achieved, and the co-extraction of calcium was limited to <3%. Figure 4 shows the aqueous and organic phase profiles of calcium and nickel across the extraction section.

Dissolved versatic acid was recovered from the raffinate by contacting the raffinate with the stripped organic phase. This operation was done in a separate reclamation mixer

settler at a controlled pH of 3, reducing the versatic acid concentration in the raffinate to <25 mg/l.

Scrubbing

The purpose of the scrubbing section was to minimize the transfer of calcium into the EW electrolyte circuit to such an extent that a bleed stream of <5% of the spent electrolyte would be sufficient to maintain the calcium concentration below the gypsum solubility limit. This meant that the calcium concentration on the scrubbed organic should be reduced to <9 mg/l. In order to scrub co-extracted calcium to this level, the correct pH profile across the scrubbing section is essential. To achieve this, the pH in the first scrubbing stage was maintained at 5.9 by the controlled addition of 300 g/l H₂SO₄.

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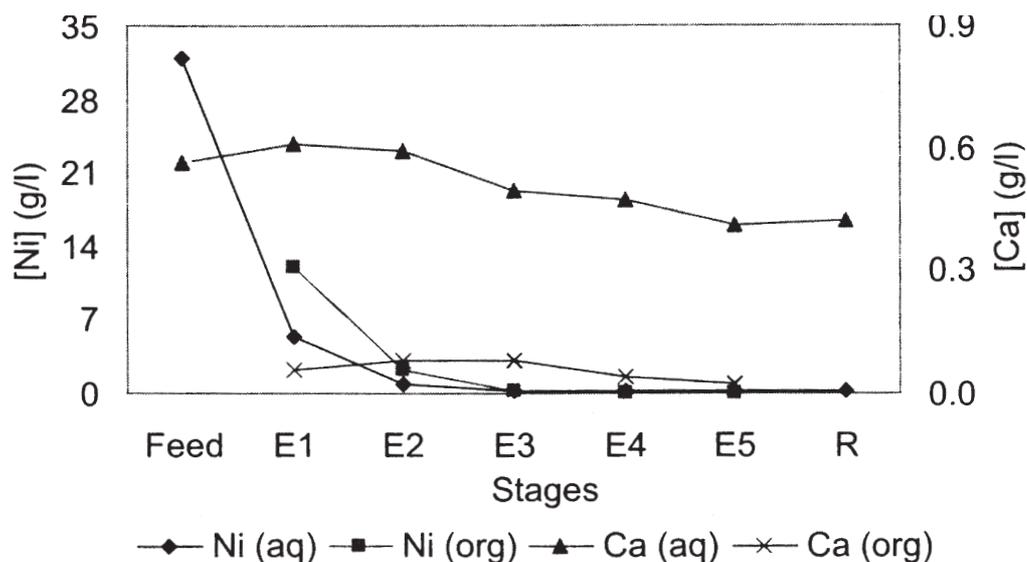


Figure 4—Nickel SX extraction profile

Table V
Composition of nickel SX stripping solutions

Element	Ni	Mn	Fe	Cu	Zn	Ca	Mg	Co
Spent (g/l)	66	0.003	0.001	0.001	0.001	0.21	0.29	0.05
Advance (g/l)	98	0.003	0.001	0.001	0.001	0.22	0.29	0.06

Stripping

The scrubbed organic phase was stripped in three stages using spent EW electrolyte. The steady-state compositions of the stripping solutions are shown in Table V.

Conclusions

The cobalt SX circuit was optimized to recover >99.5% of the cobalt in the feed stream with overall extraction of nickel <0.1%. The cobalt/nickel ratio of the loaded strip solution from this operation was >1500.

Nickel was recovered with >99% extraction efficiency in the second SX plant with overall calcium extraction into the nickel electrolyte circuit of <3%. Importantly, the nickel SX was optimized to prevent precipitation of gypsum or nickel salts in the extraction section.

Acknowledgements

This paper is published by permission of Avmin and Mintek. Start-up conditions for the nickel SX system were determined by John Preston. Pilot-plant control systems were optimized by Milton Summers. Cytec and Shell are thanked for providing the extractants.

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Symposium identifies a need for best practice in 'tracking value'*

Having an agreed approach to the measurement of value across all aspects of a mining and processing operation emerged as a major imperative from the recent Value Tracking Symposium, held in Brisbane.

While delegates were presented with wide ranging views about exactly what is meant by the term 'value' in a mining operation, there was general consensus that tracking the value of a resource as it transforms from the ground into saleable product was vital but not straight forward.

The Symposium covered a wide range of issues associated with the definition of the resource and sampling of process streams that are conventionally associated with understanding mining efficiency.

However, not everyone was convinced that these activities adequately described the value being added at an operational level nor that they respond to corporate reporting needs in a timely fashion.

These issues came into sharp focus with a question posed at the workshop on Day 2 by Newmont's Batu Hijau Process Analytical Services superintendent Karen McCaffery who asked how the value of better measurement could be assessed in monetary terms.

'It's the cost to your day-to-day operation and putting a dollar value on it that I'm trying to get a handle on,' Karen said.

She said she needed to convince management of the need to invest money in a good plant sampling system to provide better reconciliation.

'If I can't put a dollar value to it (sampling) why is it actually important to do it at all?'

Robin Polson of PricewaterhouseCoopers said that in his view there was a lot of talk about value at the Symposium, in terms of individual processes within an operation. He argued that the important point to understand was that value has to be looked at in a holistic way.

'It's very difficult to justify a project on a stand alone basis, as often you are selling it in competition with a lot of other projects.'

'The important thing is to have an economic model that covers the whole of the operation because that's what it is all about—maximizing the whole value, whatever that value is, of the operation.'

Robin said an organization as a whole needed to decide on the key value drivers, such as ore grade, blending options, capacity through the plant, and so forth.

'Having an understanding of the sensitivity of those issues makes it a lot easier to decide whether it is economically beneficial to—for example implement an enhanced drilling programme to improve grades.'

He said having this enhanced level of understanding on such issues as movement in grade had a bearing on the value of an organization.

'It is very hard at an operational level to be able to interpret value without an understanding of its impact on the organization as a whole.'

An emerging opportunity to establish best practice in a standardized way across the industry was presented by Symposium co-chairman Dr Rob Morrison and AMIRA research coordinator Richard Beck with the new P754 metallurgical accounting project.

The JKMRC-AMIRA project aims to look at the possibility of developing guidelines similar to the JORC Code from mine face to final metal.

'Forecasting in the metal accounting process is relatively imprecise, especially when compared to similar procedures in other industries', Richard Beck said.

The project outcomes would enable sponsoring companies to improve the credibility and transparency of metal accounting and the reconciliation reporting process.

'The benefits of this project will be to reduce corporate governance concerns and use metal accounting to identify opportunities to improve operational efficiencies and profitability of operations.'

Richard said AMIRA P754 would build on existing measurement technology to 'attack the problems' with current metal accounting and reconciliation practices in the minerals industry.

The JKMRC-AMIRA metal accounting project was one of a number of practical initiatives presented to Symposium delegates as a potential approach to providing industry with real benefits in terms of tracking the value of mineral industry resources.

Other Symposium highlights included a presentation by Competitive Dynamics Pty Ltd senior consultant Peter Slaughter on the relevance of good corporate governance to maximizing shareholder wealth in mineral commodity companies, MIM project geologist Chris Huddy's paper on grade control at Mount Isa, and Golder Associates principal consultant Bill Shaw's thought-provoking 'beyond grade control', which looked at the broken links in the chain of value.

A range of case studies were presented among several papers on sampling, including a presentation from the director of Mineman Systems, Omar Sehic, on the systematic analysis of error as a means of improving materials accounting and production control in mining.

Taking a holistic approach to the theme was Newcrest's Andrew Logan whose paper looked at leading measures to deliver shareholder value at Ridgeway gold mine.

A total of 27 papers were presented over the two-day event, which included two late afternoon workshops. Each of the workshops was well attended, and to the delight of the Symposium organizers almost all of the delegates stayed until the very end of the workshop late on the second day. At one stage close to 100 people had packed the venue during these sessions.

Where to now?

The principal of Hackchester Pty Ltd, Peter Stoker—a Fellow of the AusIMM and secretary of the Joint Ore Reserves Committee—presented the history and development of the JORC and VALMIN Codes and reporting standards and its application to value tracking. His presentation provided the relevant industry context in which to place any future direction for codification or establishing uniform best practice principles for value tracking.

'The first thing you have to do is make sure you have the stakeholders on side, particularly financial people, because they're interested in what the outcome of all the activity is', Peter said.

'All stakeholders must see the need for and the benefit of the guideline or code, and be involved in its development'.

However, Peter said he preferred to see the development of guidelines rather than codes, particularly prior to general acceptance.

'There's nothing worse than trying to shove something down somebody's throat—and there can never be enough consultation.'

If anyone is interested in getting involved in forming a steering committee or working party on Value Tracking, contact David Goeldner at the AusIMM Southern Queensland Branch on 07 3365 5848 or e-mail d.goeldner@uq.edu.au.

The organizers would also like to thank the Symposium's sponsors Golder Associates, Mineman Systems, ThermoGamma-Metrics and JKTech.

For a copy of the AusIMM Value Tracking proceedings and workshop notes on CD-Rom contact Angie Spry on ajspry@ausimm.com.au or phone the AusIMM on 03 9662 3166. ♦

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