Gold refining by solvent extraction—the Minataur™ Process

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Synopsis

The Minataur™ Process (Mintek Alternative Technology for Au Refining) is a novel route for the production of high-purity gold using solvent extraction. Following the successful demonstration of the process on a pilot-plant scale, a full-scale 24 t/a production plant has been commissioned at Harmony Gold Mine in Virginia, Free State. The commercial implementation of this process represents not only a significant advance in gold-refining technology, but may be instrumental in initiating important changes in the legislation regulating the marketing of gold in South Africa.

Gold of either 99.99% or 99.999% purity can be produced from intermediate process products having a wide range of gold contents. The process comprises oxidative leaching of the solid feed, followed by selective solvent extraction of the gold from the leach liquor to reject impurities, and precipitation of high-purity gold powder. This paper outlines the process, presents selected results of two pilot-plant trials in which 5 kg/d of high-purity gold was produced from silver-refining anode slime and gold-electrowinning cathode sludge, and provides some details of the cost benefits.

Introduction

A solvent-extraction route developed at Mintek for the chemical refining of gold from chloride media has shown considerable promise for the selective extraction of gold from silver, platinum-group metals (PGMs), and base metals, and has potential applications in the refining of gold from various feed materials.

During the past year, two pilot-plant campaigns were carried out in which 5 kg/d of high-purity gold was produced from silver-refining anode slime and from gold-electrowinning cathode sludge. Following the success of these trials, the first full-scale gold refinery to use this process has recently been commissioned. This refinery, at Randgold's Harmony Gold Mine in the Free State Goldfields, is designed to refine 2000 kg of gold per month.

The process

The Minataur™ Process comprises three unit operations, as shown in the flowsheet of Figure 1. Using conventional technology,[1-3] impure gold feed material is leached in hydrochloric acid under oxidising conditions. Most base metals and PGMs are also solubilised under these conditions.

The leach solution is then purified by solvent extraction. Gold is selectively extracted into the organic phase, while other soluble metal ions report to the raffinate. Small quantities of co-extracted impurities are scrubbed from the loaded organic before it is stripped to produce a purified, concentrated gold solution. The stripped organic phase is then recycled to the extraction circuit. The HCl-rich raffinate from the extraction section is recycled to the leach, with a small bleed to control the buildup of impurities in this stream.

Gold is recovered as a metal powder by direct reduction from the loaded strip liquor. A further degree of selectivity can be introduced in this step, and the choice of reducing agent depends on the purity required. Precipitation by oxalic acid should produce gold of 99.999% purity, while sulphur dioxide produces gold of 99.99% purity.

Pilot-plant results

The Minataur™ Process has been evaluated in two pilot-plant campaigns treating 5 kg of gold per day. The first campaign, at Met-Mex Peñoles silver refinery in Torreon, Mexico, operated continuously for three weeks using conditions appropriate to the production of 99.999% gold. The feed to the process was gold-containing anode slime from the silver

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Table I

Average compositions of pilot-plant feed materials

Element	Peñoles (%)	Harmony (%)
Au	98.9	67
Ag	0.9	8.6
Fe	0.09	0.4
Cu	0.002	3.6
Ni		0.5
Pb	0.016	0.4
Zn	0.003	0.7
Al		0.4
Si		5.1
Mg		0.2
Pt	0.021	
Pd	0.012	

electrorefining circuit. The slime had been upgraded by leaching the silver in hot nitric acid. The material treated during the pilot-plant campaign typically assayed 98.9% gold.

The second, five-week campaign treated gold-containing cathode sludge from a conventional carbon-in-pulp/electrowinning circuit at Randgold's Harmony Gold Mine in Virginia, South Africa. This material contained approximately 67% gold, with the balance made up of silver, silica, and various base metals. The compositions of the feed materials treated during the two campaigns are shown in Table I.

Leaching

The leaching operation was conducted on a batch basis. The feed material was leached in 6 *M* HCl, with chlorine continuously sparged into the leach reactor. After leaching, the solid residue was filtered from the gold-containing solution.

A summary of the pilot-plant leaching results is shown in Table II. Because of its high gold content, the Peñoles feed material yielded very little residue. Using the Harmony feed material, most of the chlorinated silver precipitated as silver chloride. The silver can be recovered by conventional techniques, enabling any undissolved gold to be returned directly to the leaching stage.

Solvent Extraction

The compositions of typical leach liquors that were fed to the solvent-extraction circuit and the corresponding loaded strip liquors (LSL) produced are compared in Table III. The extractant is extremely selective for gold over other metals; trace amounts of selenium are the most significant impurity

Table II

Optimised pilot-plant leaching results

Parameter	Peñoles	Harmony
Leaching time (h)	2–3	2
Gold leaching efficiency (%)	99.2	99.3
Gold concentration of feed material (%)	98.9	67
Gold concentration in leach liquor (g/l)	74	65
Fraction of initial gold reporting to residue (%)	0.85	0.67
Gold concentration of residue (%)	99.5	2.2

Table III

Typical compositions of leach liquor and loaded strip liquor

Element	Peñoles		Harmony	
	Leach liquor (g/l)	LSL (g/l)	Leach liquor (g/l)	LSL (g/l)
Au	73.6	124.0	73.1	88.5
Ag	00.53		0.63	
Al			0.15	
Cu	0.001		16.3	
Fe	0.12		0.58	
Mg			1.00	
Ni			1.06	
Pb			1.35	0.003
Pd	0.005	<0.002	0.004	<0.002
Pt	0.031	<0.002	0.007	<0.004
Se	0.007	0.01	0.10	0.005
Si			0.08	
Sn			0.008	0.001
Te	0.04		0.012	
Zn	0.004	<0.001	1.28	

Table IV

Operating efficiency of solvent-extraction circuit

Parameter	Peñoles	Harmony
Extraction		
Extraction efficiency for gold (%)	>99	>99
Organic loading of gold (g/l)	128	83
Gold concentration of raffinate (mg/l)	<100	<100
Stripping		
Stripping efficiency (%)	94	>98
Au:total impurities in LSL (%)	>99.988	>99.97

reporting to the LSL. The operating efficiencies for the solvent-extraction circuit under steady-state conditions are given in Table IV.

No attempt was made to reduce the gold concentration of the raffinate to below 0.1 g/l because the bulk of this solution is recycled to the leach (Figure 1). Implementation of this process at an operating gold mine allows the raffinate bleed to be recovered by adding this stream to the main cyanide leach carbon-in-pulp adsorption circuit. The residual gold content of the bleed stream can also be recovered in a separate precipitation step. Treating a feed material with a high gold content minimises the proportion of the raffinate which must be bled to control impurities.

The long-term stability of the organic phase was closely monitored during both pilot-plant campaigns and in laboratory studies using accelerated test conditions. The components are extremely stable, and no deterioration in performance was observed. Reagent losses are minimised by appropriate design and operation of the plant, and are estimated to contribute less than R0.03 per kilogram of gold to the production cost.

Reduction

During the Peñoles trial, oxalic acid and sulphur dioxide were tested as reducing agents to precipitate gold from the loaded strip liquor. The use of oxalic acid introduces a further degree of selectivity and enables a gold purity of 99.999% to be

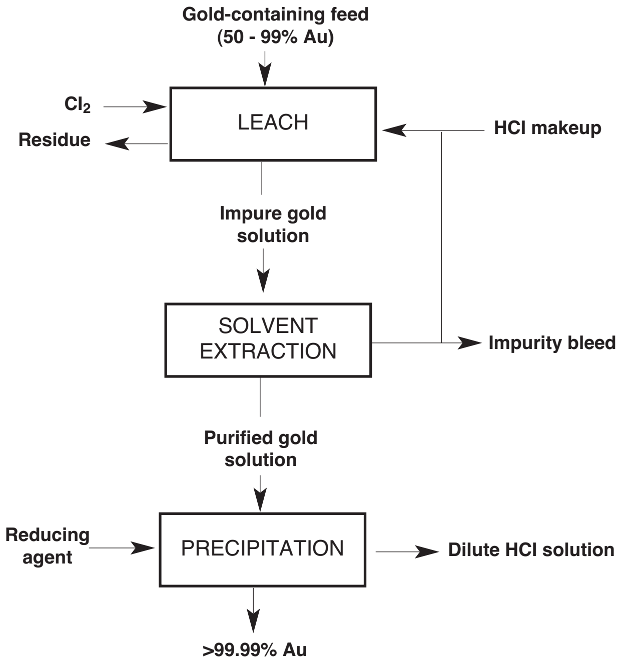

Figure 1—The Minataur™ Process

Table V

Typical analysis of high-purity gold

Element (ppm)	ASTM† 99.99%	Peñoles*	Harmony*
Au (%)	99.99	>99.999	>99.99
Ag	90	0.2	2
Cu	50	0.2‡	0.8
Pd	50	0.1‡	<0.5
Fe	20	0.3	0.32
Pb	20	0.2‡	<0.2
Si	50	0.3‡	
Mg	30	0.1‡	0.7
As	30	0.8‡	
Bi	20	0.1‡	
Sn	10	0.2‡	<0.5
Cr	3	0.1‡	
Ni	3	0.4	0.9
Mn	3	0.1‡	

* Gold determined by difference
† Minimum allowable concentration of gold; maximum allowable concentration of all other elements
‡ Detection limit for analytical technique (ICP-MS)

attained. This route is, however, slower, more costly and more difficult to control than reduction with sulphur dioxide gas, which achieves a gold purity of 99.99%[4]. Table V shows typical analyses of the gold produced in the two campaigns, with the ASTM specification[5] for 99.99% gold also shown for comparison. Analysis of 36 impurity elements was carried out, most of which were undetected.

Process economics

Based on the pilot-plant data, preliminary estimates of the capital and operating costs for a plant of 24 t/a capacity have been calculated (Table VI). Costing commences with the presentation of wet, impure gold sludge to the plant, and ends with molten, pure gold available for casting. In this estimate, a feed material with a gold-to-silver ratio of 9:1 and

Table VI

Estimated capital and operating costs for the Minataur™ Process for a plant with a refining capacity of 24 t/a

Cost item	Cost
Capital (R)	3 450 000
Operating Costs (R/a)	
Variable	359 000
Fixed	417 000
Cost (R/kg Au)	32

a site with a complete infrastructure (existing building, utilities, analytical facilities, security, technical supervision) are assumed. The capital cost excludes all taxes, owners' costs, preproduction expenses, testwork, and commissioning fees, but includes the technology fee. In estimating the fixed costs, only labour costs directly attributable to the operation of the refinery are included; maintenance and insurance are excluded. A single 8-hour shift operating for 330 days per annum is assumed. Neutralised effluent is discharged at the plant battery limits. The operating costs have assumed amortisation of the capital over five years. All costs relate to South African conditions.

It should be noted that the accurate analysis of high-purity gold requires specialised instrumentation. Also, marketing and obtaining the premium price for high-purity gold requires certification of the analytical procedures. The cost of equipping an analytical laboratory appropriately can add significantly to the capital cost of the process.

Because the solvent-extraction operation is continuous, a small inventory of gold is maintained in this circuit.

Discussion

Other feed materials

The anode slime gold content (Peñoles) averaged about 99%, while the cathode sludge composition (Harmony) varied between 48 and 82% gold on a daily basis. Other feed materials that can be satisfactorily processed include zinc precipitation solids, the gold fraction of gravity concentrates, and the residue from mill liners in metallurgical gold plants[6,7]. Small-scale leaching tests have demonstrated the amenability of these materials to leaching, producing a leach liquor suitable for solvent extraction.

The mineralogy of the material is important in ensuring that gold can be solubilised. The particle size of the feed influences the kinetics of leaching, and finely divided material is preferred.

Gold lock up

The lock-up time of the gold in the circuit depends very largely on the nature of the feed material and on how the plant is operated. By employing continuous operation, minimising tankage volumes, and optimising scheduling, it is possible to reduce the gold residence time in the circuit to less than 24 h from introduction of the feed material to the leach to production of high-purity gold powder. It is then necessary to dry the powder prior to melting and casting it into a saleable product for dispatch.

Comparison with other processes

Wohlwill electrorefining process

The major established technology for the production of high-purity gold is the Wohlwill electrorefining process[8,9]. In a typical operation, the impure gold is cast into anodes and electrorefined in an $HCl/HAuCl_4$ electrolyte. The average life time of an anode is 22 h, after which time the remaining anode material is recycled. A gold purity of 99.99% is typically achieved.

The lock up of gold in the Wohlwill process is substantial because of the necessity to cast anodes, the high concentration of gold in the electrolyte (approximately 100 g/l), and the significant recycle of gold in the circuit, both through spent anodes (~25%) and as losses to the anode slimes (up to 30%). The Minataur™ Process has a significant advantage in reducing both the residence time of the gold in the circuit and the amount of gold recycled. Purities higher than 99.99% can be produced if required.

INCO solvent-extraction process

INCO Europe Ltd installed a solvent-extraction process for the refining of gold in 1972 at their Acton precious metal refinery, UK[10,11]. The extractant used is diethyleneglycol dibutyl ether (dibutyl 'carbitol'). Although the extraction of gold from HCl solution with this solvent is quantitative (gold loading to 30 g/l) and selective with respect to PGMs, stripping is not easy. After scrubbing, the gold is recovered by direct reduction from the loaded organic with oxalic acid. Because the formation of a third phase, particularly a solid, is undesirable in a continuous solvent-extraction system, the stripping is operated as a batch process.

The major disadvantage of this process is the significant cost of solvent replacement due to solvent losses of up to 4% per cycle. The aqueous solubility of this extractant is high (~3 g/l), and the raffinate must be distilled to recover the extractant, as it is relatively expensive. The solvent also tends to adsorb onto the gold powder during reduction from the organic phase.

The Minataur™ Process, in contrast, employs inexpensive, readily available organic reagents with low aqueous solubility. The replenishment costs are a minor proportion of the operating costs of the process. Gold is readily stripped from this extractant, eliminating the need for direct reduction from the loaded organic phase and the messy solid/organic separation. This enables the reduction to be carried out on a continuous basis if required. The high selectivity for gold over base metals and PGMs achieved by the extractant system enables production of a very pure loaded strip liquor, and gold of 99.99% purity can be produced using an inexpensive reducing agent.

Current developments

Based on the success of the two pilot-plant campaigns, and favourable techno-economic and marketing feasibility studies, a full-scale refinery using the Minataur™ Process has been built and commissioned at Harmony Gold Mine. The plant is designed to refine 2000 kg/month of gold, operating 1 shift per day, with expansion capacity to accommodate 300 kg/d of gold. It is relatively small, occupying an area of about 70 m^2 (excluding bulk reagent and gas storage). In addition to producing kilogram bars, gold granules, and gold powder, Randgold anticipates establishing a jewellery manufacturing facility on site[12].

Historically, all of South Africa's gold has been refined by the Rand Refinery and marketed solely through the South African Reserve Bank. Some years ago, legislation was relaxed to allow the Rand Refinery to market one-third of its members' gold directly on the international market. In November 1996, Randgold received permission from the Reserve Bank and the Ministry of Trade and Industry to build their own refinery and independently market one-third of their gold[13,14]. This dispensation is expected to have a positive effect on Harmony's cost structure and enhance its long-term profitability.

The present cost to Harmony Gold Mine of refining gold, including smelting to produce Doré bullion, is R267/kg. It has been estimated that implementation of the Minataur™ Process will contribute to an annual saving of R3.6 million in operating costs for the mine[15].

Conclusions

A novel route for the production of high-purity gold using solvent-extraction technology has been successfully piloted using gold-containing feed materials of widely differing characteristics. The economics of the process are extremely attractive, and the first full-scale production plant has been commissioned.

Advantages of the Minataur™ Process over conventional electrorefining technology include the significantly reduced lock-up times of the gold, the ease of operation and control, and the ability to produce a high-purity product in a very forgiving circuit. The process is particularly attractive for feed materials containing significant quantities of base metals.

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A keynote speaker, Prof Markus Reuter from Delft will, with other visitors, Professor Chris Aldrich and Dr Derek Moolman of the United States, highlight the Process Control Workshop. ◆