APPLICATION FOR A STANDARD PATENT

We, NORCIM INVESTMENTS PTY. LTD.
Unit 7, 14 King Edward Road, Osborne Park, Western Australia
hereby apply for the grant of a Standard Patent for an invention entitled

"METAL RECOVERY METHOD"

which is described in the accompanying provisional specification.

For a Convention application - details of basic application(s) -

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<th>NUMBER</th>
<th>COUNTRY</th>
<th>DATE OF APPLICATION</th>
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For an application made by virtue of section 51 -

Original Application No. ____________________ by ____________________

I request that the Patent may be granted as a Patent of Addition

the Patent applied for on Application No. ____________________ to
Patent No. ____________________ in the name of ____________________

I request that the term of the Patent of Addition be the same as that for the main invention or so much of the term of the Patent for the main invention as is unexpired.

My address for service is KEVIN LORD and COMPANY, PATENT AND TRADE
MARK ATTORNEYS, 703, MURRAY STREET, WEST PERTH, WESTERN AUSTRALIA

Dated this 12th day of MARCH 1982 AUSTRALIA

CIM INVESTMENTS PTY. LTD.
their Patent Attorneys

VIN LORD and COMPANY

(Signature)

DECLARATIONS

1) of

In support of the above application for a patent for

2) and

I, ARNOLD PRESCOTT, Scarborough

do solemnly and

1. I am/we are

2. The basic application of the Act was made for a patent for

3. The said basic application was made in

4. The actual inventor of the invention is

5. The facts up to this point have been made to the best of my knowledge and belief.

THE COMMISSIONER OF PATENTS

(Signature)

DECLARED at Perth AUST

This form may be completed in parts, but the complete application must be countersigned by the applicant and filed with the Commissioner of Patents.

The present invention relates to metal recovery.

Gold is typically recovered from ores or concentrates by treating the ore or concentrate with aqueous sodium or zinc chloride. The Zadra process produces gold in a form suitable for further processing. This invention describes a method for improving the recovery of gold from such concentrates.
DECLARATION IN SUPPORT OF A CONVENTION OR NON-CONVENTION APPLICATION FOR A PATENT OR PATENT OF ADDITION

1) of applicant(s)

In support of the application made by NORCIM INVESTMENTS PTY LTD for a patent for an invention entitled "GOLD RECOVERY METHOD"

2) and 

3) of -ant(s)

and is (es)

and son(s)

3) of a
tion

3) of the -ant(s)

and is (es) is (es) and son(s)

do solemnly and sincerely declare as follows:-

1. I am/we are the applicant(s) for the patent, or am/are authorised by the abovementioned applicant to make this declaration on its behalf.

2. The basic application(s) as defined by Section 141 of the Act was/were made in the following country or countries on the following date(s) by the following applicant(s) namely:

3. The said basic application(s) was/were the first application(s) made in a Convention country in respect of the invention the subject of the application.

4. The actual inventor(s) of the said invention is/are MYSELF ARNOLD FREDERICK GRIFFIN and MICHAEL CHARLES COSTELLO of 15 Elmhurst Way, Greenwood, Western Australia, AUSTRALIA.

5. The facts upon which the applicant(s) is/are entitled to make this application are as follows:-

DECLARED at Perth, Western this 20 day of Dec 1982 AUSTRALIA.

[Signature]

This form may be completed and filed after the filing of a patent application but the form must not be signed until after it has been completely filled in as indicated by the marginal notes. The place and date of signing must be filled in. Company stamps or

the Zadra process was developed in the United States and this process with local modifications has been used by gold producers in a number of countries.
Claim 1. A process in which a particulate material containing water and having adsorbed thereon a solubilised gold compound is located in packed form in a fractionating column, a liquid containing a major proportion of water-miscible organic liquid is boiled in an evaporator such that it enters the packed fractionating column as a vapour, said water-miscible organic liquid being more volatile than water and being preferentially retained in the column.

Example 2: A charge of gold was...
Example 2 Operating plant conditions

A charge of 200 litres of pregnant carbon containing
gold was condition with a complete wash in

...
The present invention relates to metal recovery.

Gold is typically recovered from ores or concentrates by treating the ore or concentrate with aqueous sodium or calcium cyanide in the presence of oxygen. The gold forms a soluble complex with the cyanide.

To separate the soluble metal rich portion of the resulting slurry or solution the slurry or solution may be contacted with an adsorbent material such as carbon, activated carbon, ion exchange resins, attapulgite, silica or alumina gel or one of a number of organic compounds both natural and synthetic. In practice, the adsorbent material adsorbs many different compounds including gold, silver, copper, nickel, iron, calcium and magnesium both as simple and complex compounds. In an alternative process, the slurry or solution containing a soluble gold cyanide complex may be contacted with a less noble metal such as zinc or aluminium. In this case the gold is extracted from the soluble complex by cementation.

The next step is to recover the gold from the cementation product or from the adsorbent material to obtain a salable product which is called Dore Bullion.

In the past gold has been recovered from adsorbent materials by burning combustible adsorbent material which was usually activated carbon, and smelting the ash with fluxes. Thus, the adsorbent material was destroyed.

In the early 1950's an elution process called
the Zadra process was developed in the United States and this process with local modifications has been used by gold producers in a number of countries.

In the Zadra process, gold laden carbon is eluted in batches with a near-boiling caustic-cyanide solution in closed circuit with one or more electrolytic cells and a heater to maintain the desired eluant temperature. The redissolving of gold from the carbon is temperature dependant and the time of elutriation may be reduced from 4-5 days to 1-2 days by increasing the elution temperature to above 100°C by pressurizing the system.

In alternative procedures methanol, methyl cyanide and deionised water have been used as eluants in systems which are modifications of the Zadra process.

The present invention provides a process in which gold and other metals including silver and copper, may be desorbed from an adsorbent material.

The gold is mainly present on the adsorbent material in the form of a highly insoluble compound which is probably calcium aurocyanide. Thus, to enable the gold values to be recovered it is necessary to convert the gold to a soluble form and subject the adsorbent material to a leaching.

In accordance with the present invention there is provided a process in which a particulate material containing water and having adsorbed thereon a solubilised gold compound is packed in a fractionating column, a liquid containing a major proportion of water-miscible
organic liquid is boiled in an evaporator such that it enters the fractionating column as a vapour, said water soluble organic liquid being more volatile than water and being preferentially retained in the column.

Preferably, in the process of the present invention, the vapour from the boiling organic liquid enters the carbon column and condenses on the carbon, and on the liquids adsorbed onto the carbon. The entire charge is therefore heated and temperatures approaching 100°C occur on the column. The front of heated solvent rises through the carbon bed until it has heated the entire bed and the vapour then breaks through the top of the column and is piped to a condenser, condensed and subcooled. This subcooled condensed vapour may be passed through a flowmeter and then returned to the top of the column via a liquid distributor. The condensed liquid works its way by gravity down the column countercurrent to the rising vapours and the process of fractionating occurs using the carbon as the packing in a fractionating column. When the process reaches equilibrium the more volatile low boiling solvent is produced at the top of the column and the gold bearing water originally present on the carbon exists in the evaporator. The process is typically continued until at least 80% by weight preferably such time as the bulk of the gold such as/ at least 90% by weight, is in the evaporator and the bulk of the water soluble organic liquid is in the fractionating column.

By this time, the liquid in the evaporator typically contains a relatively high gold concentration which may
be in excess of 3000 milligrams per litre and can be as high as 6000 milligrams per litre or more dependant on the gold loading of the carbon used.

The organic liquid is preferably methanol but other organic liquids such as alcohols, ketones or nitriles could be used if desired. For example, the organic liquid could be acetone or acetonitrile. The particulate material is typically carbon although other particulate materials could be used if desired.

The volume of water soluble organic liquid required is relatively low being typically less than 1 bed volume. In many instances it is found that the optimum amount of water soluble organic liquid is 0.5 bed volume. Following completion of the reflux period the pure solvent may be directed away from the column and into storage for reuse in the next cycle. During this time the level in the evaporator falls and the top and bottom column temperatures rise. When the evaporator level falls to be equal to the water content of the original carbon charge the pregnant concentrate may be drained into an electrolytic cell. Steam may be injected under the carbon bed and the remaining solvent displaced from the carbon, part to storage for reuse and part to waste. This process is continued until the column top temperature reaches 99°C at which time the solvent is displaced from the carbon making it available for reuse.

The gold rich aqueous solution may be subject to a number of techniques to recover the gold for smelting and refining. The gold concentration depends on the initial
gold concentration in the particulate material. The solution contains water, gold, other desorbed soluble compounds and excess reagents initially present in the particulate material and a variety of organic compounds.

For example, the gold rich solution can be subject to evaporation of liquids, cementation by zinc/lead, electrodeposition or precipitation such as by sulphur dioxide or oxalic acid.

It has been found in particular that the solution can be subject to electrodeposition using an aluminium cathode as will be described hereinafter in more detail.

The gold deposits on the aluminium.

Subsequently, the aluminium can be removed by being dissolved in diluted aqueous sodium hydroxide.

Initially, the gold bearing particulate material may be water washed to remove material such as clay or
ore chips from the pulp. Then, the gold may be solubilised by soaking the cleaned particulate material for an appropriate time in an aqueous solution of an appropriate reagent such as aqueous strong caustic cyanide solution. The solubilising treatment may be continued for about 1 hour. The so treated particulate material is, as described above, charged into a fractionating column, and excess solution drained. Alternatively, the cleaned particulate material may be charged into the fractionating column and treated with the solubilising reagent whilst in the fractionating column. The treated material is allowed to drain until most of the water has been removed. Residual caustic cyanide solution can be discarded or reused after reagent adjustment.

The present invention will now be described, by way of example, with reference to the accompanying drawing which is a schematic side elevation of an apparatus which may be used for carrying out the process of the present invention. The apparatus shown in the drawing comprises a fractionating column 10 having insulated sides 11 and arranged to receive a charge of gold bearing particulate material. The fractionating column 10 conveniently comprises a lid or the like to enable gold laden particulate material to be loaded into it and an exit means adjacent the lower end of the column for removal of treated material. A carbon screen 10a separates the column 10 from an evaporator 12 to be described. Alternatively, the particulate
material may be contained in a separate cartridge with a carbon support screen which can be inserted as a whole in the column 10 and removed when desorption is complete.

Beneath the column 10 is an evaporator 12 containing a quantity of methanol which may contain some water. In use, the column 10 is packed with a charge of carbon having adsorbed thereon gold said gold being pre-treated or treated in the column to solubilise it as described above. The evaporator 12 also comprises an insulated heating jacket 13 connected by lines 14 and 15 to a heating system 16. The heating jacket 13, lines 14 and 15 and heating system 16 contain heating oil which is circulated by means (not shown) through the system.

Further, the column 10 contains a number of temperature sensors (not shown) to enable the temperature in the column 10 to be monitored. This assists in establishing the progress of the process at any time. On top of the column 10 is a reflux condenser system 18. The condenser system 18 is connected to the column 10 by means of lines 19 and 20. The condenser system 18 is water cooled and comprises a water inlet line 21 and a water outlet 22.

In use, about 0.5 bed volume of organic liquid such as methanol is placed in the evaporator 12. Henceforth the process will be described with particular reference to methanol and carbon although it is to be understood that other organic liquids and other particulate materials can be used if desired. The methanol is boiled by being heated by heating oil in the jacket 13 and enters the
column 10 as a vapour.

There is then established a dynamic process whereby water drains from the column 10 in preference to the more volatile methanol. Methanol vapour rises and enters the water bearing carbon packing in the column 10. The methanol vapour condenses upon contacting the colder particulate material and is thereby converted into liquid. The methanol liquid heats the water and mixes with it. Solubilised gold compounds are dissolved by the methanol and the dissolved compounds flow down the column 10 with the methanol-water mixture. This process continues until the whole of the charge is heated and has a water-methanol content of various proportions throughout the entire column of particulate material.

The approximate temperatures existing in this stabilised condition are evaporator 75°C, lower carbon layer 68°C and upper carbon layer 65°C. The amount of heat applied then controls the rate of boiling and hence the amount of methanol rich vapour issuing from the top of the column 10 and entering the condenser system 18.

The rate of reflux can be from 0.1 bed volumes per hour to 0.5 bed volumes per hour depending on the type and particle size of the carbon, the height to diameter ratio of the column 10, the available heat and time allocated for the desorption.

During the process, solution samples may be
periodically withdrawn from the evaporator and gold concentrations determined. These values may be noted and when the gold concentration reaches a steady value the gold transfer to the evaporator is considered complete.

Whilst heating continues oil is heated in the heater system 16, passed along the line 14 into the jacket 13 and then through the line 15 to the heating system 16. Methanol is driven from the top of the column 10 but it is condensed in the condenser system 18 and returned to the column 10 along the line 20. The methanol vapour passes along the line 19 to the condenser system 18, is condensed to liquid and returns to the column 10 via the line 20. The condenser system 18 is cooled by passing cool water into the condenser through the line 21 and passing used water out of the condensor along the line 22.

The concentration of methanol in the evaporator 12 gradually decreases whilst the water concentration gradually increases.

The process is preferably continued until substantially all the gold has been desorbed.

The concentrated gold solution in the evaporator 12 can be drained off through an outlet line 23 for further treatment.

Prior to this, the methanol which is condensed in the reflux condenser system 18 is directed through an outlet (not shown) in the line 20 to storage for subsequent further use. The temperature in the evaporator
12 begins to rise and the system becomes impoverished in methanol. This continues until the recovered condensate temperature reaches 75-80°C and the solution in the evaporator has a boiling point of 95-100°C. Heating is then discontinued.

The condenser system 18 can be horizontal or vertical, separate (as shown) or located directly on top of the column 10 so forming an integral part thereof. The heating system 16 can be heated by burning a fuel or by waste heat such as from diesel engines. The choice of fuel is in practice dictated by site conditions and facilities. The heating medium may also be steam from a conventional package boiler.

The solution recovered from the evaporator contains almost no methanol, but water and eluted compounds including organic material. The recovered material is removed and sampled and the gold content determined. This is usually double the concentration of gold in the original carbon.

One preferred metal recovery method involves subjecting an aqueous gold containing solution to electrodeposition in a cell using an aluminium cathode and a metal anode. The gold deposits on the aluminium and the aluminium may then be removed by being dissolved in an aqueous sodium hydroxide solution.

It has been found that aqueous alkali solutions, preferably having a pH of at least 10, and containing dissolved metal such as gold or silver, can be subjected to electrolysis so as to deposit the dissolved metal on
a variety of metal cathodes. The metals used for the cathodes may include gold, silver, copper and aluminium. The metals would typically be in foil form but they could be in shredded foil form.

The cathodes are preferably in the form of parallel plates.

The voltage is applied to the electrolytic cell before electrolytic addition to apply cathodic protection to the aluminium cathodes and prevent them being dissolved by the hot alkaline electrolyte.

The electrolysis is typically continued until the concentration of gold in the electrolyte falls to less than 10 ppm. Cell design is preferably such that adequate cathode area is provided to reduce the gold concentration from its maximum value to less than 10 ppm in an overnight 16 hour period using plain aluminium foil or in a four hour period using shredded aluminium foil. Cell design also preferably fulfils the need to contain the entire electrolyte in a single charge.

At the completion of the plating period parallel plate cathodes may be lifted from the cell and the aluminium backing dissolved in a sodium hydroxide solution leaving the gold foil in a pure form suitable for smelting. The entire cell contents are filtered to recover any fine gold which did not form an adherent plate.

Preferably, during the electrolysis step, the current density is controlled so as to selectively
electrodeposit gold and silver onto the aluminium cathode and leave other metals such as copper in the electrolyte. The silver content is with the amount of cyanide soluble silver compounds in the original ore. In cases where this silver is about equal to or less than the gold concentration then co-deposition occurs. In cases where this silver is significantly higher than the gold then the silver will be deposited before the gold. Alternatively, it can be precipitated before electrolysis by use of an appropriate amount of sodium sulphide. Most preferably, the current density is in the range from 0.0015 to 0.005 amp per sq. centimetre of cathode surface. If the cathode is not aluminium, it is conveniently formed of a material with which the deposited metal is compatible. For example, copper or silver or gold cathodes can be used for gold recovery since these can be recovered during the gold refining process. The use of aluminium is preferred however since it is cheaper, readily available, and easily dissolved on completion of the plating process.

The present invention will now be illustrated by the following examples.

Example 1. Pilot plant conditions using an apparatus of the type shown in the accompanying drawing.

A charge of 25 litres of pregnant carbon containing gold to 5330 g/t, was presoaked in a solution containing 50 g/l sodium cyanide and 10 g/l sodium hydroxide for one hour. This conditioned carbon was charged into the
fractionating column and 15 litres of methanol run into the evaporator and heated to boiling point of 67°C.

The methanol vapour rose, entered the carbon and gradually displaced water containing gold and reagent from the carbon. This aqueous liquid gravitated to the evaporator. The evaporator temperature gradually rose to 70°C and the temperature in the carbon gradually rose to near 100°C then fell to 67°C as the methanol saturated the carbon and rose as a vapour to the condenser from which it returned to the carbon as a condensate.

The elution process continued for 16 hours. Then the condensate from the condenser was directed to a storage tank and the temperature in the evaporator gradually increased to near 100°C as the system became improved of methanol.

The aqueous solution in the evaporator was then transferred to an electrolytic cell containing an aluminium cathode and subjected to electrolysis to deposit the gold. The gold content of this electrolyte was determined at 11,000 g/t. Electrolysis was continued for 4 hours when the gold content of the electrolyte was determined at 4 g/t. The plated cathode was removed and placed in an alkaline bath when the aluminium was dissolved away. The gold content of the desorbed carbon was determined at 205 g/t.

**Efficiency calculations**

- Gold desorption from carbon 96.15%
- Gold electrodeposition 99.96%
- Overall gold recovery 96.11%
Example 2 Operating plant conditions
A charge of 200 litres of pregnant carbon containing
gold was conditioned with a caustic-cyanide solution of
the same concentration and for the same time as Example
1.

This charge was charged into the 200 litre fraction-
ating column and 120 litres of methanol was heated in the
evaporator to boiling point of 67°C. The methanol vapor rose and entered the conditioned carbon and the water-gold
reagent solution gravitated to the evaporator.
The evaporator temperature soon stabilized at 70°C and the
upper portion of the carbon attained a constant temperature
of 67°C, similar to Example 1. This condition was
maintained for 16.5 hours after which the condensate was
directed out of the system to storage.

When the temperature in the system attained close
to 100°C the solution in the evaporator was transferred
to the electrolytic cell as in Example 1. It then
contained gold to the concentration of 3300 ppm. Electro-
deposition continued for 16 hours when the gold
concentration in the spent electrolyte was determined
at 3 ppm. The aluminium cathodes were removed and
dissolved as in Example 1.
The carbon was discharged and assayed for gold.

Efficiency calculations

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<tr>
<td>Gold desorbed from carbon</td>
<td>91.0%</td>
</tr>
<tr>
<td>Gold electrodeposition</td>
<td>99.9%</td>
</tr>
<tr>
<td>Overall efficiency</td>
<td>90.9%</td>
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Modifications and variations such as would be apparent to a skilled addressee are deemed within the scope of the present invention.
3.

The claims

1. A process involving water and a miscible organic compound in a column, a vapour, that is adsorbed in the column.

2. A process involving particulate material.

3. A process involving an organic liquid.


5. A process involving one bed of disso.

6. A process involving a material.

7. A process involving a material.

8. A process involving a material.
The claims defining the invention are as follows:

1. A process in which a particulate material containing water and having adsorbed thereon a solubilised gold compound is located in packed form in a fractionating column, a liquid containing a major proportion of water-miscible organic liquid is boiled in an evaporator such that it enters the packed fractionating column as a vapour, said water-miscible organic liquid being more volatile than water and being preferentially retained in the column.

2. A process according to claim 1, in which the particulate material is activated carbon.

3. A process according to claim 1 or 2, in which the organic liquid is a water miscible alcohol.

4. A process according to claim 3, in which the organic liquid is methanol.

5. A process according to any one of the preceding claims, in which the volume of organic liquid is less than one bed volume.

6. A process according to any one of the preceding claims, in which the water content of the particulate material drains into the evaporator in admixture with organic liquid and carries with it the dissolved gold.

7. A process according to claim 6, in which fractionation is continued until at least 90% by weight of the adsorbed gold has been removed to the evaporator.

8. A process according to claim 6 or 7, in which the evaporator contains at least 3000 milligrams per litre of dissolved gold at the termination of fractionation.
9. A process according to any one of claims 6 to 8, in which following completion of fractionation and solvent recovery the gold bearing aqueous liquid from the evaporator is subject to electrolysis or cementation to separate gold from the liquid.

10. A process according to claim 9, in which the gold bearing aqueous liquid is processed in a cell with an aluminium cathode and a metal anode and subjected therein to electrolysis to deposit a gold containing foil on the cathode.

11. A process according to claim 10, in which subsequent to termination of electrolysis the aluminium cathode is dissolved by an alkali.

12. A method of recovering gold from an aqueous solution, which comprises placing the gold containing aqueous solution in a cell comprising a gold, silver, aluminium or copper cathode and an inert electrode, subjecting the solution to electrolysis in the cell so as to deposit a gold foil on the cathode.

13. A method according to claim 12 in which the cathode is aluminium.

14. A method according to claim 13, in which the current density during electrolysis is between 0.0015 and 0.005 amps per square centimetre of cathode area.

15. A method according to claim 13 or 14, in which subsequent to electrolysis the aluminium cathode is dissolved by an alkali.
16. A method according to any one of claims 13 to 15, in which the gold containing aqueous solution is alkaline and has a pH of at least 10.

17. A process according to claim 1, substantially as hereinbefore described.

18. A method according to claim 12, substantially as hereinbefore described.

DATED NOVEMBER 18 1982

NORCIM INVESTMENTS PTY. LTD.

by their Patent Attorneys,

KELVIN LORD AND COMPANY,

PERTH, WESTERN AUSTRALIA