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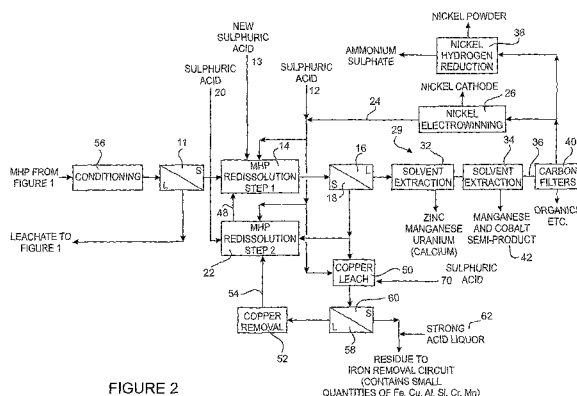


FIGURE 2

(57) Abstract: A method for the treatment of mixed hydroxide product (MHP) (10) produced in a metal extraction process such as a nickel extraction process. The method comprises the steps of treating the MHP (10) with a first acid solution (12) at a pH in the range of 4 to 8 as a first redissolution step (14) and separating a first liquor (16) formed in the first redissolution step (14) from a first residue (18) formed in this step. The method further comprises the step of treating the first residue (18) with a second acid solution (20) at a pH in the range of 0.5 to 4 as a second redissolution step (22). In this way, the impurities in the MHP may be selectively removed in the two redissolution steps.

“METHOD FOR THE TREATMENT OF MIXED HYDROXIDE PRODUCT PRODUCED IN A METAL EXTRACTION PROCESS”

Field of the Invention

5 The present invention relates to a method for the treatment of mixed hydroxide product (MHP) produced in a metal extraction process, typically a process used for the extraction of nickel and cobalt. The invention relates particularly but not exclusively to a method for the treatment of MHP to render it suitable for further treatment to produce the metals of nickel and cobalt.

10 Background to the Invention

Nickel (Ni) is primarily sold for first use as refined metal (cathode, powder, briquette, etc.) or ferronickel. About 65% of the nickel consumed in the Western World is used to make austenitic stainless steel. Another 12% goes into superalloys (e.g., Inconel 600) or nonferrous alloys (e.g.,
15 cupronickel). Both families of alloys are widely used because of their corrosion resistance. The aerospace industry is a leading consumer of nickel-base superalloys. Turbine blades, discs and other critical parts of jet engines are fabricated from superalloys. Nickel-base superalloys are also used in land-based combustion turbines, such as those found at electric
20 power generation stations. The remaining 23% of consumption is divided between alloy steels, rechargeable batteries, catalysts and other chemicals, coinage, foundry products, and plating.

Cobalt (Co) is a strategic and critical metal used in many diverse commercial,
25 industrial, and military applications. A significant use of cobalt is in superalloys, which are used to make parts for gas turbine aircraft engines. Cobalt is also used to make magnets; corrosion- and wear-resistant alloys; high-speed steels; cemented carbides (also called hardmetals) and diamond tools; catalysts for the petroleum and chemical industries; drying
30 agents for paints, varnishes, and inks; ground coats for porcelain enamels; pigments; battery electrodes; steel-belted radial tires; and magnetic recording media.

There are various ways to extract nickel and cobalt from ores, concentrates and other “intermediates” (feed materials). These include heap, vat, tank and pressure leaching of laterite and heterogenite ores, as well as atmospheric and pressure leaching of mattes, alloys and concentrates. Most of the
5 starting materials for these processes contain impurity elements such as iron, manganese, zinc, aluminium, silicon, chromium, chloride, uranium, copper and cadmium. These impurities are usually taken up into the leachate with the value metals of nickel and cobalt. Therefore, the leaching process is followed by a purification sequence to remove impurities to allow for ease of nickel
10 (and possibly cobalt) extraction.

Pyrometallurgical methods are also used for nickel extraction and involve the thermal treatment of minerals and metallurgical ores, and concentrates, to bring about physical and chemical transformations in the materials to enable
15 recovery of valuable metals. Pyrometallurgical methods involve one or more of the steps of calcining, roasting and smelting which are often energy and cost intensive.

One Australian nickel producer has treated liquor from a nickel leaching
20 process to initially remove iron, followed by a number of solvent extraction steps to remove the impurities of copper, cobalt, manganese, zinc, and so on. The final step involved a solvent extraction process for the upgrade of nickel to produce an electrolyte. However this process had a number of aspects which caused problems in consistently being able to produce on-specification
25 nickel. This process often resulted in a low grade nickel product since many of the impurities remained in high concentrations with the nickel in the end product. In addition, it required the use of sodium and/or ammonium reagents to neutralise the excess acid released in the solvent extraction steps which in most countries would attract further on-treatment costs to minimise the impact
30 on the environment. Furthermore, the process directly coupled the leach step to the metal winning step which led to overall plant availability concerns.

There has been some interest in heap leaching for the extraction of nickel and cobalt from mined sulphide and laterite ores due to the fact that this process is considered a cost effective option not requiring the high pressures of a pressure leaching process, and not having the disadvantages of pyrometallurgical methods. Heap leaching involves placing the mined ore in a heap and allowing an acid solution such as sulphuric acid, to percolate through the heap to leach out the nickel and cobalt. The solution containing the nickel and cobalt (referred to as the pregnant solution) continues percolating through the crushed ore until it reaches the bottom of the heap where it drains into a storage pond. The process can typically take about 3 to 24 months.

The pregnant solution is subject to further treatment steps which may include the steps of neutralisation to remove the excess acid, iron removal to reduce the amount of iron in the pregnant solution, followed by a solid-liquid separation step. A final precipitation step, using a precipitant such as magnesia or lime, produces a product commonly known as mixed hydroxide product (MHP). The product comprises a number of hydroxide compounds similar to $\text{Ni}_5(\text{OH})_8\text{SO}_4$ and $\text{Co}_5(\text{OH})_8\text{SO}_4$, and usually also contains other compounds such as carbonates, as well as a number of impurities. MHP produced from magnesia typically has a cobalt and nickel content of about 35 to 42%, a manganese content of about 3-7%, a magnesium content of about 3-5%, an iron content of about 0.1-3%, and a sulphur content of about 2-6%. Other impurities such as zinc, copper, uranium, cadmium, aluminium, chromium, silicon, in lesser amounts could also be present. In the case of lime being used as a precipitant in the precipitation step, there are usually larger quantities of calcium in the precipitate (MHP) and in such a case the MHP would have significantly less commercial value.

MHP is also referred to as mixed nickel/cobalt hydroxide product, mixed metal hydroxide, mixed hydroxy sulphate, mixed hydroxide intermediate and various other similar descriptions.

Since MHP is soluble in sulphuric acid, it has the benefit of being readily reprocessible. This production of a precipitate (MHP) is a decoupling step which allows for a natural disconnect between the "front", leaching step, and the "back", refinery, of the plant and hence favours improved process availability over certain other methods.

MHP requires further processing in order to extract nickel and cobalt in a suitable form for industrial use, since the presence of the impurities does not allow for the easy extraction of nickel and cobalt from the MHP. The present techniques for further processing of MHP require stringent conditions such as smelting and leaching to extract nickel and cobalt from the MHP due to the presence of the undesirable impurity elements which make the further processing difficult. As a result, the further processing of the MHP is typically only undertaken by specialist treatment plants in a few parts of the world. There are difficulties with these known methods in that they are costly, and the recovery of some of the valuable metals is difficult and sub-optimal, for example in the smelting process loss of a significant amount of cobalt occurs.

Therefore there is a need for a process to treat the MHP to render it suitable for further processing by conventional methods such as electrowinning and hydrogen reduction to extract the nickel (or cobalt).

References to prior art in this specification are provided for illustrative purposes only and are not to be taken as an admission that such prior art is part of the common general knowledge in Australia or elsewhere.

It should be noted that throughout the specification reference to composition as a percentage is reference to percentage by weight unless otherwise specified.

Summary of the Invention

The present invention provides a method for the treatment of mixed hydroxide product (MHP) produced in a metal extraction process, the method comprising the steps of:

- i) treating the MHP with a first acid solution at a pH in the range of 4 to 8 as a first redissolution step;
- ii) separating a first liquor formed in the first redissolution step from a first residue formed in this step; and
- 5 iii) treating the first residue with a second acid solution at a pH in the range of 0.5 to 4 as a second redissolution step.

Preferably the MHP is a mixed nickel/cobalt hydroxide product. Preferably the metal extraction process is a nickel and/or cobalt extraction process.

- 10 Preferably the metal extraction process is a leaching process. The MHP can be produced from the leaching of laterite or sulphide ores, or of sulphide concentrates, or of alloys, by heap leaching, atmospheric tank or vat leaching, or by various pressure leaching processes.

- 15 Preferably the first acid solution is a spent nickel electrolyte solution, typically spent nickel electrolyte solution from a nickel electrowinning process step. As an alternative, the first acid solution may be a sulphuric acid solution.

- 20 Preferably the first redissolution step takes place at a pH in the range of about 4 to 7. More preferably, the pH is about 4.2 to 6.2. Preferably the first redissolution step takes place at a temperature in the range of about 20°C to 100°C. More preferably the first redissolution step takes place at about 70°C.

- 25 The first liquor typically comprises dissolved nickel and cobalt, together with dissolved impurities of zinc, manganese, magnesium, uranium, and a small amount of calcium, lead and silicon. Insignificant levels of copper, iron, chromium and aluminium may also exist in this first liquor. The first liquor is preferably passed to a further post-treatment step comprising one or more solvent extraction steps to remove the impurities to leave an electrolyte solution which is suitable for passing to an electrowinning circuit, or to a
- 30 hydrogen reduction step. In the case of nickel recovery, cobalt is removed as an impurity to leave a nickel electrolyte solution which is suitable for passing to a nickel electrowinning circuit, or to a nickel hydrogen reduction step. The

post-treatment step may also comprise an ion exchange step, typically in the case of a cobalt electrolyte, for the removal of impurity nickel levels. In the case of a nickel extraction process, cobalt may be removed as an impurity employing a second solvent extraction step to produce a nickel electrolyte solution. Prior to passing of the nickel electrolyte solution to the electrowinning or hydrogen reduction step, the nickel electrolyte solution may be passed through activated carbon filters for removal of organics and oils.

The first redissolution step allows for the at least partial removal of the impurities of copper, iron, aluminium, chromium, and some manganese and silicon, since the first four of these impurities do not dissolve in the first acid solution, and report to the first residue. The first residue is separated from the first liquor (pregnant solution) using typically a thickener, or a decanter centrifuge or similar device, prior to treating with the second acid solution in the second redissolution step. The second redissolution step is carried out at a lower pH than the first redissolution step which ensures that a number of the impurities remaining in the residue (and which were not dissolved in the first redissolution step) will now be dissolved along with most of the valuable nickel and cobalt.

20

The second acid solution is preferably a spent nickel electrolyte solution, typically spent nickel electrolyte solution from a nickel electrowinning process step. As an alternative, the second acid solution may be a sulphuric acid solution.

25

Preferably the second redissolution step takes place at a pH in the range of about 1.0 to 2.5. More preferably, the pH is about 1.5 to 2.0. Preferably the second redissolution step takes place at a temperature in the range of about 20°C to 100°C. More preferably the second redissolution step takes place at about 70°C.

30

Following treatment of the first residue with the second acid solution in the second redissolution step, a slurry is formed comprising dissolved impurities

(together with dissolved nickel) as well as some insoluble substances. Preferably the slurry is then passed to the first redissolution step together with the MHP for further nickel recovery, as already described above. The pH of the first redissolution step following addition of the slurry is allowed to slowly
5 return to its desired range of 4 to 8. As will be appreciated, after the addition of the slurry from a step involving a lower pH, the incoming slurry will initially lower the pH of the mixture in the first redissolution step, before it can slowly rise to the desired pH of the first redissolution step.

- 10 If copper is present in significant concentrations, the first residue is preferably passed to a copper leach step and optionally a copper removal step.

The first residue may be treated with sulphuric acid solution in the copper leach step, at a pH of about 2 to 6.2. Preferably the pH is about 3.0 to 5.0 to
15 form a copper rich leachate. The copper rich leachate preferably passes to a copper removal step to remove the copper metal and form a copper barren liquor. Copper barren liquor from the copper removal step can optionally be returned to the second redissolution step, and the method of the invention repeated as previously described. The copper depleted residue from the
20 copper leach step may be treated with strong acid liquor to form a slurry from which contained nickel and cobalt may be recovered. The slurry from this acid leach can be introduced into an iron removal step so that impurities such as iron, aluminium, chromium, silicon and so on, can be removed from the circuit.

25

The method may also preferably comprise a pre-treatment conditioning step prior to the first redissolution step to at least partially remove magnesium from the MHP used in the first redissolution step. In this way, a treated MHP is formed which is purer than the MHP used in the first redissolution step.

30

The conditioning step preferably comprises contacting MHP with a pre-treatment acid solution. Preferably the pre-treatment acid solution is sulphuric acid. Preferably the conditioning step is carried out at a temperature of about

40°C to 100°C. More preferably, the conditioning step is carried out at a temperature of about 60°C to 80°C. The conditioning step is usually undertaken at a pH of between about 0.2 and 1.5 pH units below that employed in the precipitation of the MHP in the first instance. Since the pH of the precipitation of the MHP is carried out in the range of 7.0 to 8.5, the conditioning step is usually undertaken at a pH in the range of about 5.5 to 8.3. The concentration of the acid solution employed may be in the range from 1% to 98%, with retention times of between 0.25 and 8 hours.

10 The present invention also provides a method for the treatment of mixed hydroxide product (MHP) produced in a metal extraction process, the method comprising contacting the MHP with an acid solution at elevated temperature to at least partially remove magnesium from the MHP to form treated MHP of reduced magnesium content (a purer MHP). This treated reduced
15 magnesium content MHP is then passed to the first redissolution step in accordance with the invention as previously described.

Preferably the MHP is a mixed nickel/cobalt hydroxide product. Preferably the metal extraction process is a nickel and/or cobalt extraction process.
20 Preferably the metal extraction process is a leaching process. The MHP is typically produced from the leaching of laterite or sulphide ores, or of sulphide concentrates, or of alloys.

Preferably the acid solution is a solution of sulphuric acid and the contacting
25 or conditioning step is carried out at a temperature of about 40°C to 100°C. More preferably, the contacting or conditioning step is carried out at a temperature of about 60°C to 80°C. The contacting or conditioning step is usually undertaken at a pH of between about 0.2 and 1.5 pH units below that employed in the precipitation of the MHP in the first instance. The
30 concentration of the acid solution may be in the range from 1% to 98%, with retention times of between 0.25 and 8 hours.

The present invention further provides a method for the purification of a nickel electrolyte stream comprising the steps of:

- 5 a) treating mixed hydroxide product (MHP) with a first acid solution at a pH in the range of 4 to 8 as a first redissolution step;
- b) separating a nickel electrolyte stream formed in the first redissolution step from a first residue formed in this step; and
- 10 c) treating the nickel electrolyte stream with two sequential solvent extraction steps, namely a first solvent extraction step and a second solvent extraction step.

Preferably the MHP is pre-treated in a conditioning step to at least partially remove magnesium from the MHP prior to step a) in the method.

- 15 Preferably the first solvent extraction step allows for quantitative removal of zinc, uranium and a majority of the manganese. Preferably the first solvent extraction step also allows for the partial removal of calcium. Preferably the second solvent extraction step allows for the substantial removal of the remaining manganese and cobalt. A first extractant used in the first solvent
- 20 extraction step may be of the same composition as the second extractant used in the second solvent extraction step, or they could be different.

The present invention further provides a method for the purification of a cobalt electrolyte stream comprising the steps of:

- 25 a) treating mixed hydroxide product (MHP) with a first acid solution at a pH in the range of 4 to 8 as a first redissolution step;
- b) separating a cobalt electrolyte stream formed in the first redissolution step from a first residue formed in this step; and
- 30 c) treating the cobalt electrolyte stream with two sequential extraction steps, namely a first solvent extraction step followed by an ion exchange step.

Preferably the MHP is pre-treated in a conditioning step to at least partially remove magnesium from the MHP prior to step a) in the method.

5 Preferably the first solvent extraction step allows for the quantitative removal of zinc and uranium, and substantial removal of manganese. Preferably the first solvent extraction step also allows for the partial removal of calcium. Preferably the ion exchange step employs a resin displaying a high selectivity of nickel over cobalt, for example where the resin has bis-picolylamine functional groups, such as resins sold under the trade marks "TP220" and
10 "M4195". In this way the nickel can be "trimmed" to provide a suitable feed to be passed to a cobalt electrowinning step. In the case of cadmium being present in a cobalt electrolyte stream, the cadmium can also be at least partially removed by ion exchange.

15 Following treatment of either the nickel or cobalt electrolyte streams by means of solvent extraction and/or ion exchange, activated carbon may be used to remove a variety of non-metal impurities. Several types of carbon may be required to provide acceptable electrolyte purity for a metal-winning step.

20

It should be noted that by forming nickel or cobalt electrolyte streams in step b) as described above for each respective method, and subsequent treatment with solvent extraction and/or ion exchange, improved nickel or cobalt ratio to impurities is achieved which is beneficial in later electro-winning steps.

25

It should be understood that the word "electrolyte" is used in the specification to refer to the aqueous solution resulting from the redissolution steps described in the specification, whether or not this solution is ultimately treated in a metal winning step such as an electrowinning step.

30

Throughout the specification, unless the context requires otherwise, the word "comprise" or variations such as "comprises" or "comprising", will be understood to imply the inclusion of a stated integer or group of integers but

not the exclusion of any other integer or group of integers. Likewise the word "preferably" or variations such as "preferred", will be understood to imply that a stated integer or group of integers is desirable but not essential to the working of the invention.

5 **Brief Description of the Drawings**

The nature of the invention will be better understood from the following detailed description of embodiments of a method for the treatment of mixed hydroxide product (MHP) produced in a metal extraction process according to the invention, given by way of example only, with reference to the
10 accompanying drawings in which:

Figure 1 is a flow diagram showing the formation of mixed hydroxide product (MHP) in a typical metal heap leaching process;

15 Figure 2 is a flow diagram of a preferred method according to the invention for the treatment of mixed hydroxide product (MHP) from the process of Figure 1 for the recovery of nickel; and

Figure 3 is a flow diagram of a preferred method according to the invention for
20 the treatment of mixed hydroxide product (MHP) from the process of Figure 1 for the recovery of cobalt.

The letters "S" and "L" in the Figures represent "solid" and "liquid" respectively.

Detailed Description of Preferred Embodiments

25 A preferred method for the treatment of mixed hydroxide product (MHP) 10 produced in a metal extraction process, in this case a nickel and/or cobalt heap leaching process as shown in Figure 1, is illustrated in Figure 2. The preferred method comprises the steps of treating the MHP 10 (or treated purer MHP 11) with a first acid solution being sulphuric acid 12 (or shown
30 alternatively as a new stream of sulphuric acid 13) at a "near neutral" pH, that is at a pH of about 4 to 8, and more specifically at a pH of about 4.2 to 6.2.

This is referred to as the first redissolution step 14. The sulphuric acid 12 is typically of a concentration of between 0.1 and 98%.

As an alternative, the first acid solution may be spent nickel electrolyte solution 24, typically nickel electrolyte solution from a nickel electrowinning process step 26, as shown in Figure 2. In the case of cobalt recovery, the first acid solution may be spent cobalt electrolyte solution 28, typically cobalt electrolyte solution from a cobalt electrowinning process step 30, as shown in Figure 3.

10

A first liquor 16 (also referred to as nickel electrolyte stream or cobalt electrolyte stream) is separated from a first residue 18 formed in the first redissolution step 14. The first residue 18 is treated with a second acid solution being sulphuric acid 20 (at a concentration of about 0.1 to 98%) at a pH in the range of 0.5 to 4.0, and more preferably at a pH of about 1.5 to 2.0. This is referred to as the second redissolution step 22.

As an alternative, the second acid solution may be spent nickel electrolyte solution 24, typically spent nickel electrolyte solution 24 from a nickel electrowinning process step 26. In the case of cobalt recovery, the first acid solution may be spent cobalt electrolyte solution 28.

The first liquor 16 comprises dissolved nickel and cobalt, together with dissolved impurities of zinc, manganese, magnesium, uranium, and a small amount of calcium, and possibly cadmium. The first liquor 16 is preferably passed to a post-treatment step 29 comprising a first solvent extraction step 32 and a second solvent extraction step 34 to remove the impurities (and cobalt in the case of nickel recovery as shown in Figure 2) to leave a nickel electrolyte solution 36 which is suitable for passing to the nickel electrowinning step 26, or to a nickel hydrogen reduction step 38. After removal of the impurities, and prior to passing of the nickel electrolyte solution 36 to the electrowinning 26 or hydrogen reduction step 38, the nickel electrolyte solution 36 may be passed through carbon filters 40 for removal of

organics and oils. As shown in Figure 2, substantially all of the cobalt is removed together with any remaining manganese in the solvent extraction step 34 to leave a manganese/cobalt "semi-product" 42.

- 5 In the case of cobalt recovery (where nickel removal is required as shown in Figure 3), the ion exchange step 35 is used to remove nickel to leave a nickel "semi-product" 44. As in the case of nickel recovery, and as described in the relation to Figure 2, after removal of the impurities, and prior to passing of the cobalt electrolyte solution 46 to the electrowinning 30 or hydrogen reduction
10 step 31, the cobalt electrolyte solution 46 may be passed through carbon filters 40 for removal of organics and a variety of other impurities that impact product morphology.

- The first redissolution step 14 allows for the removal of the impurities of
15 copper, iron, aluminium, chromium, and some manganese plus silicon since these impurities do not dissolve in the first acid solution, and report to the first residue 18. The first residue is typically settled in a thickener, or a decanter centrifuge, prior to treating with the second acid solution 20 in the second redissolution step 22. The second redissolution step 22 is carried out at a
20 lower pH than the first redissolution step 14 which ensures that a number of the impurities remaining in the residue 18 (and which were not dissolved in the first redissolution step 14) will now be dissolved.

- Following treatment of the first residue 18 with the second acid solution in the
25 second redissolution step, a slurry 48 is formed comprising dissolved impurities (together with dissolved cobalt and nickel) as well as some insoluble substances. Preferably the slurry 48 is then passed to the first redissolution step 14 together with the MHP 10 for further nickel recovery, as already described above. The pH of the first redissolution step 14 is then
30 preferably allowed to slowly return to its desired range of 4 to 8. As will be appreciated, after the addition of the slurry 48 from a step involving a lower pH, the incoming slurry 48 will initially lower the pH of the mixture in the first redissolution step 14, before it can slowly rise to the desired pH by the

simultaneous addition of fresh MHP of the first redissolution step 14. It has been found that this two step redissolution process optimises the recovery of nickel to the nickel electrolyte solution and/or cobalt to the cobalt electrolyte, which is passed for final treatment and recovery of the nickel (and/or cobalt) to the electrowinning circuit, or to the hydrogen reduction process. At the same time, impurities which are undesirable for the electrowinning and hydrogen reduction processes are largely removed thereby facilitating the ease of operation of these two processes.

10 As an alternative to the treatment of the first residue 18 with the thickener, or the separation in a decanter centrifuge, the first residue 18 (or a portion thereof) may be passed to a copper leach step 50 and/or a copper removal step 52.

15 If copper is present in significant concentrations, then a copper leach step 50 is desirable. The first residue 18 is treated with spent nickel electrolyte solution 24 (or spent cobalt electrolyte solution 28) in the copper leach step 50 or new sulphuric acid 70, at a pH of about 3 to 6.2. Preferably the pH is about 3.0 to 5.0. The copper rich leachate 58 passes to the copper removal step 52 for treatment by a suitable means such as solvent extraction, ion exchange, precipitation or cementation, to remove the copper metal. The copper barren liquor 54 from the copper removal step 52 can be returned to the second redissolution step 22, and the method of the invention repeated as previously described. The copper depleted residue 60 remaining after the copper leach step 50 is treated with strong acid liquor 62 (which can be sourced from a variety of process streams) or reprocessed at some appropriate step in the overall flowsheet for recovery of value metals and rejection of iron, aluminium, and so on.

30 It should be noted that the method of the invention can be carried out at temperatures varying from ambient to boiling point of the aqueous fraction, and it has been found that in general fluctuations in temperature do not affect the method and the nature of the resulting nickel electrolyte solution. This is

of course beneficial since it means that the method can be carried out in fluctuating temperature conditions so that there may not be the need to incur costs to keep the temperature at a particular constant level.

- 5 The method may also preferably include a pre-treatment conditioning step 56 prior to the first redissolution step 14 to at least partially remove magnesium from the MHP used in the first redissolution step 14. It should be noted that there is often a high concentration of magnesium in MHP due to the fact that magnesia is used as a precipitant to form MHP in the first instance.

10

The pre-treatment conditioning step 56 comprises treating the MHP 10 to form a treated MHP 11 (a purer MHP) which passes to the first redissolution step 14. The conditioning step 56 comprises contacting the crude MHP 10 with a solution of sulphuric acid at a temperature of about 40°C to 100°C.

- 15 Preferably, the conditioning step 56 is carried out at a temperature of about 60°C to 80°C. The slurry formed in the conditioning step 56 is treated through a filter or other similar device to produce the purer MHP and a solution rich in magnesium is removed and recycled to the MHP precipitation step.

- 20 The conditioning step 56 is normally undertaken at a pH between about 0.2 and 1.5 pH units below that employed in the precipitation of the MHP in the first instance, typically at a pH of about 6.0 to 8.3. Acid concentrations ranging from 1% to 98% can be employed with retention times of between 0.25 and 8 hours. The filtered MHP after conditioning is washed with water to
25 displace the mother liquor and to form the treated MHP 11.

- It is desirable to remove magnesium from MHP prior to commencement of the first redissolution step 14 since it is more difficult to remove magnesium at a later stage due to the fact that magnesium tends to report with nickel and
30 cobalt to the electrolyte liquid stages of the method of the invention. The presence of magnesium can cause problems in the electrolyte solutions and can downgrade the quality of the final product. In relation to the hydrogen

reduction process 38, the presence of magnesium can impact the efficiency of the ammonium sulphate recovery process.

By way of further explanation, if magnesium compounds are present in the MHP they will almost certainly redissolve quantitatively in the MHP redissolution circuit (comprising the first and second redissolution steps). Only very small quantities will survive beyond the copper leach residue. Consequently most of the magnesium entering the circuit in the MHP will report to the electrolyte where it will impact the magnitude of the bleed stream resulting in significant recycle of nickel with increased losses. (The bleed is required to maintain equilibrium of the "inert" elements such as sodium, sulphur and magnesium for example).

Therefore, in order to minimise the transfer of magnesium to the electrolyte loop (the nickel electrowinning step 26), the MHP is pretreated in the conditioning step 56 which is designed to reduce the residual magnesium normally existing in commercial MHP semi-products.

The method also preferably includes a post-treatment step 29 following the first redissolution step 14 to remove all the impurities of zinc, manganese and uranium that would otherwise follow with the nickel and report to the final metal and a majority of the calcium. In the case of cobalt electrowinning a small quantity of manganese is beneficial in the electrolyte.

The preferred post-treatment step 29 includes a first solvent extraction step 32 involving a high 20 - 45% concentration of DEHPA in the diluent and employing a mixer settler type circuit with three or four extract steps, two scrub stages, two or three strip stages and a single wash/ preload stage, and a second solvent extraction step 34 incorporating for example a Cyanex 272 circuit for the removal of all the cobalt and any remaining manganese.

The extract pH is maintained in the pH 1.0 to 2.5 range and typically 1.4 to 2.0, with mixer retention times of approximately 1 to 3 minutes and

temperatures of 20 to 50°C. Under these conditions, only small quantities of cobalt/nickel co-extract with the impurities which are then recovered in the scrub stage along with some of the calcium.

- 5 A strong (0.5-4 molar) acid strip is employed to remove the impurities zinc, manganese, calcium and uranium from the solvent in the first solvent extraction step 32. Finally the solvent is washed and or preloaded with a nickel or cobalt bleed of the electrolyte.
- 10 Where a wash process is adopted instead of a preload then a recycle of a fraction of the extract raffinate to the first step in the first redissolution step 14 can be beneficial in optimising acid consumption in the overall MHP to metal process. However, a preload of the extractant in the first solvent extraction step can eliminate this raffinate recycle and reduce the requirement of
- 15 raffinate acid neutralisation.

In order to further describe the preferred process of the invention, a typical assay of MHP following treatment with the conditioning step was as follows:

20

Table 1

	<u>%</u>
Ni + Co	42 – 47
Mn	5.0
Fe	0.1
Cu	0.1
Ca	0.2
Al	0.1
S	5.8
Mg	0.5
Zn	0.3

This MHP had previously undergone a conditioning step which reduced the magnesium from 3 – 5% to 0.5%.

A typical assay for spent nickel electrolyte solution 24 which is reacted with MHP in the first redissolution step 14 is given below:

Table 2

	<u>g/L</u>
Ni	58
Mg	3
Mn	<2 PPM
Na	17
Ca	20 – 50 PPM
Co	<1 PPM
Pb	5 PPM
U	<0.1 PPM
Zn	<0.2 PPM
Fe	<0.2 PPM
Cu	<0.2 PPM
H ₂ SO ₄	49.5

5

The first redissolution step 14 was operated at 70°C.

A typical sample of first liquor 16 having a pH of 4.5 to 5.5 (pH measured at ambient temperature 25°C) from the first redissolution step is given below:

10

Table 3

	<u>g/L</u>
Ni	90 – 92
Mg	4.2
Mn	1.9
Zn	0.25
Na	17
Co	1.35
Pb	<1 PPM
U	10 PPM

- 19 -

Ca	0.22
Cu	<0.2 PPM
Fe	0.4 PPM

The liquor in Table 3 is eminently suited for the quantitative removal of zinc and uranium and near quantitative removal of manganese and calcium using the solvent extraction step(s) described earlier. A part of the raffinate arising from the solvent extraction step(s) can be blended with the spent electrolyte 24 from Table 2, if required, as a source of sulphuric acid in the redissolution of MHP (not shown in the drawings). Alternatively, by employing an extractant preload step with either nickel or cobalt, the release of acid to the electrolyte circuit can be reduced significantly. This in turn reduces the magnitude of the bleed of nickel or of cobalt from the electrolyte (not shown in the Figures).

The first residue 18 from the first redissolution circuit (comprising between 4 to 7% of the original MHP dry feed) assayed as follows:

Table 4

	<u>%</u>
Ni	25 – 28
Cu	1.5 – 1.8
Fe	1.5 – 1.8
Mn	31 – 35
Al	1.5 – 1.6
Mg	0.1
S	3

As can be seen, the impurities of copper, iron, aluminium, and manganese have been collected in this first residue instead of passing into the first liquor 16 (as shown in Table 3). This residue in Table 4 can be processed to remove copper as described earlier and then bled from the circuit for further treatment to recover residual nickel and cobalt prior to disposal for rejection of contained iron, aluminium, chromium and manganese.

As can be seen in the Figures, the present invention also provides a method for the treatment of mixed hydroxide product (MHP) 10 produced in a metal extraction process, the method comprising contacting the MHP 10 with an acid solution at elevated temperature to at least partially remove magnesium from the MHP to form MHP 11 of treated reduced magnesium content (a purer MHP 11). This treated reduced magnesium content MHP 11 is then passed to the first redissolution step 14 in accordance with the invention as previously described.

10

As can be seen in Figure 2, the invention also provides a method for the purification of a nickel electrolyte stream 16 comprising the steps of a) treating mixed hydroxide product (MHP) 10 or 11 with a first acid solution 12 at a pH in the range of 4 to 8 as a first redissolution step 14; b) separating a nickel electrolyte stream 16 formed in the first redissolution step 14 from a first residue 18 formed in this step; and c) treating the nickel electrolyte stream 16 with two sequential solvent extraction steps, namely a first solvent extraction step 32 and a second solvent extraction step 34.

15

As can be seen in Figure 3, the invention also provides a method for the purification of a cobalt electrolyte stream 16 comprising the steps of a) treating mixed hydroxide product (MHP) 10 or 11 with a first acid solution 12 at a pH in the range of 4 to 8 as a first redissolution step 14; b) separating a cobalt electrolyte stream 16 formed in the first redissolution step 14 from a first residue 18 formed in this step; and c) treating the cobalt electrolyte stream 16 with two sequential extraction steps, a first solvent extraction step 32 followed by an ion exchange step 35.

20

Now that preferred embodiments of a method for the extraction of nickel and cobalt according to the invention have been described in detail, it will be apparent that the embodiments provide a number of advantages over the prior art, including the following:

30

- (i) The method produces a nickel or cobalt electrolyte solution which has improved properties for use in conventional electrowinning or hydrogen reduction circuits due to the removal of undesirable impurities.
 - 5 (ii) The method provides a way of treating MHP for use in nickel or cobalt recovery without the more stringent conditions required in present methods;
 - (iii) By using the conditioning step to pre-treat the MHP, the inefficiencies and problems associated with magnesium in nickel or cobalt electrolyte solutions are substantially reduced.
10
 - (iv) By using the first solvent extraction step (common to both nickel and cobalt electrolyte streams) for the quantitative removal of zinc, uranium as well as some calcium and manganese, and recycling a fraction of the raffinate, where relevant, to the MHP first redissolution step, the optimisation of acid utilisation in this redissolution step can be improved. This recycle may not be required if an adequate nickel or cobalt preload of the extractant in the first solvent extraction step is performed.
15
- 20 It will be readily apparent to persons skilled in the relevant art that various modifications and improvements may be made to the foregoing embodiments, in addition to those already described, without departing from the basic inventive concepts of the present invention. Therefore, it will be appreciated that the scope of the invention is not limited to the specific embodiments
25 described.

The Claims defining the Invention are as follows:

1. A method for the treatment of mixed hydroxide product (MHP) produced in a metal extraction process, the method comprising the steps of:
 - 5 i) treating the MHP with a first acid solution at a pH in the range of 4 to 8 as a first redissolution step;
 - ii) separating a first liquor formed in the first redissolution step from a first residue formed in this step; and
 - 10 iii) treating the first residue with a second acid solution at a pH in the range of 0.5 to 4 as a second redissolution step.
2. A method for the treatment of MHP as defined in claim 1, wherein the MHP is a mixed nickel/cobalt hydroxide product.
3. A method for the treatment of MHP as defined in any one of the preceding
15 claims, wherein the metal extraction process is a nickel and/or cobalt extraction process.
4. A method for the treatment of MHP as defined in any one of the preceding claims, wherein the metal extraction process is a leaching process.
5. A method for the treatment of MHP as defined in any one of the preceding
20 claims, wherein MHP is produced from the leaching of laterite or sulphide ores, or of sulphide concentrates, or of alloys, by heap leaching, atmospheric tank or vat leaching, or by various pressure leaching processes.
6. A method for the treatment of MHP as defined in any one of the preceding claims, wherein the first acid solution is a spent nickel electrolyte solution.
- 25 7. A method for the treatment of MHP as defined in claim 6, wherein the spent nickel electrolyte solution is obtained from a nickel electrowinning process step.
8. A method for the treatment of MHP as defined in any one claims 1 to 5, wherein the first acid solution is a sulphuric acid solution.

9. A method for the treatment of MHP as defined in any one of the preceding claims, wherein the first redissolution step takes place at a pH in the range of about 4 to 7.

10. A method for the treatment of MHP as defined in claim 9, wherein the first
5 redissolution step takes place at a pH in the range of about 4.2 to 6.2.

11. A method for the treatment of MHP as defined in any one of the preceding claims, wherein the first redissolution step takes place at a temperature in the range of about 20°C to 100°C.

12. A method for the treatment of MHP as defined in claim 11, wherein the
10 first redissolution step takes place at a temperature of about 70°C.

13. A method for the treatment of MHP as defined in any one of the preceding claims, wherein the first liquor comprises dissolved nickel and cobalt, together with dissolved impurities of zinc, manganese, magnesium, uranium and calcium.

14. A method for the treatment of MHP as defined in any one of the preceding
15 claims, wherein the first liquor is passed to a further post-treatment step comprising one or more solvent extraction steps to remove impurities to leave an electrolyte solution which is suitable for passing to an electrowinning circuit, or to a hydrogen reduction step.

15. A method for the treatment of MHP as defined in claim 14, wherein the
20 post-treatment step in the case of a cobalt electrolyte further comprises an ion exchange step for the removal of impurity nickel levels.

16. A method for the treatment of MHP as defined in claim 14, wherein in the
25 case of a nickel extraction process, cobalt is also removed as an impurity employing a second solvent extraction step to produce a nickel electrolyte solution which is passed through carbon filters prior to passing to the electrowinning circuit or hydrogen reduction step.

17. A method for the treatment of MHP as defined in any one of the preceding claims, wherein the first residue is separated from the first liquor using a

thickener, or a decanter centrifuge or similar device, prior to treating with the second acid solution in the second redissolution step.

18. A method for the treatment of MHP as defined in any one of the preceding claims, wherein the second acid solution is a spent nickel
5 electrolyte solution.

19. A method for the treatment of MHP as defined in claim 18, wherein the spent nickel electrolyte solution is obtained from a nickel electrowinning process step.

20. A method for the treatment of MHP as defined in any one of claims 1 to
10 17, wherein the second acid solution is a sulphuric acid solution.

21. A method for the treatment of MHP as defined in any one of the preceding claims, wherein the second redissolution step takes place at a pH in the range of about 1.0 to 2.5.

22. A method for the treatment of MHP as defined in claim 21, wherein the
15 second redissolution step takes place at a pH in the range of about 1.5 to 2.0

23. A method for the treatment of MHP as defined in any one of the preceding claims, wherein the second redissolution step takes place at a temperature in the range of about 20°C to 100°C.

24. A method for the treatment of MHP as defined in claim 23, wherein the
20 second redissolution step takes place at about 70°C.

25. A method for the treatment of MHP as defined in any one of the preceding claims, wherein following treatment of the first residue with the second acid solution in the second redissolution step, a slurry is formed which is passed to the first redissolution step together with the MHP for further nickel recovery.

25 26. A method for the treatment of MHP as defined in claim 25, wherein the pH of the first redissolution step following addition of the slurry is allowed to slowly return to its desired range of 4 to 8.

27. A method for the treatment of MHP as defined in any one of claims 1 to 16, wherein the first residue is passed to a copper leach step.

28. A method for the treatment of MHP as defined in claim 27, wherein the first residue is treated with sulphuric acid solution in the copper leach step.

29. A method for the treatment of MHP as defined in claim 27 or claim 28, wherein the copper leach step is carried out at a pH of about 2 to 6.2.

5 30. A method for the treatment of MHP as defined in claim 29, wherein the copper leach step is carried out at a pH of about 3.0 to 5.0 to form a copper rich leachate and a copper depleted residue.

31. A method for the treatment of MHP as defined in claim 30, wherein the copper rich leachate passes to a copper removal step to remove the copper
10 metal and form a copper barren liquor.

32. A method for the treatment of MHP as defined in claim 31, wherein the copper barren liquor from the copper removal step is returned to the second redissolution step.

33. A method for the treatment of MHP as defined in any one of claims 30 to
15 32, wherein the copper depleted residue is treated with strong acid liquor to form a slurry from which contained nickel and cobalt may be recovered.

34. A method for the treatment of MHP as defined in claim 33, wherein the slurry is introduced into an iron removal step.

35. A method for the treatment of MHP as defined in any one of the
20 preceding claims, wherein the method also comprises a pre-treatment conditioning step prior to the first redissolution step to at least partially remove magnesium from the MHP used in the first redissolution step.

36. A method for the treatment of MHP as defined in claim 35, wherein the pre-treatment conditioning step comprises contacting MHP with a pre-
25 treatment acid solution.

37. A method for the treatment of MHP as defined in claim 36, wherein the pre-treatment acid solution is sulphuric acid.

38. A method for the treatment of MHP as defined in claim 36 or claim 37, wherein the pre-treatment conditioning step is carried out at a temperature of about 40°C to 100°C.

39. A method for the treatment of MHP as defined in claim 38, wherein the
5 pre-treatment conditioning step is carried out at a temperature of about 60°C to 80°C.

40. A method for the treatment of MHP as defined in any one of claims 35 to 39, wherein the pre-treatment conditioning step is undertaken at a pH of between about 0.2 and 1.5 pH units below that employed in the precipitation
10 of the MHP in the first instance.

41. A method for the treatment of MHP as defined in claim 40, wherein the pre-treatment conditioning step is usually undertaken at a pH in the range of about 5.5 to 8.3.

42. A method for the treatment of MHP as defined in any one of claims 36 to
15 41, wherein the concentration of the pre-treatment acid solution is in the range from 1% to 98%.

43. A method for the treatment of MHP as defined in claim 42, wherein the retention time is between 0.25 and 8 hours.

44. A method for the treatment of mixed hydroxide product (MHP) produced
20 in a metal extraction process, the method comprising contacting the MHP with an acid solution at elevated temperature to at least partially remove magnesium from the MHP to form treated MHP of reduced magnesium content.

45. A method for the treatment of MHP as defined in claim 44, wherein the
25 MHP is a mixed nickel/cobalt hydroxide product.

46. A method for the treatment of MHP as defined in claim 44 or claim 45, wherein the metal extraction process is a nickel and/or cobalt extraction process.

47. A method for the treatment of MHP as defined in any one of claims 44 to 46, wherein the MHP is produced from the leaching of laterite or sulphide ores, or of sulphide concentrates, or of alloys.

48. A method for the treatment of MHP as defined in any one of claims 44 to 5 47, wherein the acid solution is a solution of sulphuric acid.

49. A method for the treatment of MHP as defined in any one of claims 44 to 48, wherein the contacting step is carried out at a temperature of about 40°C to 100°C.

50. A method for the purification of a nickel electrolyte stream comprising the 10 steps of:

- a) treating mixed hydroxide product (MHP) with a first acid solution at a pH in the range of 4 to 8 as a first redissolution step;
- b) separating a nickel electrolyte stream formed in the first redissolution step from a first residue formed in this step; and
- 15 c) treating the nickel electrolyte stream with two sequential solvent extraction steps, namely a first solvent extraction step and a second solvent extraction step.

51. A method for the purification of a nickel electrolyte stream as defined in 20 claim 50, wherein the MHP is pre-treated in a conditioning step to at least partially remove magnesium from the MHP prior to step a) in the method.

52. A method for the purification of a nickel electrolyte stream as defined in claim 50 or claim 51, wherein the first solvent extraction step allows for the quantitative removal of zinc, uranium and a majority of the manganese.

25 53. A method for the purification of a nickel electrolyte stream as defined in claim 52, wherein the first solvent extraction step further allows for the partial removal of calcium.

54. A method for the purification of a nickel electrolyte stream as defined in claim 53, wherein the second solvent extraction step allows for the substantial 30 removal of the remaining manganese and cobalt.

55. A method for the purification of a cobalt electrolyte stream comprising the steps of:

- a) treating mixed hydroxide product (MHP) with a first acid solution at a pH in the range of 4 to 8 as a first redissolution step;
- 5 b) separating a cobalt electrolyte stream formed in the first redissolution step from a first residue formed in this step; and
- c) treating the cobalt electrolyte stream with two sequential extraction steps, namely a first solvent extraction step followed by an ion exchange step.

10

56. A method for the purification of a cobalt electrolyte stream as defined in claim 55, wherein the MHP is pre-treated in a conditioning step to at least partially remove magnesium from the MHP prior to step a) in the method.

57. A method for the purification of a cobalt electrolyte stream as defined in
15 claim 55 or claim 56, wherein the first solvent extraction step allows for the quantitative removal of zinc and uranium, and substantial removal of manganese.

58. A method for the purification of a cobalt electrolyte stream as defined in
20 claim 57, wherein the first solvent extraction step further allows for the partial removal of calcium.

59. A method for the purification of a cobalt electrolyte stream as defined in any one of claims 55 to 58, wherein the ion exchange step employs a resin displaying a high selectivity of nickel over cobalt.

60. A method for the purification of a cobalt electrolyte stream as defined in
25 claim 59, wherein the resin has bis-picolylamine functional groups, for example "TP220" and "M4195".

61. A method for the purification of a cobalt electrolyte stream as defined in claim 55, wherein the activated carbon is used after the ion exchange step to remove a variety of non-metal impurities.

62. A method for the treatment of MHP substantially as herein described with reference to and as illustrated in any one or more of the accompanying drawings.

63. A method for the purification of a nickel electrolyte stream substantially as
5 herein described with reference to and as illustrated in any one or more of the accompanying drawings.

64. A method for the purification of a cobalt electrolyte stream substantially as herein described with reference to and as illustrated in any one or more of the accompanying drawings.

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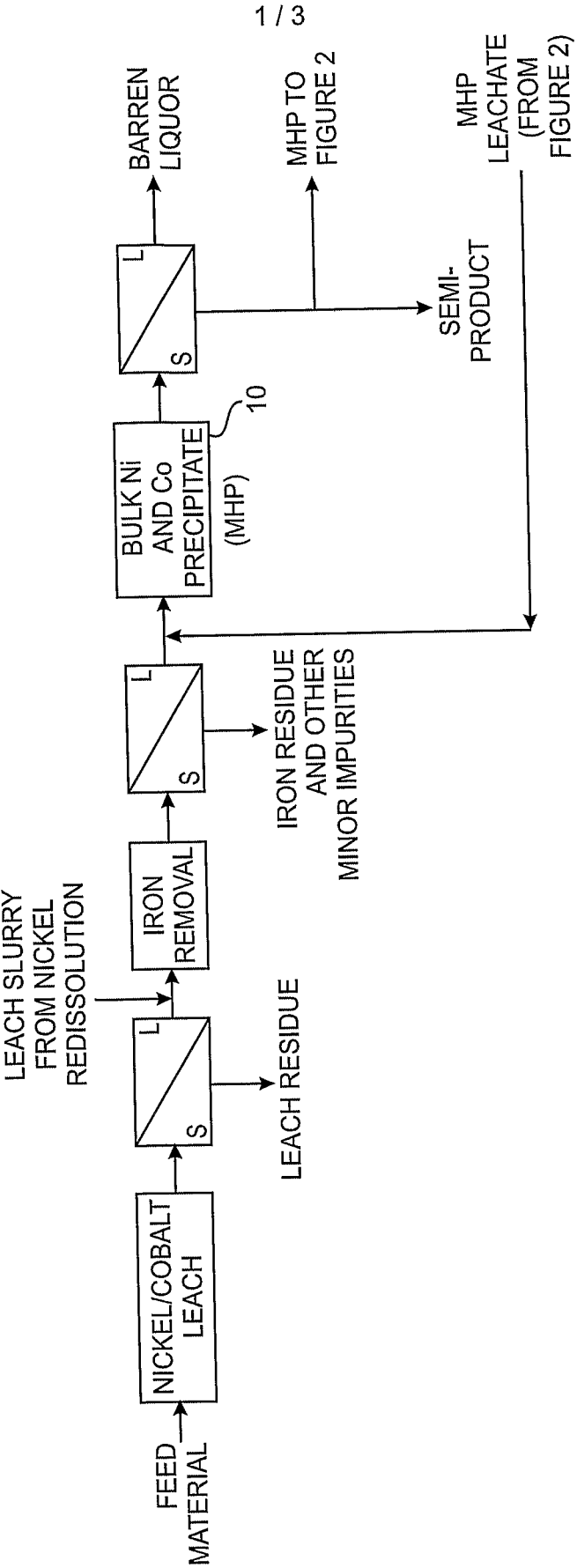


FIGURE 1

2 / 3

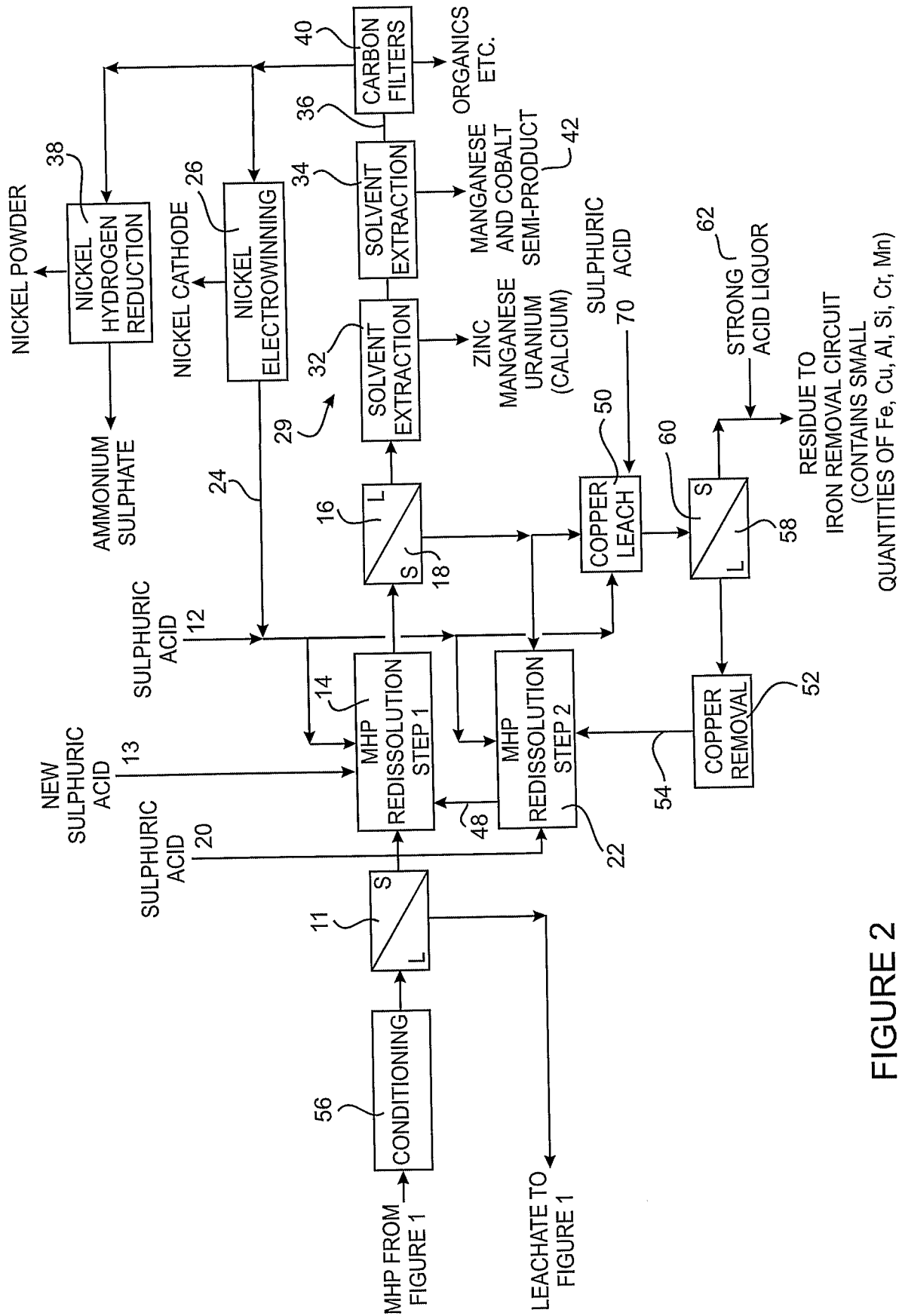


FIGURE 2

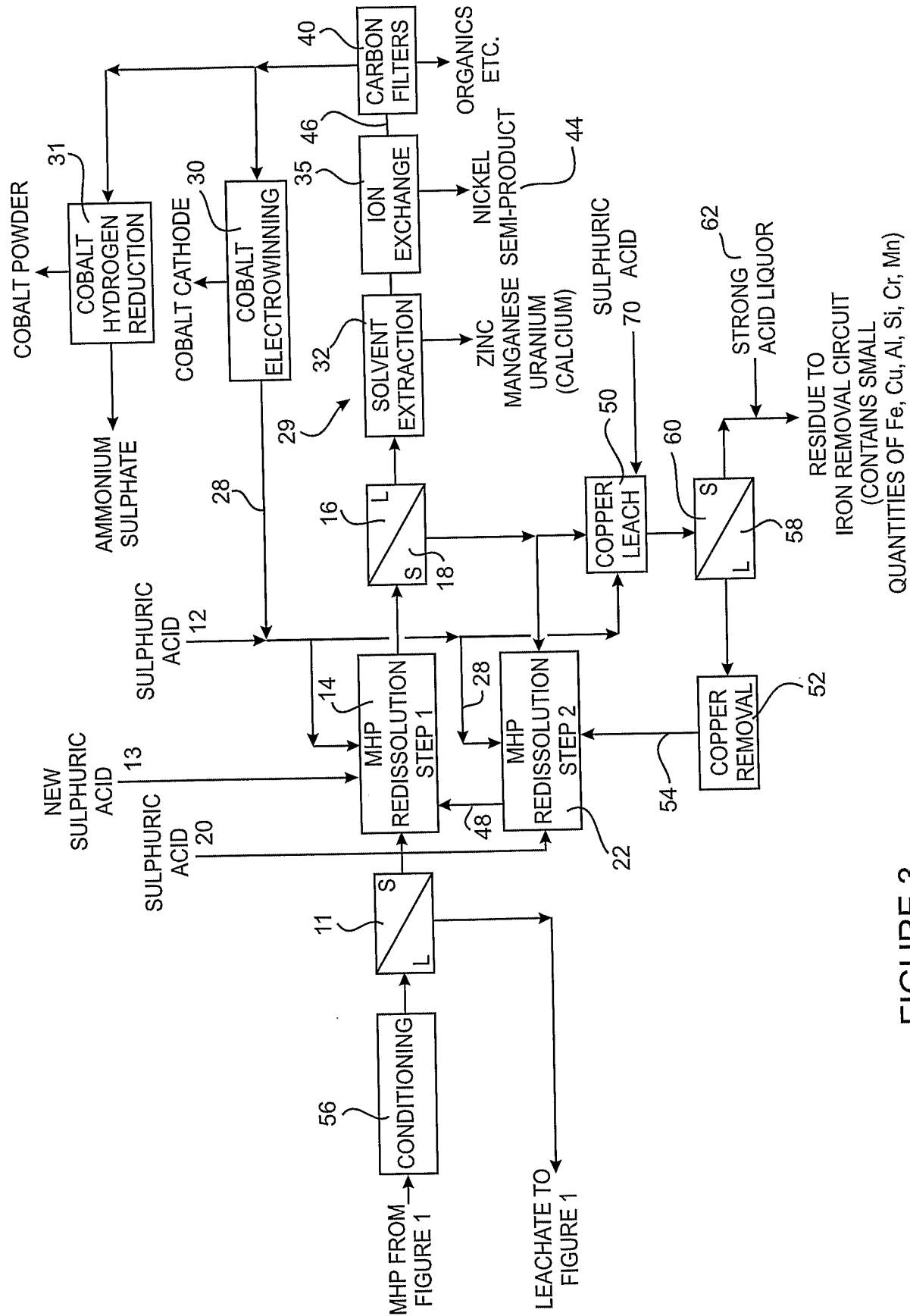


FIGURE 3

INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU2009/000486

A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl.

C22B 3/20 (2006.01)

C22B 3/06 (2006.01)

C22B 3/04 (2006.01)

C22B 23/00 (2006.01)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPOQUE (EPODOC, WPI): IPC C22B 3/20, C22B 23/00, C22B 3/04 & keywords (Laterite OR sulphite OR heterogenite) AND (Nick+ OR cobalt) AND (acid) AND (mixed OR intermediate+ OR metal)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	CA 618826 A (FREEPORT SULPHUR CO) 25 April 1961 Pg 11 lines 21-32, Pg 12 lines 1-14 & figs 3-4	
A	US 2007/0166214 A1 (ANDERSON et al) 19 July 2007 Pg 1 para 2-15, pg 2 para 24 & 28, figs 1-2	
A	US 2004/0052707 A1 (O' CALLAGHAN et al) 18 March 2004 Pg 2 para 17, para 24 -29, pg 3 para 33 - 35, figs 1-3	



Further documents are listed in the continuation of Box C



See patent family annex

* Special categories of cited documents:	
"A" document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"E" earlier application or patent but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"O" document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family
"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search
04 June 2009

Date of mailing of the international search report
23 JUN 2009

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU2009/000486

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☒ Claims Nos.: 62-64
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

The subject matter of claims 62-64 does not comply with Rule 6.2(a) because it relies on references to the drawings. Therefore, the requirements of PCT Rule 6.2(a) are not met and no search has been established for these claims.

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a)

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- ☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- ☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/AU2009/000486

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member			
CA	618826	NONE			
US	2007166214	AU	2004228982	BR	PI0409223
		CN	1774518	EP	1613785
		ZA	200507886	CA	2521888
				WO	2004090176
US	2004052707	AU	73737/01	CA	2444104
		US	2008003154	EP	1409753
				WO	0196621
				ZA	200302850
Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.					
END OF ANNEX					