



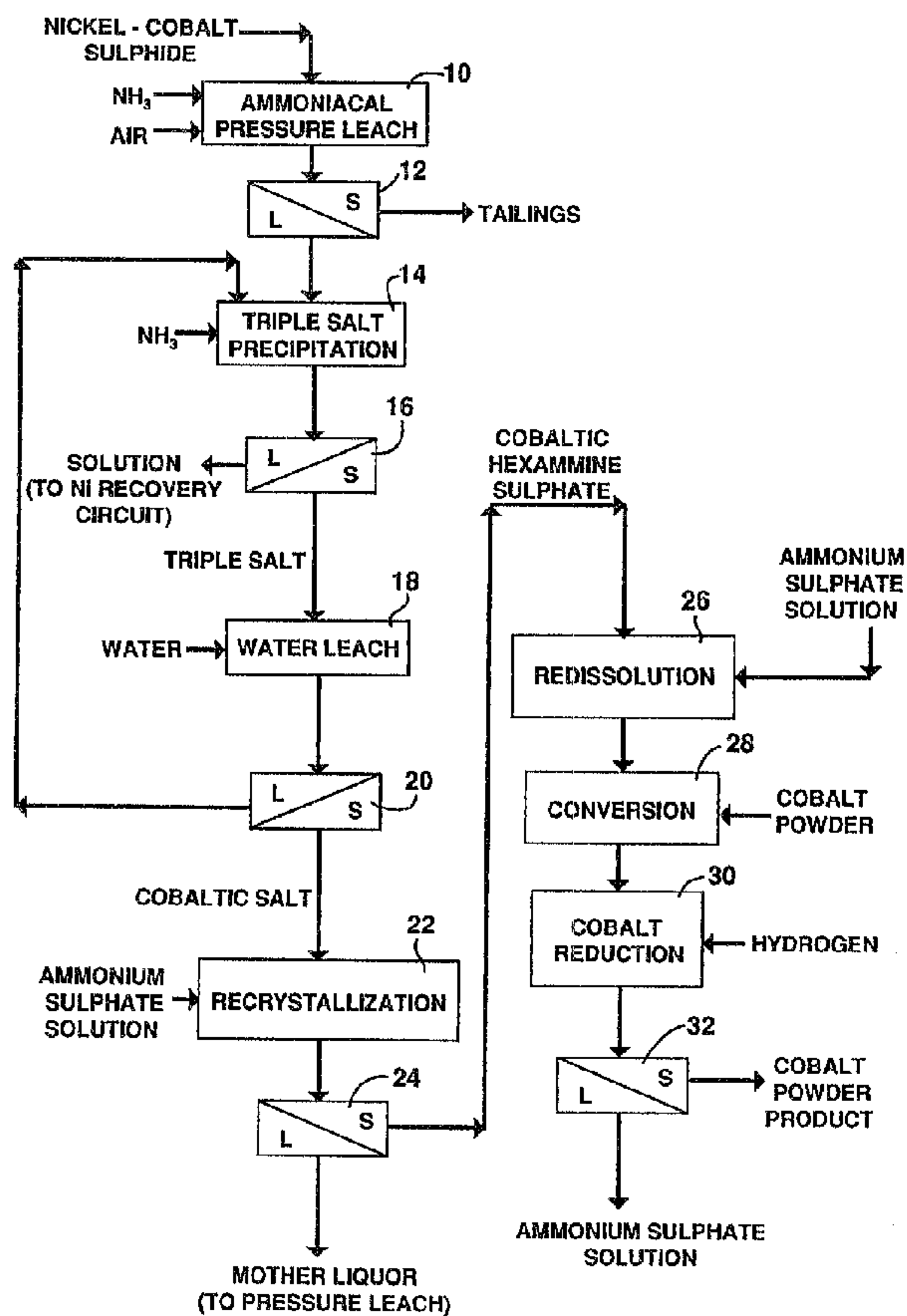
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(54) **PROCEDE DE SEPARATION DU COBALT ET DU NICKEL**

(54) **PROCESS FOR THE SEPARATION OF COBALT FROM NICKEL**



(57) A process is disclosed for separating cobalt in the form of cobalt (III) hexammine sulphate from an aqueous solution containing cobalt (III) hexammine sulphate and nickel (II) hexammine sulphate comprising adding ammonium sulphate to provide an effective amount of ammonium sulphate, ammoniating the solution with an effective amount of ammonia at a temperature whereby the triple salt of cobalt (III) hexammine sulphate, nickel (II) hexammine sulphate and ammonium sulphate is precipitated, recovering the precipitated triple salt from the solution, and repulping the triple salt with an effective amount of water or aqueous ammonia solution to selectively leach nickel (II) hexammine sulphate and ammonium sulphate to produce a crystalline cobalt (III) hexammine sulphate with a Co:Ni ratio of at least 100:1. In a preferred embodiment, the aqueous solution is produced by leaching nickel-cobalt sulphides in an ammoniacal ammonium sulphate solution under an elevated pressure of an oxygen bearing gas such as air or oxygen and at an elevated temperature with an effective ammonia to metal mole ratio to oxidize nickel and cobalt sulphides to sulphates and to produce an ammoniacal leach liquor in which dissolved cobalt is predominantly in the (III) oxidation stage.



ABSTRACT OF THE DISCLOSURE

A process is disclosed for separating cobalt in the form of cobalt (III) hexammine sulphate from an aqueous solution containing cobalt (III) hexammine sulphate and nickel (II) hexammine sulphate comprising adding ammonium sulphate to provide an effective amount of ammonium sulphate, ammoniating the solution with an effective amount of ammonia at a temperature whereby the triple salt of cobalt (III) hexammine sulphate, nickel (II) hexammine sulphate and ammonium sulphate is precipitated, recovering the precipitated triple salt from the solution, and repulping the triple salt with an effective amount of water or aqueous ammonia solution to selectively leach nickel (II) hexammine sulphate and ammonium sulphate to produce a crystalline cobalt (III) hexammine sulphate with a Co:Ni ratio of at least 100:1. In a preferred embodiment, the aqueous solution is produced by leaching nickel-cobalt sulphides in an ammoniacal ammonium sulphate solution under an elevated pressure of an oxygen bearing gas such as air or oxygen and at an elevated temperature with an effective ammonia to metal mole ratio to oxidize nickel and cobalt sulphides to sulphates and to produce an ammoniacal leach liquor in which dissolved cobalt is predominantly in the (III) oxidation stage.

Field of the Invention

This invention relates to a process for the recovery of cobalt from an aqueous solution containing nickel and cobalt and, more particularly, relates to the separation of cobalt from nickel dissolved in an ammoniacal leach liquor to produce a substantially nickel-free cobalt solution from which high purity cobalt metal and salt products can be obtained.

Background of the Invention

A refinery employing a hydrometallurgical process for the treatment of nickel-cobalt-copper sulphide concentrates and mattes to produce high grade nickel and cobalt powder products has been operated for many years by Sherritt Gordon Limited at Fort Saskatchewan, Alberta, Canada. In this process, which is described in detail in the publication "The Winning of Nickel", J. R. Boldt, Jr. (Ed), Toronto, Longmans Canada Limited, 1967, the nickel sulphide feed materials are leached in ammoniacal ammonium sulphate solution, under elevated air pressure, in horizontal autoclaves. The metals: nickel, cobalt and copper, are dissolved as metal ammine complex ions. Iron is oxidized to insoluble hematite, and sulphide sulphur is partially oxidized to form a range of unsaturated sulphur anions, including thiosulphate, trithionate, and sulphamate, which remain in the leach solution. The hematite residue is filtered off and discarded, and the leach solution is treated to totally remove copper, to partially remove the free ammonia, and to oxidize the sulphur anions to sulphate. The purified solution which contains about 60 g/L Ni and 1 g/L Co, with ammonia at an ammonia:metal mole ratio of about 2:1, together with 400 g/L ammonium sulphate is then treated with hydrogen under elevated pressure and temperature to precipitate nickel selectively as the metal powder. The cobalt remains in the reduced solution which contains about 1 g/L Ni and 1 g/L

Co. The residual metals are then precipitated using hydrogen sulphide to produce a mixed nickel cobalt sulphide which forms the feed to the cobalt refining process.

The recovery of cobalt in this nickel refining process is much lower than that of nickel, as a result of the adsorption of cobalt by the iron oxide leach residue. Furthermore, the selectivity of the reduction process, for nickel, depends on the maintenance of a low level of cobalt in the purified leach solution. It is only possible to produce nickel powder meeting the market specification for cobalt, if the Ni:Co ratio is maintained above about 25:1. It will be appreciated therefore that this nickel refining process is not economically effective for the treatment of nickel feed materials with high cobalt content, since significant losses of cobalt to the leach residue and to the nickel powder product will be incurred.

A cobalt refining process currently operated by Sherritt Gordon Limited is based on a soluble cobaltic pentammine process for separating nickel from cobalt, which is described in U.S. Patents Nos. 2,694,005, 2,694,006, 2,767,054 and 2,767,055 to Schaufelburger in which he proposed using the formation of the soluble cobaltic pentammine sulphate, $[\text{Co}(\text{NH}_3)_5\text{OH}]\text{SO}_4$, to permit the selective precipitation of nickel as nickel (II) ammonium sulphate, leaving cobalt (III) in solution, when the solution was acidified. In this process, mixed nickel-cobalt sulphides with a typical Ni:Co ratio of 1:1 are first leached under air pressure in dilute sulphuric acid solution to dissolve the nickel and cobalt and oxidize the sulphide to sulphate. The acidic leach solution, containing 80 to 100 g/L Ni + Co, is purified to remove iron and trace metals such as Cu, Cd and Zn, and ammonia is then added to neutralize the free acid and to adjust the ammonia to metals mole ratio to about 5:1 to form the

pentammine ions of divalent nickel and cobalt. The ammonium sulphate concentration is also adjusted. The solution is then treated with air under pressure in an autoclave, to oxidize the cobalt (II) pentammine ion to the cobalt (III) pentammine ion. The nickel (II) pentammine ion is not oxidized under these conditions. The oxidized solution is then treated in a two-stage process with concentrated sulphuric acid to adjust the solution pH to about 2.5. As a result of the acidification, nickel is selectively precipitated as the nickel ammonium sulphate double salt ($\text{NiSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4$), leaving cobalt (III) pentammine sulphate in solution. After the second stage of nickel removal, the cobaltic solution is essentially nickel-free, with a Co:Ni ratio greater than 1000:1. The cobalt (III) solution is then treated with metallic cobalt powder to reduce the cobalt (III) ion to cobalt (II), and acid is added to adjust the NH_3 :Co mole ratio to about 2:1. This solution is treated with hydrogen at elevated temperature and pressure to produce cobalt metal powder containing less than 0.1% Ni. This two-stage process, as described in U.S. Patent No. 2,822,264, is in operation to produce cobalt powder with a Co:Ni ratio of greater than 1000:1.

This separation process is designed to treat mixed nickel-cobalt sulphides with Ni:Co ratios of about 1:1. As the nickel content of the feed sulphide increases the process becomes less viable technically and economically, since the amount of nickel double salt to be precipitated and recycled to the nickel circuit increases, while the cobalt concentration in the purified solution decreases. In practice, a Ni:Co ratio of about 3:1 is the maximum that can be handled in this process. It will be appreciated therefore that neither of the above-described existing processes is suitable for the treatment of nickel-cobalt sulphides with Ni:Co weight ratios in the range 3:1 to

25:1.

When limonitic nickel laterite ores are treated by high temperature sulphuric acid pressure leaching, the leached nickel and cobalt are both recovered in high yields as a mixed sulphide typically containing 55% Ni and 5% Co, i.e. with a Ni:Co ratio of about 10:1. This process is expected to become increasingly important economically in the future as the nickel mining industry switches to the treatment of laterite ores, as economically viable sulphide ore reserves are depleted. Material of this composition cannot be treated economically by either of the above existing hydrometallurgical refining processes.

Methods of refining mixed nickel-cobalt sulphides of this type have been proposed in recent years, although none is known to have been commercialized successfully. A process described in Chemical Engineering, September 7, 1959, page 145, included an acidic pressure leach to oxidize nickel and cobalt sulphides to the corresponding sulphates, solution purification to remove iron, aluminum, copper, lead and zinc, and selective reduction of nickel with hydrogen. However, since the cobalt content of the solution fed to the nickel reduction operation was about 5 g/L, excessive contamination of the nickel powder with cobalt was unavoidable, and the nickel powder would not meet current market cobalt specifications.

This problem was addressed in U.S. Patent No. 3,751,558, which proposed a method of selectively precipitating cobalt (II) from the acid leach liquor produced by the pressure leaching of nickel-cobalt sulphides. In this process, a predetermined amount of ammonia was added to the acidic solution at low temperature (below 50°C) to precipitate a basic cobalt (II) sulphate. The basic cobalt sulphate was contaminated with nickel and typically had a Co:Ni ratio in the range 1:1 to

5:1. The degree of cobalt removal increased as the concentration of ammonium sulphate decreased, and the addition of calcium hydroxide was proposed to precipitate sulphate as gypsum. The product solution typically had a Ni:Co ratio of over 1000:1. No methods of treating the basic sulphate precipitate were proposed in this patent. Canadian Patent No. 1,147,970 describes a similar approach. Precipitation of the basic cobalt (II) sulphate using ammonia was originally described in German Patent No. 595,688 in 1934.

A number of methods proposed for the separation of cobalt from nickel have been based on the differential solubilities of the ammine complexes of nickel (II) and cobalt (III).

In U.S. Patent No. 2,728,636, Van Hare proposed a process in which cobaltic hexammine sulphate was precipitated directly from solution containing equal amounts of nickel and cobalt. The hexammine salt was formed by saturating a solution containing nickel (II) and cobalt (II) sulphates with ammonia, adjusting the sulphate concentration by adding ammonium sulphate to ensure a Co:SO₄ mole ratio of 2:3, and heating the solution under elevated oxygen pressure to over 100°C to oxidize Co(II) to Co(III). Under these conditions the cobalt (III) hexammine sulphate was precipitated selectively. The precipitated salts had Co:Ni ratios of 140:1 to 380:1.

In U.S. Patent No. 3,227,513, Alexa and Reichert proposed the use of an insoluble cobalt(III)pentammine sulphite complex, $(2\text{Co}(\text{NH}_3)_5\text{SO}_3](\text{SO}_4)_2$, which is formed in the oxidative ammoniacal leaching of mixed nickel-cobalt sulphides at 70°C at atmospheric pressure. This complex salt was separated from the leach solution, and roasted to produce cobalt (II) sulphate with a Co:Ni ratio of about 100:1.

In U.S. Patent No. 3,967,957, Fonseca proposed utilizing the formation of insoluble copper tetrammine sulphate, $\text{Cu}(\text{NH}_3)_4 \text{SO}_4$, for the recovery of copper from ammoniacal leach liquor. Nickel and cobalt were recovered together with the copper in the precipitated salt when the leach liquor was saturated with ammonia. No method of separating the nickel and cobalt from the copper salt was proposed.

In U.S. Patent No. 4,208,380, Hamalainen et al proposed a modification to the soluble cobaltic pentammine process in which a catalyst was used to treat the oxidized solution containing cobalt (III) pentammine, and nickel (II) pentammine sulphates, to convert the Co(III) complex ion to the hexammine ion, $[\text{Co}(\text{NH}_3)_6]^{3+}$. Cobaltic hexammine sulphate crystallized out when the solution was cooled below 50°C . The precipitated salt typically had a Co:Ni ratio of over 1000:1.

Separation techniques in which solvent extraction is used to extract cobalt from nickel containing solutions, which have been applied successfully in both chloride and sulphate refining processes where the metals are recovered by electrowinning, are not compatible with metal recovery by hydrogen reduction, and cannot be used for the treatment of nickel-cobalt sulphides with Ni:Co weight ratios above 3:1.

The existing refining processes utilize solutions which typically contain high levels of free ammonia and ammonium sulphate. For this reason, it is desirable that the nickel-cobalt sulphide should be leached directly in an ammoniacal pressure leach system, in preference to the acid pressure leaching process which has been widely used in existing operations, to produce an ammoniacal leach solution containing nickel and cobalt values. It is a principal object of the present invention to provide an

ammoniacal pressure leach process in which an ammoniacal leach solution containing nickel and cobalt values can be treated to selectively remove cobalt to reduce the cobalt content of the solution feed to the nickel reduction step, at least to the level at which it would not contaminate the nickel powder product, i.e. the solution Ni:Co ratio should be greater than about 25:1. It is another object of the invention that the cobalt precipitate from a cobalt separation process should be amenable to upgrading to a nickel-free cobalt product at minimum cost, and preferably in existing circuits. Another object of the present invention is the provision of a process for the treatment of nickel-cobalt sulphides with Ni:Co weight ratios above 3:1.

Summary of the Invention

Applicant has found surprisingly that these objects can be achieved by a relatively simple process. In its broad aspect, the process of the present invention relates to the separation of cobalt in the form of cobalt (III) hexammine sulphate from an aqueous solution containing cobalt (III) hexammine sulphate and nickel (II) hexammine sulphate with a Ni:Co ratio greater than 1:1 comprising adding ammonium sulphate to provide an effective amount of ammonium sulphate, ammoniating the solution with an effective amount of ammonia at a temperature whereby the triple salt of cobalt (III) hexammine sulphate, nickel (II) hexammine sulphate and ammonium sulphate is precipitated, usually at a temperature below 50°C and preferably at a temperature in the range of 20 to 40°C, recovering the precipitated triple salt from the solution, and repulping the triple salt with an effective amount of water or aqueous ammonia solution to selectively leach nickel (II) hexammine sulphate and ammonium sulphate to produce a crystalline cobalt (III) hexammine sulphate, preferably

with a Co:Ni ratio of at least 100:1, and a nickel enriched solution.

More particularly, the process of the invention relates to the production of a cobalt (III) hexammine sulphate which comprises leaching nickel-cobalt sulphides in an ammoniacal ammonium sulphate solution under an elevated pressure of an oxygen-bearing gas such as air or oxygen, at a temperature of preferably at least 80°C with an effective ammonia to metal mole ratio preferably in the range of 5:1 to 6.5:1 to oxidize nickel and cobalt sulphides to sulphates, and to produce an ammoniacal leach liquor in which dissolved cobalt is predominantly in the (III) oxidation state and a leach residue. The leach liquor which may be separated from the leach residue is ammoniated by saturating the leach liquor with an effective amount of ammonia and the ammoniated leach liquor cooled to below 50°C whereby the triple salt of cobalt (III) hexammine sulphate, nickel (II) hexammine sulphate and ammonium sulphate is precipitated, the precipitated triple salt recovered from the leach liquor, and the triple salt repulped with an effective amount of water or aqueous ammonium hydroxide solution to selectively leach nickel (II) hexammine sulphate and ammonium sulphate and to produce a crystalline cobaltic (III) hexammine sulphate with a Co:Ni ratio of preferably at least about 100:1 and a nickel-enriched leach liquor.

In a preferred aspect of the invention, the process additionally comprises recovering the cobaltic (III) hexammine sulphate, dissolving the recovered cobaltic (III) hexammine sulphate in hot ammonium sulphate solution, and cooling and ammoniating the solution to precipitate recrystallized cobaltic (III) hexammine sulphate having a Co:Ni ratio of at least about 1000:1.

In another preferred aspect, the process of the

invention additionally comprises recovering the recrystallized cobaltic (III) hexammine sulphate from the solution, redissolving the recrystallized cobalt (III) hexammine sulphate in hot ammonium sulphate solution, treating the said hot ammonium sulphate solution containing redissolved cobaltic (III) hexammine sulphate with an effective amount of cobalt powder to reduce Co(III) to Co(II) and with an effective amount of acid to adjust the ammonia:cobalt mole ratio to about 2:1 to produce cobalt (III) diammine sulphate in solution, and treating the cobalt (III) diammine sulphate solution to produce cobalt powder therefrom.

The aqueous solution or the ammoniacal leach liquor containing cobalt (III) hexammine sulphate and nickel (II) hexammine sulphate is ammoniated by saturation with an effective amount of ammonia by adding anhydrous ammonia or a solution of ammonium hydroxide to provide about 170 to about 190 g/L terminal free ammonia. The ammoniacal leach liquor preferably contains about 150 to 250 g/L ammonium sulphate and the saturated leach liquor preferably is cooled to a temperature in the range of 20 to 40° C. It has been found that repulping the precipitated triple salt with an effective amount of water at a rate to maintain titratable ammonia at about 110 to 130 g/L selectively leaches nickel (II) hexammine sulphate and ammonium sulphate, preferably at a water temperature in the range of about 20 to 40° C.

Brief Description of the Drawing

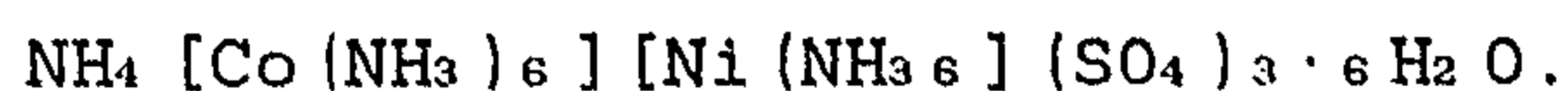
The process of the invention will now be described with reference to the drawing in which Figure 1 is a schematic flowsheet of a preferred embodiment of the process.

Description of the Preferred Embodiments of the Invention

Mixed nickel-cobalt sulphides in a finely divided

state are first pressure leached in ammoniacal pressure leach 10, separately from other refinery nickel feeds, in an ammoniacal ammonium sulphate solution under elevated air pressure of at least 500kPa, at a temperature of at least 90° C, to oxidize the nickel and cobalt sulphides to the corresponding sulphates. The ammonia:metal mole ratio is controlled in the range of about 5:1 to 6.5:1, preferably at about 6:1, to maximize the formation of hexamine complex ions of nickel (II) and cobalt (III). It will be understood that the dissolved cobalt in the ammoniacal leach liquor will be predominantly in the (III) oxidation state, in contrast to acidic leach liquors where the cobalt is predominantly in the (II) oxidation state.

The successful operation of the process of this invention depends on the formation of a triple salt containing cobalt (III) hexamine sulphate, nickel (II) hexamine sulphate and ammonium sulphate. On the basis of chemical analysis and solution ultra violet-visible spectroscopic data, it is believed that the composition of this salt may be represented as:



The theoretical cobalt and nickel contents of this salt are 8.00% and 7.97% respectively. Samples of the salt usually analyze 7.5 to 9.0% Co and 8.0 to 9.3% Ni. It should be noted that since the salt is totally or partially soluble in water or aqueous ammonia solution, at ambient temperatures, it is difficult to isolate the pure salt free from entrained metal and ammonia-bearing solutions. We have found no prior description of this compound in either the chemical or patent literature. Double salts formed between nickel (II) hexamine sulphate and ammonium sulphate and between cobalt (III) hexamine sulphate and ammonium sulphate, are described by Mellor J. W. in "A Comprehensive Treatise on Inorganic and Theoretical Chemistry" (Longmans Greene Co Ltd., 1946) Vol XV p. 468

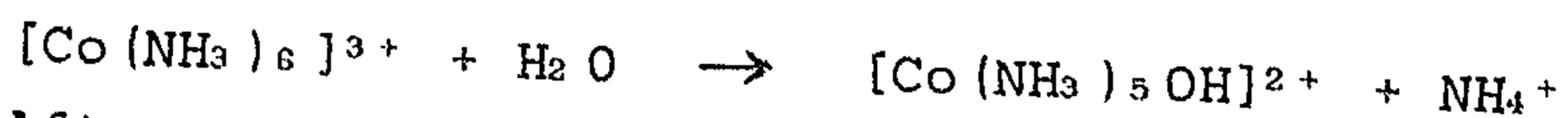
and Vol XIV p. 791 respectively. Both these salts form needle shaped crystals similar to those formed by the triple salt, which are quite different from the granular crystals formed by cobaltic hexammine sulphate and nickel hexammine sulphate. The triple salt is a pale tan in colour, while the cobaltic hexammine salts are a deep orange, and the nickel hexammine salts are a deep purple.

Another important aspect of the process of the invention is the discovery that this cobalt enriched triple salt can be precipitated very simply by sparging anhydrous ammonia into ammoniacal ammonium sulphate leach liquor containing cobaltic and nickel hexammine sulphate, at a low temperature of less than 50°C, preferably at a temperature in the range of 20 to 40°C. U.S. Patent No. 2,728,636 in distinction indicates that a high temperature autoclave oxidation of above 100°C was required after the solution was saturated with ammonia in order to precipitate cobaltic hexammine sulphate. It will be understood that for the precipitation to be effective, the bulk of the cobalt and nickel in the solution must be present as the cobaltic and nickel hexammine ions respectively.

It has also been found that a high concentration of ammonium sulphate, i.e. above about 150 g/L, is necessary for the most effective precipitation of the triple salt. However, excessively high free ammonia or ammonium sulphate levels in the leach liquor can lead to premature precipitation of the triple salt when the autoclave discharge slurry is cooled before the liquid solid separation step to remove the leach residue. In such a situation, significant losses of cobalt to the leach residue may result. Generally, therefore, the ammonia level in the leach liquor should not exceed about 150 g/L NH_3 , and the ammonium sulphate level should not exceed about 250 g/L $(\text{NH}_4)_2\text{SO}_4$.

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The first essential step in the process of the invention is therefore to operate the ammoniacal pressure leach 10 of mixed nickel-cobalt sulphide under conditions which promote the maximum conversion of dissolved cobalt to the cobalt (III) hexammine ion. It is particularly important to minimize the concentration of cobaltic pentammine sulphate, which does not precipitate in the triple salt precipitation, and which is formed by the aquation or hydrolysis of the cobaltic hexammine ion, by reaction with water:



After a liquid-solid separation 12 to remove the leach residue containing insoluble iron oxide and gangue from the ammoniacal pressure leach 10, the leach liquor which usually contains about 65 g/L Ni and 6 to 12 g/L Co is saturated with ammonia and cooled to below 50°C in triple salt precipitation 14. Under these conditions a crystalline triple salt, containing cobalt (III) hexammine sulphate, nickel (II) hexammine sulphate, and ammonium sulphate, with a Co:Ni ratio of about 1:1, is precipitated, typically leaving about 2g/L Co in solution. The residual solution which typically has a Ni:Co ratio of about 25:1, is suitable for processing in existing nickel circuits to produce nickel powder meeting market specifications for cobalt content. Normally the residual liquor from the triple salt precipitation step 14 is blended with leach liquor from circuits treating low cobalt content nickel feeds, so that the Ni:Co ratio in the nickel reduction feed solution will be much greater than 25:1. The residual cobalt in the nickel solution passes through a nickel reduction step and is precipitated with hydrogen sulphide as a nickel-cobalt sulphide with a Ni:Co ratio of 1:1 in a sulphide precipitation step, not shown. This sulphide is recycled as feed to the ammoniacal pressure leach 10.

When the cobaltic hexammine sulphate and ammonium sulphate are in the specified ranges, the triple salt can be precipitated by increasing the free ammonia content of the solution to the range 170 to 190 g/L NH_3 , and cooling the solution to 50°C or lower temperature. Ammonia may be added either by sparging anhydrous ammonia into the solution, or by adding a concentrated solution of ammonium hydroxide. The precipitation of cobalt from the solution does not go to completion, since cobaltic hexammine sulphate remains slightly soluble in the leach liquor even at the high ammonia concentration, and usually some of the cobaltic ion is present in the form of pentammine complex species, which do not precipitate with the triple salt. Lower temperatures than about 20°C do not significantly increase the efficiency of cobalt precipitation, the preferred temperature range being 20 to 40°C. Precipitation of the triple salt occurs rapidly, within seconds of the ammonia addition. Neither increased retention time beyond about 15 minutes, nor additional staging, results in a significant increase in cobalt precipitation.

After precipitation of the triple salt, the nickel hexammine solution will typically contain about 2 g/L of residual cobalt. The amount of cobalt precipitated will therefore depend on the concentration of cobalt in the leach liquor. Generally, for solutions containing 6 g/L to 12 g/L Co, the amount of cobalt removed will range from 50 to 80%. If excessive ammonia is added to the leach liquor, or if the ammonium sulphate concentration is too high, i.e. greater than about 300 g/L, nickel hexammine sulphate coprecipitates with the triple salt, resulting in a precipitate with a low Co:Ni ratio. It appears that the solubility of the triple salt depends largely on the concentration of unbound ammonia in the solution. (Unbound ammonia is to be understood to refer to dissolved free

ammonia which is not complexed with the metal ions, i.e. the free ammonia above that required to form the hexamine ions). As a result, the optimum precipitation of cobalt, in percentage terms, occurs at about 170 to 190 g/L NH_3 when the nickel content of the solution is less than about 75 g/L Ni. While more efficient cobalt removal can be achieved from more dilute nickel solutions, it will be appreciated that dilution of the nickel concentration, below about 50 g/L Ni, is economically undesirable, since it will result in an excessively dilute solution feed to the nickel reduction circuit, thus reducing the productivity of the refinery.

The preferred solution composition and process conditions for the triple salt precipitation step are thus defined as follows:

Feed Solution Composition

Nickel Concentration	50 to 75 g/L
Cobalt Concentration	5 to 15 g/L
Ni:Co Mole Ratio	4:1 to 15:1
Ammonia:Metals Mole Ratio	5:1 to 6.5:1
Maximum Free Ammonia Concentration	150 g/L
Ammonium Sulphate Concentration	150 to 250 g/L

Process Conditions

Temperature	20 to 40°C
Terminal Free Ammonia	170 to 190 g/L
Retention Time	15 minutes

The triple salt which is recovered by filtration 16 from the cooled liquor, is repulped with a controlled amount of water, and is leached at about 40°C in water leach 18 to selectively leach nickel hexamine sulphate and ammonium sulphate, as well as other trace metals such as copper, zinc and cadmium, to produce a crystalline cobaltic hexamine sulphate with a Co:Ni ratio of 100:1 or higher. The leach liquor which typically contains 65 g/L Ni and 10 to 15 g/L Co

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with 120 g/L NH_3 is recycled to the triple salt precipitation step, where most of the cobalt is reprecipitated, and the soluble nickel is returned to the nickel circuit.

Another important aspect of the process of the invention thus is the surprising discovery that the triple salt can be leached in water at ambient temperature to extract over 99% of its nickel hexammine sulphate and ammonium sulphate contents, with only about 20% of its cobaltic hexammine sulphate content, to yield a crystalline cobaltic hexammine sulphate with a Co:Ni ratio of over 100:1. The leach liquor, which typically contains about 65 to 70 g/L Ni, and 10 to 15 g/L Co, with 110 to 130 g/L NH_3 and 60 to 80 g/L ammonium sulphate, is very suitable for recycle to the triple salt precipitation step, where most of the cobalt is reprecipitated as the triple salt, and most of the nickel and ammonium sulphate remain in the nickel solution. Consequently, a process consisting of the triple salt precipitation step, followed by water leaching of the triple salt, with recycle of the leach liquor to the precipitation step, provides an effective and economical method of separating cobalt from the ammoniacal nickel-cobalt leach liquors. The relatively pure cobalt salt produced by this process can be further upgraded by recrystallization to a cobaltic hexammine sulphate with a Ni:Co ratio of over 1000:1, to be described, which is particularly suitable as a feed to prior art processes for producing high grade cobalt powder and cobalt salts.

The key to successful operation of the water leach is to control the rate of water addition. If too much water is added quickly, all the cobalt hexammine sulphate is leached along with the nickel hexammine sulphate and the ammonium sulphate. If too little water is added slowly, the extraction of nickel hexammine sulphate is incomplete, and too much nickel remains in the cobaltic hexammine salt. It has been found that the most effective leaching is achieved if the rate of water

addition is controlled so that the leach liquor contains 110 to 130 g/L of titratable ammonia.

In this system, titratable ammonia is to be understood as dissolved ammonia which can be titrated with sulphuric acid to an end point at pH 4. The cobaltic hexammine ion is much more stable in aqueous solutions than the nickel hexammine ion. As a result, ammonia complexed with nickel can be titrated with acid, while ammonia complexed with the cobaltic ion cannot. In solution therefore ammonia associated with nickel reports as titratable ammonia, while the six ammonia molecules complexed with each cobaltic ion do not. Any additional free ammonia present in the leach solution, over that required to form the nickel and cobaltic hexammine ion, will also report as titratable ammonia.

The principal source of titratable ammonia in the solution produced by leaching the triple salt in water, is therefore the nickel hexammine sulphate which is leached. Each nickel hexammine ion, $[\text{Ni}(\text{NH}_3)_6]^{2+}$, dissolves six ammonia molecules which go into solution. Thus, if 65 g/L of nickel dissolves, 112 g/L of titratable ammonia will go into solution. Other titratable ammonia may also come from other metal ammine ions, e.g. Cu and Zn, which also leach with the nickel, and from entrained liquor in the triple salt, which contains 170 to 190 g/L NH_3 .

In operating the water leach of the triple salt, the objective is to maintain a sufficiently high titratable ammonia concentration in the solution to minimize the solubility of cobaltic hexammine sulphate while keeping the titratable ammonia concentration low enough to permit the leaching of nickel hexammine sulphate. It has been found that titratable ammonia concentrations above about 130 g/L NH_3 inhibit the leaching of nickel hexamine sulphate at 20 to 40°C. Consequently, the water additional rate is adjusted to produce a leach solution containing 110 to 130 g/L titratable ammonia

when $\geq 99\%$ of the nickel has been extracted. The process is controlled in practice by maintaining the specified levels of nickel at about 65 to 75 g/L and titratable ammonia at about 110 to 130 g/L. Fine control of the titratable ammonia level may also be achieved if required by the addition of anhydrous ammonia to the leach tanks.

The water leaching of the triple salt should be carried out at as low a temperature as practical, and preferably in the temperature range about 20 to 40°C, to minimize both the dissolution of cobalt hexamine sulphate and the loss of ammonia from the solution by evaporation. In a continuous leaching system, a retention time of 15 to 30 minutes and at least two leaching stages, to minimize short circuiting of solids, are sufficient to extract over 99% of the nickel content of the triple salt.

The final stage in the separation of cobalt from nickel thus is the recrystallization of the cobaltic hexamine sulphate produced in the water leaching step 18. The solubility properties of cobaltic hexamine sulphate in water and in aqueous solutions of ammonia and ammonium sulphate have been described in detail by I. Y Lesch et al, in Zh. Prikl. Khim., 43 (1970) p 1665. The process of dissolving the cobaltic salt in hot water or aqueous ammonium sulphate solution, and reprecipitating the cobaltic hexamine sulphate by cooling the solution and adding ammonia, to minimize the solubility of the cobaltic hexamine ion, upgrades the cobaltic salt from a Co:Ni ratio of over 100:1 to a Co:Ni ratio of over 1000:1. Nickel remains in the recrystallization mother liquor which is recycled to the nickel circuit.

In addition to separating nickel from cobalt, the water leach also separates other ammonia-soluble base metals, such as copper, zinc and cadmium from cobalt. In both the triple salt precipitation step and in the water leach, these metals follow nickel, rather than cobalt. As a result, the recrystallized

cobaltic hexammine sulphate salt contains very low levels of copper and cadmium. In the prior art processes, such as the soluble cobaltic pentammine process, these metals follow cobalt in the cobalt-nickel separation step, and must be removed from the solution in an additional sulphide precipitation circuit.

Since the leach liquor contains over 60 g/L of nickel, it is essential for efficient removal of nickel from the cobaltic hexammine salt that the solids should be well washed with a nickel-free solution to displace the entrained solution, during the liquid-solid separation step. Since the cobaltic salt is appreciably soluble in cold water and aqueous ammonium sulphate solution, the preferred wash medium is an aqueous ammonium hydroxide solution with an ammonia content of 130 to 150 g/L NH_3 , in which the cobaltic salt is only slightly soluble.

The cobaltic hexammine sulphate is recovered by liquid-solid separation 20 and is further upgraded by recrystallization in step 22 wherein the salt is dissolved in hot ammonium sulphate solution and is reprecipitated by cooling and ammoniation to yield a cobaltic hexammine sulphate product with a Co-Ni ratio of 1000:1 or higher. The mother liquor is recycled to ammoniacal pressure leach 10 and the purified cobalt salt can potentially be used as the feed to processes for the production of cobalt metal or cobalt chemicals, using known prior art procedures. For the production of cobalt powder by hydrogen reduction, for example, the purified salt is recovered by liquid-solid separation 24 and redissolved in hot ammonium sulphate solution in redissolution step 26, and is then treated with cobalt powder to reduce Co(III) to Co(II), and with acid to adjust the ammonia:cobalt mole ratio to about 2:1 in conversion step 28. This solution then forms the feed to the cobalt reduction step 30 in which cobalt powder is produced using hydrogen at elevated temperature and pressure and separated from the ammonium sulphate solution by liquid

solid separation 32. Alternatively, the purified cobaltic hexammine sulphate can be pyrolized to cobalt (II) sulphate, or cobalt oxides, as described in U.S. Patent No. 4,208,380.

The process of this invention represents a major simplification in the process of separating and refining of cobalt, from nickel-cobalt sulphides, with a corresponding benefit in process economics. Since the by-product nickel-cobalt sulphides, recovered from the nickel reduction end solution, can be recycled to the ammonia pressure leach, a large part of the existing cobalt refinery process can be by-passed. Thus, the acid pressure leach, iron removal, zinc-cadmium removal, oxidation and the two stages of nickel removal can be eliminated and replaced by the water leach, recrystallization and dissolution steps.

The process of the invention has a number of further economic and technical advantages over the prior art technology. The only chemical reagents used are ammonia and ammonium sulphate, neither of which is consumed in the process, and both can be recovered for reuse in very high yield. Since there is a substantial reduction in the use of sulphuric acid in the leaching and cobalt separation steps compared with the existing process, the production of by-product ammonium sulphate is reduced. The separation steps are carried out at low temperatures with consequent reduction in energy (steam) consumption. The recovery of cobalt as a purified solid of constant composition, instead of a purified aqueous solution of variable concentration, provides a greater degree of control in adjusting the composition of solution feeds to the subsequent metal recovery steps, thus permitting increased utilization of existing equipment. Finally, all the precipitated solids are easy to separate from the solutions, either by sedimentation, filtration or centrifuging.

The process of the invention is further illustrated

with reference to the following non-limitative examples.

EXAMPLE 1

This example illustrates the process of the invention for triple salt precipitation from ammoniacal ammonium sulphate leach liquid containing nickel and cobalt. All percentages are by weight unless otherwise indicated for this and subsequent examples. A sample of a nickel-cobalt sulphide, containing 53.2% Ni, 5.1% Co, 0.58% Fe and 35.7% S, was batch pressure leached in a solution containing 220 g/L ammonium sulphate and 160 g/L ammonia for 2h at 85°C with a 200 kPa oxygen overpressure. The discharge slurry was filtered without cooling to separate the leach liquor from the leach residue. Metal extractions were 98% Ni and 97% Co and the residue contained 27% Ni, 3.1% Co, 12.4% Fe and 19.3% S. The leach solution contained 73 g/L Ni, 7.0 g/L Co and 140 g/L titratable ammonia. The Ni:Co weight ratio was 10.4:1 and the NH₃:Ni mole ratio was 6.6:1.

2.0 L of the filtered leach liquor from the pressure leach was cooled to 40°C and sparged with anhydrous ammonia vapour to increase the titratable ammonia level to about 180 g/L NH₃. The resulting slurry was cooled to 25°C and filtered. The liquor contained 63 g/L Ni, 1.5 g/L Co and 177 g/L NH₃, while the crystalline precipitate (133g) contained 9.75% Ni, 8.0% Co and 14.7% S. The precipitate had a Ni:Co ratio of 1.2:1, while the solution had a Ni:Co ratio of 42:1. The precipitate contained 76% of the cobalt content of the leach liquor and only 9% of the nickel content.

EXAMPLE 2

This example indicates the effect of temperature on triple salt solubility. Three samples of an ammoniacal ammonium sulphate solution, produced by the pressure

leaching of the same nickel-cobalt sulphide as used in Example 1, containing 69 g/L Ni, 6.4 g/L Co, 118 g/L NH_3 and 220 g/L ammonium sulphate were placed in a continuous laboratory autoclave and sparged with anhydrous ammonia to 180 g/L NH_3 at 25 °C, and filtered at temperatures of 25, 40 and 85°C respectively. The compositions of the resulting precipitates and solutions are summarized in Table I

Table I

Filtration Temperature, °C		25	40	85
Weight of Precipitate, Solution, g/L	g/L	47.5	45.3	40.1
	Ni	53	54	60
	Co	1.6	1.6	2.6
Precipitate, %	NH_3	162	169	152
	Ni	10.4	10.1	10.1
	Co	7.8	7.7	7.8
Ratio	Ni:Co	1.3	1.3	1.3
Cobalt Precipitation, %		70	70	57

EXAMPLE 3

This example illustrates triple salt precipitation from solution with a 5:1 nickel:cobalt ratio. 420 litres of a solution containing 60 g/L Ni, 10.8 g/L Co, and 4.2 g/L Cu, from a commercial ammonia pressure leach circuit treating nickel-cobalt sulphides, was sparged with anhydrous ammonia in a single stage continuous circuit with a retention time of 30 minutes at 40°C, to raise the titratable ammonia level to 170 g/L NH₃. The resulting slurry was filtered to yield 475 litres of solution containing 46 g/L Ni, 3.9 g/L Co and 3.7 g/L Cu, and 47 kg of crystalline triple salt filter cake with a moisture content of about 25%. The filter cake analyzed 6.8% Ni and 5.7% Co. Cobalt recovery to the triple salt was approximately 60%, together with 13% of the nickel and 3% of the copper contents of the leach solution.

EXAMPLE 4

A water leach of triple salt is described in this example. A 145 g sample of triple salt containing 7.5% Ni and 6.2% of Co was leached in 200 mL tap water at 40°C for 30 minutes. The slurry was filtered to separate the solids from the leach solution, and the filter cake was washed with 100 mL of 150 g/L aqueous ammonia solution. The leach residue (30g) analyzed 18.4% Co, 0.11% Ni with a Co:Ni ratio of 170:1, and the combined leach and wash solutions analyzed 35 g/L Ni and 11.2 g/L Co with a Ni:Co ratio of 3.1:1. Approximately 60% of the cobalt content of the triple salt was recovered in the cobaltic hexamine sulphate leach residue.

EXAMPLE 5

A water leach of triple salt at increased pulp density is described in this example. Three 10 kg batches of wet triple salt filter cake, produced in Example 3, were each leached in 7.5 litres of tap water at 30°C for 30

minutes with vigorous agitation. The leach slurries were filtered on a batch vacuum filter and each filter cake was displacement washed with 2.5L of 150 g/L aqueous ammonia solution. The primary and wash filtrate solutions were combined for analysis. The leach residue and solution analyses are summarized in Table II.

Table 2

Batch	Leach Residue			Leach Solution		
	%		Ratio	g/L		Ratio
	Co	Ni	Co:Ni	Ni	Co	Ni:Co
1	17.4	0.140	124	64.3	15.0	4.3
2	17.7	0.126	141	65.8	15.7	4.2
3	17.3	0.188	92	58.9	14.3	4.1

Over 70% of the cobalt content of the triple salt was recovered in the cobaltic hexammine sulphate leach residues.

EXAMPLE 6

The effect of pulp density on water leach of triple salt is illustrated in this example. Table 3 summarizes data obtained in a series of leach tests in which 200g of wet triple salt filter cake (164g dry) was leached in increasing volumes of tapwater from 100 mL up to 400 mL at 25°C for 30 minutes. In each case the leach residue was displacement washed with 150 g/L aqueous ammonia solution to remove entrained nickel containing leach liquor.

Table 3

Volume of Water, mL		100	150	200	300	400
Volume of Leachate, mL		170	240	290	380	470
Leachate, g/L	NH ₃	130	95	87	67	52
	Ni	82	55	49	39	32
	Co	18	19	15	13	11
	Ratio, Ni:Co	4.5	2.9	3.2	3.0	2.9
Residue, %	Co	15.0	17.1	17.3	17.3	17.3
	Ni	0.05	0.07	0.13	0.08	0.10
	Ratio, Co:Ni	300	240	130	220	170
Co Recovery to Residue, %		72	63	65	63	58

EXAMPLE 7

This example illustrates the recrystallization of cobaltic hexammine sulphate. A 5.8 blended batch of the cobaltic hexammine sulphate produced in Example 5, with a Co:Ni ratio of 116.1, was dissolved in 10 litres of an aqueous solution containing 200 g/L ammonium sulphate solution at 80°C. The solution was cooled to 40°C and anhydrous ammonia vapour was sparged into the solution to increase the titratable ammonia level to about 100 g/L NH₃. The orange cobaltic hexammine sulphate crystals were filtered off and displacement washed with 150 g/L aqueous ammonia solution. The recrystallized salt analyzed 18.2% Co, and 0.0053% Ni, corresponding to a Co:Ni ratio of 3400:1. The salt also contained 750 g/t Zn, 20 g/t Fe and less than 10 g/t Cu and Cd. The combined mother liquor and wash liquors contained only 3.3 g/L Co with 0.05 g/L Ni, 0.018 g/L Zn.

EXAMPLE 8

The production of cobalt powder from cobaltic

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hexamine sulphate is described in this example. The recrystallized cobaltic hexamine salt was redissolved in ammonium sulphate solution, treated with cobalt powder to reduce the cobaltic salt to cobaltous, and treated with hydrogen at high temperature and pressure to produce cobalt powder, using prior art procedures. The powder product contained impurity levels of 700 g/t Ni, 300 g/t Fe, 40 g/t Cr, 8 g/t Cd and less than 5 g/t Cu and Zn

It will be understood that other embodiments and examples of the invention will be readily apparent to a person skilled in the art, the scope of the invention being defined in the appended claims.

We claim:

1. A process for separating cobalt in the form of cobalt (III) hexamine sulphate from an aqueous solution containing cobalt (III) hexamine sulphate and nickel (II) hexamine sulphate with a Ni:Co ratio greater than 1:1 comprising adding ammonium sulphate to provide an effective amount of ammonia sulphate, adding ammonia to the solution to provide an effective amount of ammonia at a temperature whereby the triple salt of cobalt (III) hexamine sulphate, nickel (II) hexamine sulphate and ammonium sulphate is precipitated, recovering the precipitated triple salt from the solution, and repulping the triple salt with an effective amount of water or aqueous ammonia solution to selectively leach nickel (II) hexamine sulphate and ammonium sulphate to produce a crystalline cobalt (III) hexamine sulphate with a Co:Ni ratio of at least 100:1 and a nickel enriched solution.
2. A process as claimed in claim 1 in which the ammonia is added to the aqueous solution at a temperature below about 50°C.
3. A process as claimed in claim 1 in which the ammonia is added to the aqueous solution at a temperature in the range of about 20 to 40°C.
4. A process for producing cobalt (III) hexamine sulphate, comprising leaching nickel-cobalt sulphides in an ammoniacal ammonium sulphate solution under an elevated pressure of an oxygen bearing gas and at an elevated temperature with an effective ammonia to metal mole ratio to oxidize nickel and cobalt sulphides to sulphates and to produce an ammoniacal leach liquor in which dissolved cobalt is predominantly in the (III) oxidation state and a leach residue, ammoniating the leach liquor by saturating the leach liquor with an effective amount of ammonia and cooling the ammoniated leach liquor whereby the triple salt of cobalt (III) hexamine sulphate, nickel (II) hexamine sulphate and ammonium sulphate is precipitated, recovering the precipitated triple salt from the leach liquor, and repulping the triple salt with an effective amount of water or aqueous ammonia solution to selectively leach nickel (II) hexamine sulphate and ammonium sulphate and to produce a crystalline cobaltic (III) hexamine sulphate and a nickel-enriched leach liquor.
5. A process as claimed in claim 4 in which the nickel-cobalt sulphides are leached under an elevated air pressure at an elevated temperature of at least 90°C with an effective ammonia to metal mole ratio in the range of 5:1 to 6.5:1.

6. A process as claimed in claim 5 in which the ammoniated leach liquor is cooled to a temperature below 50°C whereby the triple salt of cobalt (III) hexamine sulphate, nickel (II) hexamine sulphate and ammonium sulphate is precipitated.
7. A process as claimed in claim 5 in which the ammoniated leach liquor is cooled to a temperature in the range of 20 to 40°C whereby the triple salt of cobalt (III) hexamine sulphate, nickel (II) hexamine sulphate and ammonium sulphate is precipitated, and repulping the triple salt with an effective amount of water to selectively leach nickel (II) hexamine sulphate and ammonium sulphate and to produce a crystalline cobaltic (III) hexamine sulphate with a Co:Ni ratio of at least 100:1.
8. A process as claimed in claim 7 which additionally comprises recovering the cobaltic (III) hexamine sulphate, dissolving the recovered cobaltic (III) hexamine sulphate in hot ammonium sulphate solution, and cooling and ammoniating the solution to precipitate recrystallized cobaltic (III) hexamine sulphate having a Co:Ni ratio of at least 1000:1.
9. A process as claimed in claim 8 which additionally comprises recovering the recrystallized cobaltic (III) hexamine sulphate from the solution, redissolving the recrystallized cobalt (III) hexamine sulphate in hot ammonium sulphate solution, treating the said hot ammonium sulphate solution containing redissolved cobaltic (III) hexamine sulphate with an effective amount of cobalt powder to reduce Co (III) to Co (II) and with an effective amount of acid to adjust the ammonia:cobalt mole ratio to about 2:1 to produce cobalt (II) diammine sulphate in solution, and treating the cobalt (II) diammine sulphate solution to produce cobalt powder therefrom.
10. A process for producing cobalt powder from nickel cobalt sulphides comprising leaching said nickel cobalt sulphides in an ammoniacal ammonium sulphate solution under an elevated pressure of an oxygen bearing gas at a temperature of at least 80°C with an effective ammonia to metal mole ratio in the range of 5:1 to 6.5:1 to oxidize the nickel and cobalt sulphides to sulphates and to produce an ammoniacal leach liquor in which dissolved cobalt is predominantly in the (III) oxidation state and a leach residue, separating the leach liquor from the leach residue, adding ammonia to saturate the leach liquor with an effective amount of anhydrous ammonia and cooling the saturated leach liquor to below 50°C whereby a triple salt of cobalt (III) hexamine sulphate, nickel (II) hexamine sulphate and ammonium sulphate is precipitated, recovering the

precipitated triple salt from the leach liquor, repulping the triple salt with an effective amount of water to selectively leach nickel (II) hexamine sulphate and ammonium sulphate and to produce a crystalline cobaltic (III) hexamine sulphate with a Co:Ni ratio of at least 100:1 and a nickel-enriched leach liquor, recovering the cobaltic (III) hexamine sulphate, dissolving the recovered cobaltic (III) hexamine sulphate in hot ammonium sulphate solution, and cooling and ammoniating the solution to precipitate recrystallized cobaltic (III) hexamine sulphate having a Co:Ni ratio of at least 1000:1 and treating the recrystallized cobaltic (II) hexamine sulphate to produce cobalt powder therefrom.

11. A process as claimed in claim 10 which comprises treating the recrystallized cobaltic (III) hexamine sulphate by recovering the recrystallized cobaltic (III) hexamine sulphate from the solution, redissolving the recrystallized cobalt (III) hexamine sulphate in hot ammonium sulphate solution, treating the said hot ammonium sulphate solution containing redissolved cobaltic (III) hexamine sulphate with an effective amount of cobalt powder to reduce Co (III) to Co (II) and with an effective amount of sulphuric acid to adjust the ammonia:cobalt mole ratio to about 2:1, and treating the said hot ammonium sulphate solution to produce cobalt powder therefrom.

12. A process as claimed in claim 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 or 11 in which the ammonia concentration in the aqueous solution or the ammoniacal leach liquor is increased to provide an effective amount of ammonia by adding anhydrous ammonia or a solution of ammonium hydroxide to provide about 170 to 190 g/L terminal free ammonia.

13. A process as claimed in claim 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 or 11 in which the ammonia concentration in the aqueous leach solution or the ammoniacal leach liquor is increased by adding anhydrous ammonia or a solution of ammonium hydroxide to provide about 170 to 190 g/L terminal free ammonia, and in which the aqueous leach solution or the ammoniacal leach liquor contains about 150 to 250 g/L ammonium sulphate.

14. A process as claimed in claim 4, 5, 6, 7, 8, 9, 10 or 11 in which the ammonia concentration in the ammoniacal leach liquor is increased by adding anhydrous ammonia or a solution of ammonium hydroxide to provide about 170 to 190 g/L terminal

free ammonia in which the ammoniacal leach liquor contains about 150 to 250 g/L ammonium sulphate and in which the saturated leach liquor is cooled to a temperature in the range of 30 to 40°C.

15. A process as claimed in claim 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 or 11 in which the precipitated triple salt is repulped with an effective amount of water at a rate to maintain titratable ammonia at about 110 to 130 g/L.

16. A process as claimed in claim 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 or 11 in which the precipitated triple salt is repulped with an effective amount of water at a rate to maintain titratable ammonia at about 110 to 130 g/L and in which nickel in solution is maintained at about 56 to 75 g/L.

17. A process as claimed in claim 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 or 11 in which the precipitated triple salt is repulped with an effective amount of water at a rate to maintain titratable ammonia at about 110 to 130 g/L, in which nickel in solution is maintained at about 65 to 75 g/L, and in which the water is maintained at a temperature in the range of about 20 to 40°C.

18. A process as claimed in claim 1, 2, 3, 4, 5, 6, 7, 8, 9, 10 or 11 in which the precipitated triple salt is repulped at a temperature in the range of about 20 to 40°C with an effective amount of water at a rate to maintain titratable ammonia at about 110 to 130 g/L, and in which nickel in solution is maintained at about 65 to 75 g/L for a retention time of 15 to 30 minutes in at least two repulp stage.

FIG. 1.

