

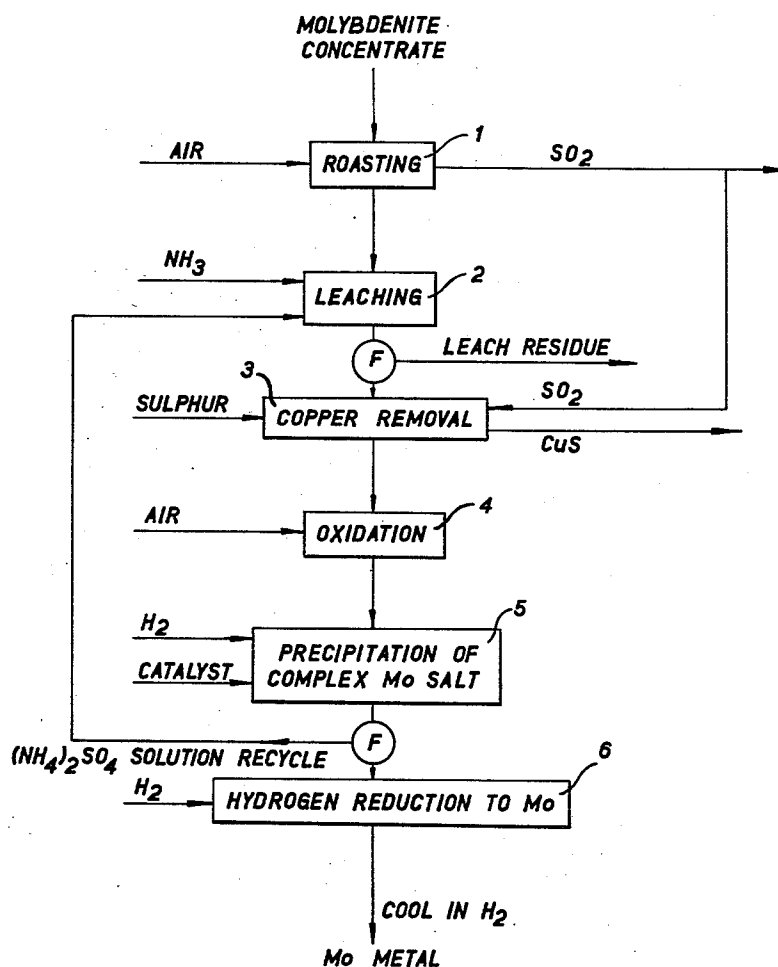
July 20, 1965

V. KUNDA

3,196,004

MOLYBDENUM RECOVERY PROCESS

Filed April 1, 1963



INVENTOR

Vasyl Kunda

by Arne J. Fors

ATTORNEY

1

3,196,004

MOLYBDENUM RECOVERY PROCESS

Vasyl Kunda, Fort Saskatchewan, Alberta, Canada, assignor to Sherritt Gordon Mines Limited, Toronto, Ontario, Canada, a company of Canada

Filed Apr. 1, 1963, Ser. No. 269,642

9 Claims. (Cl. 75-84)

This invention relates to a novel process for the production of molybdenum metal from molybdenum bearing material and more particularly from molybdenum sulphide concentrates.

Molybdenite, MoS_2 , is the primary commercial source of molybdenum metal today. The mineral generally makes up less than 1 weight percent of the ore in which it occurs, and it is separated from the gangue and recovered as a high grade bulk concentrate by flotation. Where the molybdenite is the major mineral constituent of the ore, it is separated directly from the gangue as a high grade concentrate containing 90-95% MoS_2 . However, in many cases, the molybdenite occurs as a minor constituent of copper sulphide ores in which case it is usually floated along with the copper minerals to yield a copper concentrate containing about 25 weight percent copper sulphide and less than about 2 weight percent molybdenite. The molybdenite is separated from the copper minerals by further flotation. Usually, multiple flotations and regrindings are required to obtain 50-60 percent of the molybdenite as a 90-95% weight percent MoS_2 concentrate containing less than 1 weight percent copper.

There are two methods commonly used for the treatment of molybdenite concentrates for the recovery of molybdenum metal. In the first method, the molybdenite is roasted at about 1000° F. to drive off substantially all the sulphur as sulphur dioxide, leaving molybdenum trioxide, MoO_3 . The molybdenum trioxide is leached with a dilute aqueous ammonia solution such as an aqueous ammonium carbonate or ammonium hydroxide solution. The leach solution is separated from the residue and then evaporated until ammonium molybdate, $(\text{NH}_4)_2\text{MoO}_4$, crystallizes. Molybdenum powder is prepared from the ammonium molybdate by reduction with hydrogen at a temperature between about 1900° F. to about 2000° F. In the second method the concentrate is roasted, as it is in the first method, to convert the MoS_2 to molybdenum trioxide. However, the molybdenum trioxide from the roasting step is not leached but is purified by volatilization in a silica lined furnace; and the condensed, purified MoO_3 is reduced with hydrogen to produce pure molybdenum metal.

There are a number of problems encountered in the treatment of molybdenite by these known conventional methods. In the first mentioned process, the purity of the recovered molybdenum is low. Also, the presence of copper in the molybdenite subjected to treatment by this process is objectionable and only molybdenite containing less than 1% copper can be processed. Ores treated primarily for their molybdenite content generally also contain copper, although not in sufficient quantity to make its recovery by flotation economic. This copper is usually highly disseminated in the ore and its elimination from the molybdenite concentrate is expensive and time consuming because of the exceedingly fine grinding required to ensure its effective separation. The elimination of copper is also a particularly serious problem where the molybdenite concentrate is obtained as a secondary product from molybdenum bearing copper sulphide

2

concentrates. For example, to obtain a 90-95% molybdenum concentrate containing less than 1% copper from a copper sulphide concentrate containing 1% molybdenite, about 10 flotations with at least 2 regrindings are necessary, and the recovery of molybdenite is generally only 40-60%.

The second process referred to above yields molybdenum of higher purity than is obtained in the first, but the overall recovery of molybdenum is relatively low—in the order of 60 to 70%. Also, it is apparent that this method would be expensive and time consuming.

A further problem, common to each of these known methods, is the difficulty in reducing the ammonium molybdate or the purified MoO_3 to a metallic powder having satisfactory physical characteristics and purity. Very close control of the temperature, time, and hydrogen flow rates is required, and, even then, impurities such as oxygen compounds are not always completely removed.

We have developed a method for recovering molybdenum from molybdenite which overcomes the problems of the prior processes and provides a relatively simple and efficient method of recovering high purity powdered molybdenum from this material. The method is also adaptable for application to other molybdenum bearing materials as will become evident in the following detailed description.

Briefly, in our process, the molybdenite is converted to MoO_3 by any of the conventional methods; the MoO_3 is then leached in an ammoniacal ammonium sulphate solution at ambient temperatures; the solution is purified, if required, to remove copper and other metal impurities; the molybdenum is precipitated, in the presence of a catalyst, at elevated temperature and pressure with a reducing gas such as hydrogen, as a complex salt containing about 60% Mo; and this complex salt is then reduced with hydrogen at about 1800° F. to produce pure molybdenum powder.

While the invention can be simply stated, there are important factors which affect the operation of the process. Such factors include, but are not necessarily limited to, the control of the free ammonia, molybdenum, and ammonium sulphate concentrations in the leach solution, particularly in the molybdenum precipitation step; the selective and efficient removal of copper from the leach solution, and the utilization of a catalyst or activating agent to effect precipitation of the molybdenum from the leach solution with hydrogen.

The process of this invention permits the treatment of concentrates with a high copper content with equal ease as those low in copper, thus eliminating the expensive and time consuming multiple flotation and re-grinding steps required to produce a concentrate suitable for processing by the known methods. Also, the molybdenum precipitate produced in the precipitation step is very easy to filter and wash and is much more simply and quickly reduced to pure molybdenum metal than is the corresponding product produced by the precipitation or the sublimation steps of the prior art processes.

In order to more fully describe the invention, it will be discussed in detail below with reference to the accompanying flowsheet which illustrates a preferred embodiment of the invention. The flowsheet is illustrative only and is not intended to limit the invention in any way.

The starting material, which may be a high grade molybdenite concentrate containing, for example, about 55 weight percent molybdenum or a low grade material containing about 35 weight percent molybdenum or less, is

first treated to remove the sulphur and convert it to molybdenum trioxide. Preferably, the concentrate is subjected to an oxidizing roast 1 in which the sulphur contained in the concentrate or ore is driven off as sulphur dioxide. The roast can be conducted in conventional roasting equipment adapted to provide heat under oxidizing conditions such as for example a rotary kiln or a Herreshoff multiple hearth furnace. The temperature is maintained within the range of about 1000° F. to about 1200° F. Lower temperatures result in very slow oxidation rates and higher temperatures are undesirable because the molybdenum trioxide tends to volatilize. The rate of oxidation is dependent on the oxygen flow rate as well as the temperature and, therefore, it is desirable to maintain a strong flow of air through the roasting kiln. The roasting is continued until essentially all of the molybdenite is converted to molybdenum trioxide. Other methods for converting the MoS₂ content of the concentrate to MoO₃ could be used if desired; the only requirement for the purposes of the present invention being that all or substantially all of the molybdenum content of the starting material be converted to MoO₃ before it is passed to the leaching step 2.

The leaching step 2 can be carried out in an open vessel at ambient temperatures or slightly elevated temperatures below the solution boiling point. The preferred leach solution is an aqueous ammoniacal-ammonium sulphate solution. The ammonium sulphate concentration is not critical in this step; any amount between about 0 g.p.l. (grams per litre) and about 350 g.p.l. will be satisfactory. However, because the ammonium sulphate concentration is important in the subsequent molybdenum precipitation step 5, it is preferable to maintain about 200 g.p.l. in the leach solution. The required amount of ammonium sulphate can be added to the leach solution at the outset, and thereafter proper concentration can be maintained by re-cycling the end solution from the molybdenum precipitation step 5, as shown on the flow sheet. Various adjustments in the operating procedure will, of course, be required to maintain the proper ammonium sulphate content in practice.

The free ammonia concentration in the leach solution is important. At least 1 mole of ammonia per mole of molybdenum must be provided in order to dissolve the MoO₃. The NH₃/Mo molar ratio must, therefore, be maintained above 1 and preferably in the range of 1.5 to 2.5. Higher ratios do not appreciably improve results and, in any event, it is preferable that the solution passed to the molybdenum precipitation step have as low a free ammonia content as is consistent with maintaining the molybdenum in solution. The term "free ammonia" as used herein refers to that ammonia which is titratable with sulphuric acid.

Under the leaching conditions described above, at least about 99% of the molybdenum and from about 85 to about 95% of any copper present is extracted and dissolved in the leach solution. More than 200 grams per litre of molybdenum can be readily dissolved in the leach solution, but it is preferred to adjust the leach solution-calcine proportions such that a solution containing about 150 grams per litre of molybdenum is obtained.

The leach solution containing dissolved metal values is separated from the undissolved residue by conventional procedures, such as by filtration and is passed to a copper removal step 3. In this step, any copper in solution is preferentially precipitated as copper sulphide. Any other metal impurities that will form insoluble sulphides under the conditions of the copper precipitation step, for example, nickel, cobalt and zinc will, of course, be removed from the solution along with the copper. The precipitation may be effected by any conventional means, such as by the addition of ammonium sulphide, (NH₄)₂S, sodium sulphide, or hydrogen sulphide. The preferred method, however, is to use elemental sulphur and sulphur dioxide gas as the precipitant. This method is particular-

ly suitable in the present process because it results in highly selective copper precipitation and very little molybdenum is co-precipitated with the copper; also, sulphur dioxide is readily available from the roasting step. Canadian Patent No. 586,406 describes in detail the procedure for precipitating copper from aqueous ammoniacal solutions using elemental sulphur and sulphur dioxide. A high grade copper sulphide concentrate is obtained as the product of this step and, of course, it can be treated for recovery of copper metal by conventional methods. The copper removal step can, of course, be eliminated if the leach feed material is sufficiently free of copper contamination.

The copper precipitation step will generally result in the formation of unsaturated sulphur compounds such as sulphur dioxide, thionates, and polythionates in the solution. We have found that it is necessary to remove these compounds prior to the molybdenum precipitation step 5 where a precious metal salt, such as palladium chloride, is used as the catalyst in that step. It is believed that these sulphur compounds "poison" the catalyst by reacting with it to form a non-catalytic complex compound. Where metallic powders are used as a catalyst, this "poisoning" effect is not present, and thus it is not essential to treat the solution to remove the unsaturated sulphur compounds in that case. The unsaturated sulphur compounds are removed by treating the solution at elevated temperature and pressure with a free oxygen bearing gas such as air, oxygen enriched air, or oxygen alone until they are substantially completely oxidized. An oxygen overpressure of about 10 to about 50 p.s.i. and a temperature of about 300° F. to about 400° F. for about 1 to about 2 hours is generally satisfactory.

The solution is next passed to the molybdenum precipitation step 5 where the molybdenum is precipitated from the solution by reacting it at elevated temperature and pressure with a reducing gas such as hydrogen. We have found that, in the presence of a catalyst, molybdenum is precipitated from the aqueous ammoniacal-ammonium sulphate leach solution with hydrogen gas as a complex compound containing about 60 weight percent molybdenum and about 5% ammonia.

We have found two basic types of suitable catalysts: First, salts of the precious metals silver, gold, palladium and platinum that are soluble in the ammoniacal reduction solution; secondly, finely divided (in the range of 0.5 to 300 microns) metal powders, preferably nickel powder or molybdenum powder. The soluble precious metal salts have a catalytic effect even when provided in relatively small quantities, in the order of 0.1 g.p.l. and less, for example. Satisfactory results have been obtained using about 0.005 to about 0.05 g.p.l. of PdCl₂ and Ag₂SO₄.

Metal powders must be supplied in much larger quantities in order to effectively serve as catalysts. For example, from about 5 to about 20 g.p.l. of nickel and from about 50 to about 200 g.p.l. molybdenum powder are required in order to promote the precipitation reaction at a satisfactory rate. We have found, also, that once an initial precipitation has been completed using a precious metal salt or a metal powder as the catalyst, the particles of molybdenum compound so produced can be used as the catalyst for further reductions. Because of the high cost of the precious metal salts, the preferred procedure, therefore, is to use one of these materials only to promote the initial precipitation reaction and then to recycle the material precipitated, either before or after further reduction to pure molybdenum powder, to catalyze the subsequent precipitation reactions. The size of the particles of molybdenum compound precipitated and thus the size of the final molybdenum metal particles can be controlled merely by controlling the number of precipitations or "densifications" conducted on a given batch of powder. Nickel powder should be used to initiate the precipitation reac-

tion only where its contaminating effect is not undesirable.

The free ammonia concentration of the reduction solution has a marked effect on the molybdenum precipitation reaction. There is apparently a linear relationship between NH_3/Mo molar ratio and molybdenum precipitation rate, the rate decreasing with increasing ammonia concentration. It is therefore desirable that the NH_3/Mo molar ratio of the solution be less than about 3.0 and preferably about 1.5. At this level, up to about 200 g.p.l. of molybdenum will dissolve in the solution without difficulty and the rate of molybdenum precipitation with hydrogen is satisfactory. During the precipitation reaction, only about 0.4 mole of NH_3 precipitates with each mole of molybdenum, and thus the NH_3/Mo molar ratio increases as the reaction proceeds, and the rate of precipitation decreases. Thus, for this reason also, it is preferable that the free ammonia concentration of the reduction solution be maintained at the minimum level necessary to retain the dissolved molybdenum in solution.

The ammonia sulphate concentration in the reduction solution is also important because, up to a concentration of about 350 g.p.l., the rate of molybdenum precipitation increases with increase of ammonia sulphate concentration. At concentrations below about 100 g.p.l. the rate of molybdenum precipitation becomes too slow to be useful for practical purposes, and at concentrations above about 250 g.p.l., there is very little increase in rate of precipitation with increased ammonium sulphate concentration. The preferred range for ammonium sulphate concentration is thus from about 200 g.p.l. to about 300 g.p.l.

With the free ammonia concentration and the ammonium sulphate concentration adjusted as described above, the reaction conditions under which the molybdenum precipitation reaction will proceed in the presence of a catalyst are: a temperature within the range of from about 200° F. to about 450° F., preferably from about 325° F. to about 350° F.; a hydrogen overpressure within the range of from about 200 p.s.i. to about 500 p.s.i., preferably above about 325 p.s.i.; and a molybdenum concentration between about 60 g.p.l. and about 200 g.p.l., preferably about 150 g.p.l.

Under these conditions and in the presence of a catalyst, the molybdenum precipitates as dark coloured complex compound. Although it is clear from its composition and colour that the precipitate is not ammonium molybdate, the exact nature of the precipitate is not known. However, it is believed to be an ammonia salt of a lower molybdenum oxide. The precipitate is generally composed of about 60% molybdenum, about 5% ammonia, about 0.5% sulphur, and the balance oxygen. It is in the form of uniform, oval-shaped particles of about 1 micron to about 10 microns in size.

The precipitate from the initial reduction can be separated from the solution and passed directly to the subsequent steps for conversion to molybdenum metal. In practice, however, it is preferable to use this precipitate as a catalyst for conducting further reductions, the cycles or densifications being repeated until particles of the desired size are obtained.

Upon completion of the desired number of densifications, the precipitated particles are separated from the solution by conventional liquids-solids separation procedures, such as filtration, and passed to the final hydrogen reduction step 6. The solution, or a portion thereof, containing any residual molybdenum values and ammonium sulphate may be recycled to the leaching step as shown on the flowsheet. In the final reduction step the precipitate is heated in a hydrogen atmosphere at a temperature between about 1600° F. and about 1800° F., preferably about 1750° F. Sufficient hydrogen is supplied to combine with the oxygen con-

tained in the precipitate, and the reduction-heating is continued until substantially pure molybdenum powder remains. About 1300 litres of hydrogen per pound of molybdenum precipitate has been found satisfactory. In treating the molybdenum precipitate under the foregoing conditions, there is no problem of MoO_3 volatilization as is encountered in the known commercial processes referred to above. Also, there is no problem of agglomeration of the powder if the temperature is kept below 1800° F., and the final product is generally at least 99.7% pure molybdenum.

The following examples illustrate the operation of the invention in practice.

Example 1

This example illustrates the application of the invention to a high-grade, natural molybdenite concentrate. The analysis of the concentrate was as follows:

	Percent
Mo	56.0
Cu	0.5
Fe	1.2
S	39.3
Si	0.89
Al	0.72
Insol.	5.3

300 grams of this concentrate were roasted in a rotary kiln for 1 hour at a temperature of 1200° F. An air flow of 34.0 l./min./lb. concentrate was maintained through the kiln. The roasted calcine weighed 256 grams and analyzed 64.6% Mo, 0.54% Cu, and 0.77% S. Thus, 98.3% of the sulphur had been removed. This calcine was leached in 1000 m. of aqueous ammoniacal-ammonium sulphate solution for 1 hour at a temperature of 180° F. At the outset, the leach solution contained 200 g.p.l. $(\text{NH}_4)_2\text{SO}_4$ and had an NH_3/Mo molar ratio of 2.0. After leaching, 23 grams of residue remained containing 0.69 gram of Mo, 0.17 gram of copper. Thus, 99.6% of the Mo and 88.5% of the Cu was extracted. The leach solution, which contained 1.2 g.p.l. Cu was treated with 1.5 g.p.l. $(\text{NH}_4)_2\text{S}$ and after treatment contained 0.0016 g.p.l. copper. The copper-stripped solution was oxidized with oxygen at 50 p.s.i. overpressure for 1 hour at 350° F. The bromate value of the solution was reduced from 196 to less than 1 ("bromate value" as used herein is an empirical measure of the amount of unsaturated sulphur compounds present and is equal to the ml. of 0.008 N. KBrO_3 solution required to oxidize 5 millilitres of the solution). One litre of the oxidized solution contained 150 g.p.l. Mo, 200 g.p.l. $(\text{NH}_4)_2\text{SO}_4$, and 54 g.p.l. NH_3 . 0.05 g.p.l. PdCl_2 was added as a catalyst. The autoclave was sealed, purged with hydrogen, heated to 350° F. and 350 p.s.i. hydrogen overpressure was applied. The reduction was complete in 1 hour, and the reduction end solution contained 0.1 g.p.l. Mo. Thus, 99.9% of the Mo was precipitated. The precipitate was washed and then heated at 1750° F. in a 2 inch tube furnace with an H_2 flow of 5.0 l./min. The bed thickness was 3/4". After 2 hours, the sample analyzed 99.4% Mo and after 3 hours 99.8% Mo. The final product was in powder form; the particles being about 1 to about 5 microns in size.

Example 2

In this example, the same conditions as in Example 1 above were observed except that 100 g.p.l. Mo powder were used as a catalyst in place of PdCl_2 . 98.1% of the Mo was precipitated from the solution in 90 minutes.

Example 3

This example illustrates the application of the invention to the treatment of low grade copper bearing molybdenite concentrate. The material treated analyzed 33% Mo, 5.3% Cu, 6.0% Fe, 36.0% S, 0.4% Zn, and 2.0%

insol. This material was treated under essentially the same conditions as described in Example 1:

- (1) Roasting—1200° F. for 1 hour
- (2) Leaching—180° F. for 1 hour in an aqueous ammoniacal ammonium sulphate solution containing 200 g.p.l. $(\text{NH}_4)_2\text{SO}_4$ and having an NH_3/Mo molar ratio of 2.0
- (3) Copper removal—(by $\text{S}^0 + \text{SO}_2$) for 1 hour
- (4) Oxidation—350° F., 50 p.s.i. O_2 for 1 hour
- (5) Mo precipitation from solution with H_2 —0.10 g.p.l. PdCl_2 catalyst, 350° F.—350 p.s.i. H_2 for 1 hour
- (6) Reduction of Mo precipitate with H_2 to Mo metal—1750° F. for 2 hours.

With this treatment, 95.3 weight percent of the molybdenum and 97.5 weight percent of the Cu contained in the concentrate was recovered. The Mo was in the form of a powder with an average Fisher micron size of 1.48 and contained less than 0.05 weight percent Cu.

It will be understood, of course, that modifications can be made in the preferred embodiment of the invention described and illustrated herein without departing from the scope of the invention defined by the appended claims.

What I claim as new and desire to protect by Letters Patent of the United States is:

1. A process for producing high purity molybdenum which comprises:

- (a) leaching molybdenum trioxide bearing material with an aqueous ammoniacal ammonium sulphate solution to obtain substantially complete extraction of molybdenum therefrom and its dissolution in the leach solution;
- (b) maintaining the free ammonia concentration of said leach solution at at least about 1 mole of ammonia per mole of molybdenum in solution and maintaining the ammonium sulphate concentration of said leach solution at above about 100 grams per litre;
- (c) separating undissolved residue from the leach solution and reacting said solution, in the presence of a catalyst, at a temperature of from about 200° F. to about 450° F. with hydrogen gas provided in sufficient amount to maintain a hydrogen partial pressure above about 200 pounds per square inch, said catalyst being at least one member selected from the group consisting of salts of the precious metals silver, gold, palladium and platinum which are soluble in the ammoniacal leach solution and finely divided metal powders;
- (d) continuing said reducing reaction to precipitate molybdenum from said solution as a complex molybdenum salt; and
- (e) separating the precipitated complex molybdenum salt from the last-mentioned solution and heating said precipitate, in the presence of a reducing agent, at a temperature below that at which molybdenum trioxide volatilizes to reduce the molybdenum precipitate to substantially pure molybdenum metal.

2. The process according to claim 1 in which the catalyst is palladium chloride provided in amount between about 0.005 and 0.5 gram per litre.

3. The process according to claim 1 in which the catalyst is finely divided molybdenum powder provided in amount equal to at least about 50 grams per litre.

4. The process according to claim 1 in which the catalyst is finely divided nickel powder provided in amount equal to at least about 5 grams per litre.

5. The process according to claim 1 in which, the complex molybdenum salt produced in the initial operation of the process is utilized as the catalyst for subsequent operations of the process.

6. A process for the production of molybdenum from molybdenite ores and concentrates containing metal impurities including at least copper in which the molybdenite bearing material is first roasted in the presence of

oxygen to convert the molybdenum content thereof to molybdenum trioxide which comprises:

- (a) leaching the roasted molybdenum trioxide containing material with an aqueous ammoniacal ammonium sulphate solution to obtain substantially complete extraction of molybdenum therefrom and its dissolution in the leach solution;
- (b) maintaining the free ammonia concentration of said leach solution within the range of from about 1 to about 2.5 moles of ammonia per mole of molybdenum in solution and maintaining the ammonium sulphate concentration of said leach solution at above about 100 grams per litre;
- (c) separating undissolved residue from the leach solution and reacting said leach solution with a sulphidizing agent to precipitate metal impurities, including copper, from said solution as sulphides;
- (d) separating said sulphide precipitate from the solution and reacting the said solution at a temperature of from about 200° F. to about 450° F. in the presence of a catalyst selected from the group comprising finely divided molybdenum powder and finely divided nickel powder with hydrogen provided in amount sufficient to maintain a partial pressure of hydrogen of from about 200 to about 500 pounds per square inch;
- (e) continuing said reducing reaction to precipitate molybdenum from said solution as a complex salt; and
- (f) separating the complex salt precipitate from the last-mentioned solution and heating said precipitate at a temperature below about 1800° F. in the presence of hydrogen to reduce the precipitate to substantially pure molybdenum metal.

7. The process according to claim 6 in which the finely divided metal powder catalyst is molybdenum powder and is provided in amount within the range of about 50 to about 200 grams per litre.

8. A process for the production of molybdenum from molybdenite ores and concentrates containing metal impurities including at least copper in which the molybdenite bearing material is first roasted in the presence of an oxygen bearing gas to convert substantially all the molybdenum content thereof to molybdenum trioxide which comprises:

- (a) leaching the roasted molybdenum trioxide bearing material with an aqueous ammoniacal ammonium sulphate solution to obtain substantially complete extraction of molybdenum therefrom and its dissolution in the leach solution;
- (b) maintaining the free ammonia concentration of said leach solution within the range of from about 1 to about 2.5 moles of ammonia per mole of molybdenum in solution and maintaining the ammonium sulphate concentration of said leach solution at above about 100 grams per litre;
- (c) separating undissolved residue from the leach solution and reacting the solution with a sulphidizing agent to precipitate metal impurities including copper from said solution as sulphides;
- (d) separating said sulphide precipitate from the solution and oxidizing said solution with a sulphur-free oxygen bearing gas at a temperature above about 300° F. and under a partial pressure of oxygen above about 10 p.s.i.;
- (e) reacting the oxidized solution at a temperature of from about 200° F. to about 450° F. in the presence of a catalyst selected from the group consisting of palladium salts, silver salts, platinum salts and gold salts, said salts being soluble under the conditions of reaction, with hydrogen gas provided in a sufficient amount to maintain a partial hydrogen pressure of from about 200 to about 500 pounds per square inch;

9

(f) continuing said reducing reaction to precipitate molybdenum from said solution as a complex molybdenum salt; and

(g) separating said complex molybdenum salt precipitate from the last-mentioned solution and heating said precipitate at a temperature below about 1800° F. in the presence of hydrogen to reduce the precipitate to substantially pure molybdenum metal.

9. The process according to claim 8 in which the catalyst is palladium chloride provided in amount between about 0.005 and about 0.1 gram per litre.

10

References Cited by the Examiner

UNITED STATES PATENTS

2,796,343	6/57	Show	75—109
2,822,261	2/58	Mackiw	75—103
2,836,485	5/58	Schaufelberger	75—0.5
3,053,614	9/62	Foss	75—84

OTHER REFERENCES

Pauling: General Chemistry, 2nd Ed., W. H. Freeman Co., San Francisco, 1954, page 446.

BENJAMIN HENKIN, *Primary Examiner*.