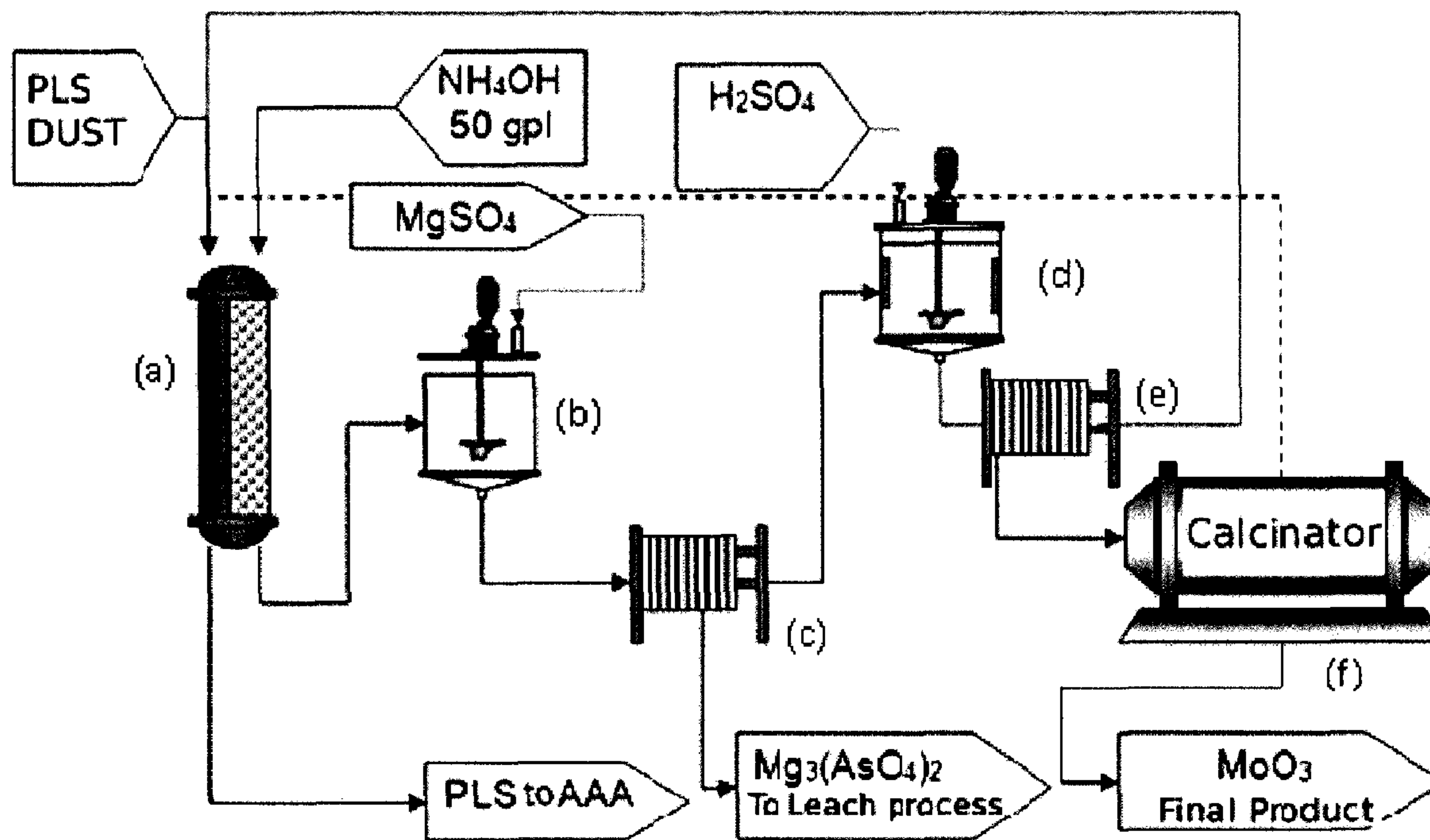




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 (72) **Inventeurs/Inventors:**  
 LAGNO SANCHEZ, FELIPE ANDRES, CL;  
 SEPULVEDA LETELIER, RICARDO FERNANDO, CL;  
 ACUNA GOYCOLEA, MARCELO GUSTAVO, CL  
 (73) **Propriétaire/Owner:**  
 ECOMETALES LIMITED, CL  
 (74) **Agent:** ROBIC

(54) **Titre : PROCÉDE DE RECUPERATION DE MOLYBDENE DE QUALITE TECHNIQUE A PARTIR DE SOLUTIONS ACIDES  
 DILUEES DE LIXIVIATION, HAUTEMENT CONCENTREES EN ARSENIC PROVENANT DE RESIDUS METALLURGIQUES**  
 (54) **Title: PROCESS FOR RECOVERY OF TECHNICAL GRADE MOLYBDENUM FROM DILUTED LEACHING ACID SOLUTIONS  
 (PLS), WITH HIGHLY CONCENTRATED ARSENIC, FROM METALLURGICAL RESIDUES**



(57) **Abrégé/Abstract:**

The invention relates to a method for recovering technical-grade molybdenum from diluted acid leaching solutions (PLS) that have a high arsenic concentration, said method comprises the following steps consisting in: (a) bringing a pre-filtered acid leaching solution (PLS), originating from the leaching of smelter dust, into contact with an anionic ion-exchange resin; (b) washing the loaded resin with water; (c) extracting the molybdenum from the ion-exchange resin with an alkaline ammonium regenerant solution at a pH value of between 8 and 12, in order to form ammonium molybdenum in solution; (d) washing the unloaded resin

**(57) Abrégé(suite)/Abstract(continued):**

with water; (e) adding iron and/or magnesium salts to the recovered ammoniacal solution, in order to obtain a precipitate which is transferred to the arsenic abatement step and a solution containing ammonium molybdate in solution; (f) adding sulphuric acid to the arsenic-free ammoniacal solution in order to precipitate the molybdenum in the form of ammonium molybdate in an acid environment at a pH value of between 1.5 and 4; (g) separating the precipitate formed by filtering the molybdate and re-circulating the solution obtained with the initial PLS solution; (h) calcining the separated precipitate in order to obtain ammonia and molybdenum trioxide; and (i) recovering the released ammonia for its subsequent use in the method as a recirculated regenerant solution.

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ECOMETALES LIMITED [CL/CL]; Nueva de Lyon 72,  
Piso 17, Of. 1701, Santiago, 7510078 (CL).

(72) Inventores; e

(75) Inventores/Solicitantes (para US solamente): LAGNO SÁNCHEZ, Felipe Andrés [CL/CL]; Nueva de Lyon 72, Piso 17, Of. 1701, Santiago, 7510078 (CL). SEPÚLVEDA LETELIER, Ricardo Fernando [CL/CL]; Nueva de Lyon 72, Piso 17, Of. 1701, Santiago, 7510078 (CL). ACUÑA GOYCOLEA, Marcelo Gustavo [CL/CL]; Nueva de Lyon 72, Piso 17, Of. 1701, Santiago, 7510078 (CL).

(74) Mandatario: CLARKE MODET Y CO CHILE;  
Huérfanos N°835, Piso 10, Santiago, Santiago, 8320176  
(CL).

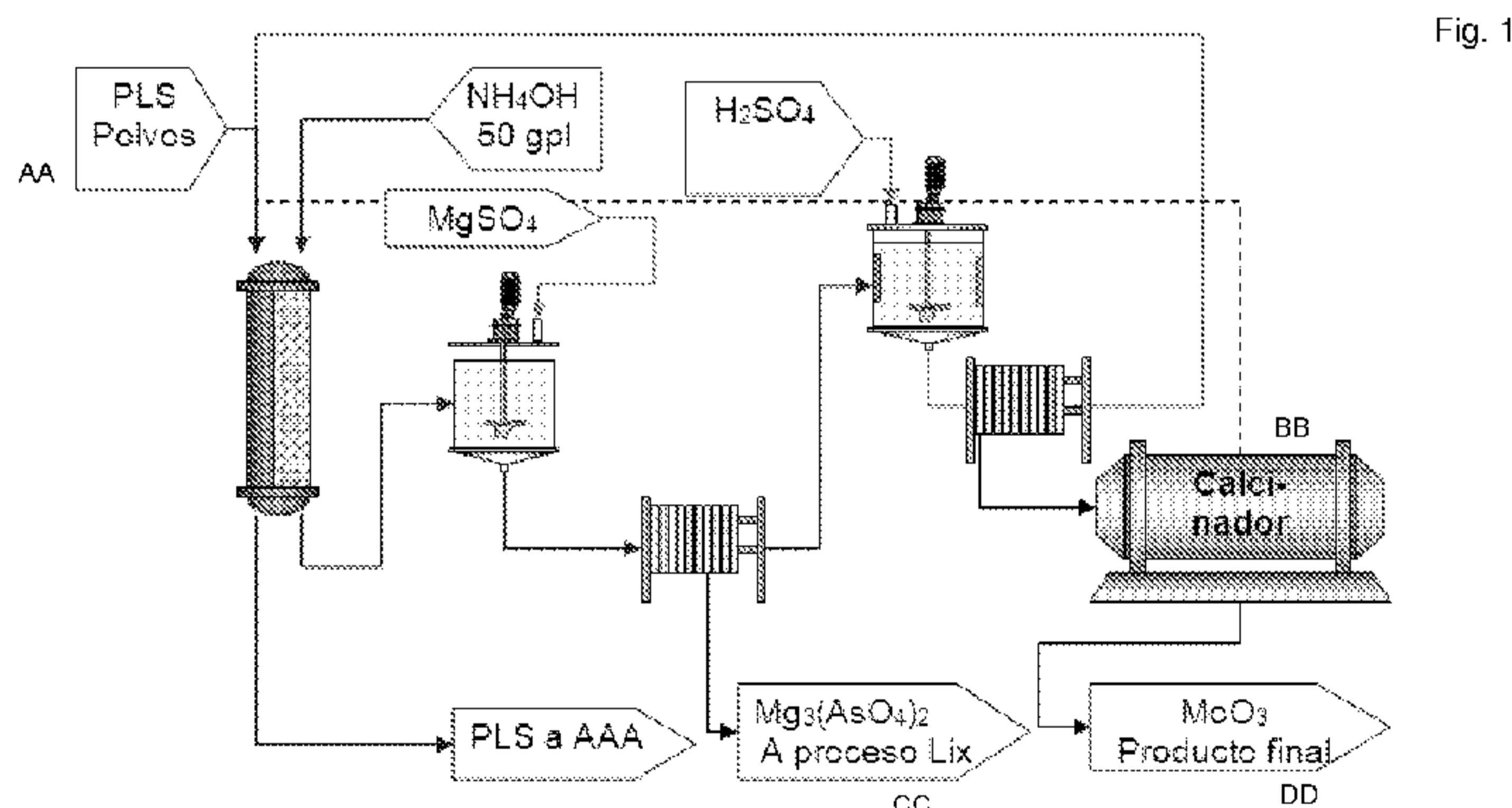
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[Continúa en la página siguiente]

(54) Title: METHOD FOR RECOVERING TECHNICAL-GRADE MOLYBDENUM FROM DILUTED ACID LEACHING SOLUTIONS (PLS) THAT HAVE A HIGH ARSENIC CONCENTRATION AND ORIGINATE FROM METALLURGICAL WASTE

(54) Título : PROCESO PARA LA RECUPERACIÓN DE MOLIBDENO GRADO TÉCNICO A PARTIR DE SOLUCIONES ÁCIDAS DILUIDAS DE LIXIVIACIÓN (PLS), ALTAMENTE CONCENTRADAS EN ARSÉNICO, PROVENIENTES DE RESIDUOS METALÚRGICOS



(57) Abstract: The invention relates to a method for recovering technical-grade molybdenum from diluted acid leaching solutions (PLS) that have a high arsenic concentration, said method comprises the following steps consisting in: (a) bringing a pre-filtered acid leaching solution (PLS), originating from the leaching of smelter dust, into contact with an anionic ion-exchange resin; (b) washing the loaded resin with water; (c) extracting the molybdenum from the ion-exchange resin with an alkaline ammonium regenerant solution at a pH value of between 8 and 12, in order to form ammonium molybdenum in solution; (d) washing the unloaded resin with water; (e) adding iron and/or magnesium salts to the recovered ammoniacal solution, in order to obtain a precipitate which is transferred to the arsenic abatement step and a solution containing ammonium molybdate in solution; (f) adding sulphuric acid to the arsenic-free ammoniacal solution in order to precipitate the molybdenum in the form of ammonium molybdate in an acid environment at a pH value of between 1.5 and 4; (g) separating the precipitate formed by filtering the molybdate and re-circulating the solution obtained with the initial PLS solution; (h) calcining the separated precipitate in order to obtain ammonia and molybdenum trioxide; and (i) recovering the released ammonia for its subsequent use in the method as a recirculated regenerant solution.

(57) Resumen:

[Continúa en la página siguiente]

**WO 2013/030741 A3** **Declaraciones según la Regla 4.17:**

- sobre la identidad del inventor (Regla 4.17(i))
- sobre el derecho del solicitante para solicitar y que le sea concedida una patente (Regla 4.17(ii))
- sobre el derecho del solicitante a reivindicar la prioridad de la solicitud anterior (Regla 4.17(iii))
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La presente invención reivindica un proceso para la recuperación de molibdeno grado técnico a partir de soluciones ácidas diluidas de lixiviación (PLS), altamente concentradas en arsénico, donde el proceso comprende las etapas de: (a) poner en contacto una solución ácida de lixiviación (PLS) previamente filtrada proveniente de la lixiviación de los polvos de fundición con una resina aniónica de intercambio iónico; (b) lavar con agua la resina cargada; (c) extraer el molibdeno de la resina de intercambio iónico con una solución regenerante alcalina de amonio a un valor de p H entre 8 y 12, para formar molibdato de amonio en solución; (d) lavar con agua la resina descargada; (e) agregar sales de magnesio y/o hierro a la solución amoniaca recuperada, para obtener un precipitado el cual es llevado a la etapa de abatimiento de arsénico, y una solución que contiene el molibdato de amonio en solución; (f) agregar ácido sulfúrico a la solución amoniaca libre de arsénico, para precipitar el molibdeno en la forma de molibdato de amonio en ambiente ácido a un valor de p H entre 1,5 y 4; (g) separar el precipitado formado mediante filtración del molibdato y recircular la solución obtenida a la solución inicial de PLS; (h) calcinar el precipitado separado para obtener trióxido de molibdeno y amoníaco; y (i) recuperar el amoníaco desprendido para su posterior retorno al proceso como solución regenerante recirculada.

PROCESS FOR RECOVERY OF TECHNICAL GRADE MOLYBDENUM FROM DILUTED LEACHING ACID SOLUTIONS (PLS), WITH HIGHLY CONCENTRATED ARSENIC, FROM METALLURGICAL RESIDUES.

**SPECIFICATION**

The present invention discloses a process for obtaining technical grade molybdenum trioxide from diluted leaching acid solutions (PLS), with highly concentrated arsenic, antimony or bismuth. Said solution is obtained by leaching of metallurgic residues with a high concentration of these impurities, such as casting powders.

More specifically, the present invention discloses a process for recovering molybdenum through ionic exchange, through which molybdenum is separated from other metals also present in said solution, through the use of ionic exchange resins and controlled precipitation of As, Sb, and Bi, with magnesium or iron salts, followed by an ammonium molybdate precipitation, which is later calcinated to obtain technical grade molybdenum trioxide.

BACKGROUND OF THE INVENTION.

A mining process or method is the summation of methods through which, starting from a deposit, metals and/or metallic compounds of commercial purity and quality are obtained, in a profitable manner and with an acceptable environmental impact.

In the known leaching process, one or many mineral values contained in an ore or a concentrate are dissolved, generally using an aqueous solution of a leaching agent. The term can also be extended to include dissolving secondary materials, such as scrap metals, residues, and waste.

Leaching produces an aqueous solution rich in extracted ions of the valued metal (PLS, *pregnant leaching solution*) from which it is possible to separate this metal and recovering it with a high level of purity. Also, a solid residue or gravel is produced, that, ideally, has a sufficiently low level of valued leached minerals, as to discard it in tailings or dump.

If the solid leaching residue is impregnated with salts or precipitates that eventually can release toxic agents once exposed to the environment, before discard, the residue must be properly treated in order to achieve elimination or stabilization of the potentially contaminant compounds. In some cases, leaching can have a different objective than the one previously described. For example, when a concentrate is leached for selectively extracting certain impurities, and thus increase the quality of concentrate (for example during removal of

copper from molybdenite concentrates).

In purification and enrichment processes of solutions, leaching processes are not necessarily selective and, in consequence, produce leaching solutions containing, besides the metal of interest, a range of impurities. This, added to the fact that concentration of the metal of interest may not be too high, making impossible a direct recovery of the metal of interest from leaching solution, in this way, these solutions must be previously treated through purification and enrichment steps.

Purification allows to eliminate impurities, effectively isolating valuable elements. Enrichment of solutions is also particularly beneficial for reducing the volumes of solution to be treated in subsequent steps of metal recovery. This contributes to lessen investment costs and increase efficiency in recovery.

In the present invention, a process for recovery of molybdenum as molybdenum trioxide, from diluted acid leaching solutions of metallurgic residues, having a high concentration of arsenic, antimony, or bismuth, is disclosed.

The object of the present invention is recovering molybdenum, through ion exchange, contained in diluted leaching solutions containing molybdenum, but having a high concentration of arsenic, among others, in such a manner that a molybdenum product is obtained efficiently and profitably.

Prior art shows documents disclosing molybdenum recovery through ion exchange. Such is the case of US Patent 4,891,067, disclosing a process for selective separation of molybdenum present in an acid solution at pH 2 and containing molybdenum and at least one of the elements in the group conformed by uranium, iron, arsenic, phosphorous, silicon, and vanadium. Said process comprises to contact the acid solution with a stationary phase consisting of a resin with an active oxime group and eluting said stationary phase with an alkaline solution to recover molybdenum. US Patent 4,596,701 discloses a procedure for purifying molybdenum trioxide, specifically disclosing a method for preparing ammonium molybdate comprising contacting said concentrate with an aqueous solution of sulfuric acid, ammonium, ammonium sulfate and persulfate, for solubilizing at least 2% of the molybdenum values present in said concentrate.

A Chilean patent application CL N° 3137-2005 discloses a process for molybdenum and copper extraction, which are contained in solidified slags from fusion processes in copper concentrates. This document does not interfere with the present patent application since the slag does not contain arsenic, therefore treating a technical problem different to the one addressed in the present patent application.

None of the previously mentioned processes interfere with the present application since said

documents of the prior art show processes with different operative variables, and therefore, do not provide the benefits obtained with the present invention, which, as disclosed in the present application, consist on efficiently and profitably recovering technical grade  $\text{MoO}_3$  from leaching solutions with high arsenic content. Through the present invention, a technical grade molybdenum trioxide is obtained, which means having a molybdenum content over 58%, and a content of non-regulated metals, such as As, Sb, and Bi, in values lower than 0.1%.

#### SUMMARY OF THE INVENTION

In accordance with one aspect, there is provided a process for recovery of technical grade molybdenum from diluted leaching acid solutions (PLS) containing highly concentrated arsenic, from metallurgical residues, wherein the process comprises the steps of:

- (a) contacting a pre-filtered acid leaching solution (PLS), from leaching of casting powders, with an anionic ion exchange resin to produce a charged ion exchange resin;
- (b) washing the charged ion exchange resin of step (a) with water;
- (c) extracting molybdenum from the washed ion exchange resin using an alkaline regenerating ammonium solution having a pH value between 8 and 12, to form ammonium molybdate in solution, which is recycled for adjusting molybdenum concentration;
- (d) washing the resin of step (c) with water;
- (e) adding magnesium and/or iron salts to the ammonium molybdate solution recovered in step (c) to form a precipitate of  $\text{Mg}_3(\text{AsO}_4)_2$  and/or  $\text{FeAsO}_4$  which is carried to an arsenic abatement step, and a solution containing ammonium molybdate in solution;
- (f) adding sulfuric acid to the ammonium molybdate solution obtained in step (e) for precipitating molybdenum in the form of ammonium molybdate ( $(\text{NH}_4)_4\text{Mo}_8\text{O}_{26}$ ) in an acid medium having a pH value between 1.5 and 4;
- (g) separating the precipitate formed in step (f) by filtrating ammonium molybdate and recycling the obtained solution to the PLS solution of step (a);
- (h) calcining the precipitate separated in step (g) to obtain molybdenum trioxide ( $\text{MoO}_3$ ), and ammonia; and
- (i) recovering ammonia produced in step (h) for returning to the process as recycled regenerating solution.

#### BRIEF DESCRIPTION OF THE FIGURES

Figure 1. Flowsheet. A "PLS Powder" solution is input to a column containing a IX (a) resin.

The molybdenum is extracted from the ion exchange resin with an alkaline regenerating ammonium solution. The resulting solution containing molybdenum is cleaned by immediately adding magnesium sulfate (b), causing precipitation of arsenic selectively (c). The cleaned ammonium solution is acidified (d), precipitating a molybdenum salt. This salt is recovered by filtration (e) and calcined (f) to generate a high quality trioxide. The remaining solution is recycled to step (a).

Figure 2. Detail of the cleaning of ammonium. By adding magnesium sulfate, an arsenic precipitate (sampling points indicated) is generated. Molybdenum precipitation by acid results in a cake, which will be washed by re-pulping with process water and re-filtration.

Figure 3. Molybdenum precipitate heating up curve versus time, wherein temperature increase in steps, allowing the completion of each of the decomposition sub-stages, before proceeding to the next stage (or step). This profile is an example of the importance of the knowledge of the chemical decomposition - direct heating to the final temperature result in a substandard product.

#### DESCRIPTION OF THE INVENTION

The process of the present invention discloses recovery of molybdenum through ion exchange, followed by an increase in the concentration of molybdenum in the regenerant, precipitating impurities, precipitating molybdenum, passing through drying, and calcination, to obtain the final product of technical grade molybdenum trioxide, as shown in Figure 1.

More specifically, the present invention discloses a process to recover over 70% or more, specifically around 90% of molybdenum present in PLS as technical grade  $\text{MoO}_3$ . Specifically, the invention consists in selective recovery of molybdenum, in the form of  $\text{MoO}_3$ , from PLS solution generated from leaching of casting powders.

The present invention develops a process for recovering molybdenum through ion exchange. The process has been validated in a pilot plant and the fundamental aspects of the process, validation steps, and methodology of industrial scaling, as disclosed in the present invention, show a novel and inventive process.

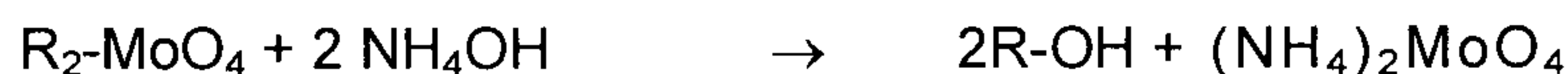
The ion exchange process considers two steps which repeat in ion exchange cycles: charge and regeneration of the resin; each one followed of water washing steps. The charge process of the resin consists on capturing molybdenum ion in molybdate form from the acid leaching solution, while the regeneration process of the resin consists on re-extraction or

discharge of the molybdate ion using an alkaline regenerating solution. The equations corresponding to the process are shown below:

Resin charge step:



Resin regeneration step:



Wherein R represents the ion exchange resin. The resin used in this process is of anionic weak base or anionic weak/string base type, presenting functional groups selected among secondary, tertiary, tertiary/quaternary amines, and polyamines.

Molybdenum extraction with ion exchange resins is performed contacting PLS solution containing Mo with an anionic resin. This contact can be discontinuous, adding the resin to the solution and agitating, or continuously, by using columns.

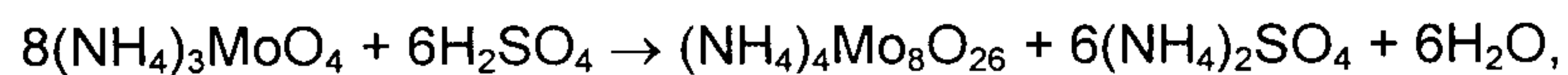
Ion exchange is highly selective for Mo using a particular resin. However, traces of undesirable elements that are contained in the solution can be entrapped in the resin, in particular in the present application As, Sb and Bi. Therefore, it is required to send the charged solution to the step of impurity removal (figure 2).

Afterwards, charged Mo must be discharged from the resin contacting the charged resin with an alkaline solution of ammonium hydroxide, wherein a Mo charged solution is obtained, contaminated with As, Sb, or Bi which are partially co-extracted with Mo.

Removal of these impurities can be performed by precipitating with a magnesium or iron salt, obtaining a solid product that must be treated for further disposition and obtaining a regenerating solution charged with Mo and virtually free of impurities (Fig. 2). Chemical reactions using magnesium sulfate for arsenic, antimony, and bismuth precipitations are:

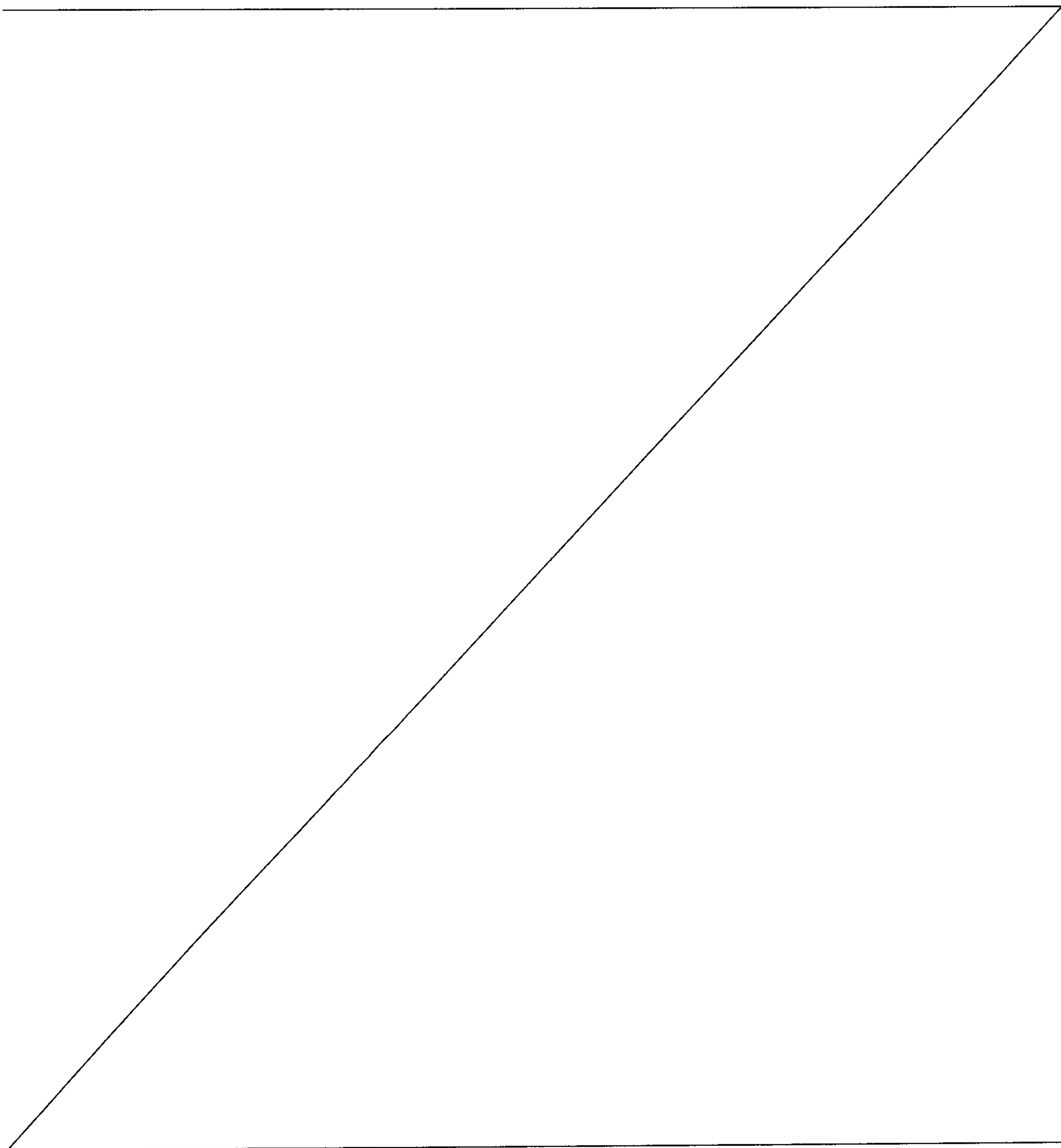


After removal of As, Sb, and Bi, a charged regenerating solution is obtained, free of impurities, which is then sent to the Mo precipitation step (Fig. 2) by adding sulfuric acid, Mo precipitates as ammonium molybdate with an efficiency higher than 70%, according to the following reaction:



The last step to obtain molybdenum trioxide is the calcination step, wherein the ammonium molybdate precipitate is subjected to controlled heating from 20 to 700°C. The temperature profile used (Figure 3) in the calcination step of the present invention considers:

1.- Heating from 20 to 260°C and holding for approximately 20 minutes to 2 hours, for removing hydration water.



- 2.- Heating from 260 to 370°C and holding for approximately 20 minutes to 2 hours, for removing water and ammonia.
- 3.- Heating from 370 to 500°C and holding for approximately 20 minutes to 2 hours, for removing As as arsenic oxide.
- 4.- Heating from 500 to 700°C and holding for approximately 20 minutes to 2 hours, for decomposition and desorption of sulfur and arsenic.

In summary, using the present invention allows to produce molybdenum trioxide with impurities, such as As, Sb, and Bi, with values lower than 0.1%. The present invention in more detail has the following correlative steps:

- 1.- Contacting acid aqueous solution (PLS) previously filtered with a pH lower than 1.6, with an ion exchange resin of anionic type. Such as for example, the ones described in Table 1.
- 2.- Wash with water the resin post-charge to avoid potential precipitation of solid elements that are dissolved in PLS due to its acidity (such as iron) which could precipitate when in contact with the regenerant due to the basic pH of the ammonium hydroxide solution.
- 3.- Extracting molybdenum from the ion exchange resin with an alkaline solution of ammonium hydroxide in a concentration range from around 5 g/L to 150 g/L, reaching a pH of around 8 to 12, more preferentially between 8.5 and 9.5, in the form of ammonium molybdate in solution.
- 4.- Wash with water the resin post-discharge, similar to step 2.
- 5.- Adding a magnesium or iron salt, such as for example magnesium sulfate, magnesium chloride, or ferric sulfate, to the solution obtained in step 3, to obtain a pulp with an arsenic and other impurities precipitate, which is separated in 2 lines: the ammonia solution obtained, that contains Mo, passes to the molybdenum precipitation step and the solid obtained is carried to an abatement step and external disposition to the process of the present invention.
- 6.- Adding  $H_2SO_4$  to the ammonia solution obtained in step 5 to precipitate the molybdenum in the form of ammonium molybdate  $((NH_4)_4Mo_8O_{26})$  in an acid medium with a pH between 1.5 to 4, more preferentially at pH 3.3, and in a temperature range between 50°C to 90°C, more preferentially at 70°C.
- 7.- Separating the precipitate of step 6, using filtration of molybdate, and the solution obtained is recirculated to mix it with the initial solution of PLS.
- 8.- Calcinating in a ramp or steps of temperature between 20 to 700°C the filtered product of step 7 to technical grade molybdenum trioxide ( $MoO_3$ ). This step allows removal of sulfur,

arsenic, and ammonium traces, therefore the product complies with the requirements for the market of technical grade molybdenum trioxide.

9.- Additionally recovering ammonia generated in calcination step 8 in a condenser and/or gas scrubber, for further return to the process as regenerant.

Weak (WBA) and mixed weak/strong (WBA/SBA) anionic ion exchange resins were tested for the process of the present invention. As an example and without limiting the invention, the resins indicated in Table 1 were tested. Resins A170/4675 and A100 Mo are manufactured by Purolite. Resins A365 (LEWATIT® A 365), MP64 (LEWATIT® M+ MP 64) and MP62 (LEWATIT® MP 62) are manufactured by Lanxess. After a large number of batch and in column tests, the results described in Table 2 were obtained.

Table 1. Specification of resins used in batch and column tests.

Resin	Type	Base	Appearance	Size (µm)	Structure	Functional Group	Capacity (eq./L)
A 170/4675	WBA	Free	Spherical (pearls)	875 +/- 125	Macroporous PES/DVB	Complex Amine	1.3 (base Free)
A 100 Mo	SBA/WBA	Chloride	Spherical (pearls)	800 to 1300	Macroporous PES/DVB	Tertiary amine	3.8 (base Cl-)
MP 64	SBA/WBA	Free	Spherical (pearls)	590 +/- 50	Macroporous PES/DVB	Tertiary/quaternary amine	1.3 (base Free)
MP 62	WBA	Free	Spherical (pearls)	470 +/- 60	Macroporous PES/DVB	Tertiary amine	1.7 (base Free)
A 365	WBA	Free	Spherical (pearls)	> 400	Acrylate gel/DBV	Polyamine	3.4 (base Free)

Table 2. Results obtained for column tests.

Resin	Mo Extraction efficiency from PLS	Mo Re-extraction efficiency from resin
MP64	90%	52.3%
MP62	86%	65%
A365	61%	47%
A170/4675	62%	72.1%
A100Mo	81%	56.1%

As can be seen in Table 2, all resins present an acceptable percentage of affinity for Mo, standing out resins MP64, MP62 and A100Mo. Nevertheless, in Mo re-extraction capacity from the resin, A170/4675 and MP62 resins stand out.

The variables that are considered relevant for obtaining a product with less impurities are reflected in Table 3.

Table 3. Analyzed operating variables for precipitation of impurities and Mo.

Variable	Level	
	Low	High
Mo concentration in charged regenerating solution	≈ 5,000 mg/L	≈ 10,000 mg/L
Feed solution pH for precipitation of As	≈ 8.5	≈ 9.5
Feed solution temperature for precipitation of As	room temperature	60 °C
Residence time for precipitation of As	30 min	120 min
As concentration in feed for precipitation of As	2,500 mg/L	≈ 8,000 mg/L
Sb concentration in feed for precipitation of Sb	39 mg/L	≈ 150 mg/L
Concentration of Bi in feed for precipitation of Bi	29.2 mg/L	≈ 64.9 mg/L
Feed solution pH for precipitation of Mo	≈ 7.3	≈ 8.5
Residual solution pH for precipitation of Mo (at 70 °C)	≈ 2.8	≈ 3.3
Residence time for precipitation of Mo	30 min	120 min

#### EXAMPLE OF APPLICATION

330 L of PLS solution were contacted with 6 L of Lanxess® MP-62 resin (for exemplification, and without limiting the invention). The resin was disposed in a static column which allowed passing PLS solution at a 4.5 l/h flow rate, after this period, a “refine” or Mo-free solution was obtained, as shown in Table 4.

Afterward, 12 L water were passed, at a 4.5 L/h flow rate for washing the resin. After washing, the resin was regenerated, extracting the captured Mo, passing an alkaline ammonium hydroxide solution (50 g/L NH<sub>4</sub>OH) through the column. This procedure was performed for 47 cycles of charge/discharge, using in each cycle 330 L of fresh PLS solution. The regenerating solution was not renovated since its concentration increases in each cycle; maintaining the pH near to 9.

Table 4. PLS solution concentration before and after Mo extraction

	[Mo] (mg/L)	[Sb] (mg/L)	[Bi] (mg/L)	[As] (mg/L)
PLS Solution	315	93	78	13,840
Refine solution	26	51	59	13,692
Charged regenerating solution	11,740	2,230	951	8,330

The charged regenerating solution was fed to a 5 L reactor for precipitating impurities, contacting 215.8g of magnesium sulfate (5% above stoichiometric value). The pulp was left reacting for 120 min. The solid was filtered and washed for disposition. The results obtained show a high efficiency in precipitation of impurities, obtaining a solution with only 41 mg/L of As and concentrations of Sb and Bi lower than 10 mg/L. No co-precipitation with Mo was detected.

Afterwards, the treated or clean solution was loaded to the Mo precipitation reactor, wherein it was heated to 60°C. Under this condition, sulfuric acid was added, turning the solution pH to 3.3.

In these conditions, a precipitation of Mo of 70% was obtained. The solid was filtered and washed with water in a ratio of 3 parts of water for 1 part of solid in weight. From this procedure, a solid with the concentrations described in Table 5 was obtained.

Table 5. Ammonium molybdate concentrations.

Mo	Sb	Bi	As
57.4%	0.07%	0.02%	4.1%

This solid was further calcinated in an electrical oven for 3.5 h, reaching a temperature of 650°C. The results of the concentration for the product are shown in Table 6.

Table 6. Concentrations of elements in the produced molybdenum trioxide.

Mo	As	Sb	Bi	K	Fe	Ca	Al	Cu	Mg
64%	0.085%	0.027%	0.016%	0.026%	0.018%	0.002%	0.002%	0.010%	0.022%

Using the present invention, the final molybdenum product exceeds the conventional commercial standards of concentration. The obtained purity is compatible with the conventional market of technical grade molybdenum trioxide.

## CLAIMS

1- A process for recovery of technical grade molybdenum from diluted leaching acid solutions (PLS) containing highly concentrated arsenic, from metallurgical residues, wherein the process comprises the steps of:

(a) contacting a pre-filtered acid leaching solution (PLS), from leaching of casting powders, with an anionic ion exchange resin to produce a charged ion exchange resin;

(b) washing the charged ion exchange resin of step (a) with water;

(c) extracting molybdenum from the washed ion exchange resin using an alkaline regenerating ammonium solution having a pH value between 8 and 12, to form ammonium molybdate in solution, which is recycled for adjusting molybdenum concentration;

(d) washing the resin of step (c) with water;

(e) adding magnesium and/or iron salts to the ammonium molybdate solution recovered in step (c) to form a precipitate of  $Mg_3(AsO_4)_2$  and/or  $FeAsO_4$  which is carried to an arsenic abatement step, and a solution containing ammonium molybdate in solution;

(f) adding sulfuric acid to the ammonium molybdate solution obtained in step (e) for precipitating molybdenum in the form of ammonium molybdate ( $(NH_4)_4Mo_8O_{26}$ ) in an acid medium having a pH value between 1.5 and 4;

(g) separating the precipitate formed in step (f) by filtrating ammonium molybdate and recycling the obtained solution to the PLS solution of step (a);

(h) calcining the precipitate separated in step (g) to obtain molybdenum trioxide ( $MoO_3$ ), and ammonia; and

(i) recovering ammonia produced in step (h) for returning to the process as recycled regenerating solution.

2.- Process for recovery of technical grade molybdenum according to claim 1, wherein the ion exchange resin used is selected among weak base type or weak/strong base type, presenting functional groups selected among tertiary amines, tertiary/quaternary amines, and polyamines.

3.- Process for recovery of technical grade molybdenum according to claim 2, wherein the ion exchange resin is A170/4675 manufactured by Purolite, LEWATIT® M+MP6

manufactured by Lanxess, LEWATIT® MP62 manufactured by Lanxess, A100 Mo manufactured by Purolite or LEWATIT® A 365 manufactured by Lanxess.

4.- Process for recovery of technical grade molybdenum according to claim 1, wherein in step (e) magnesium sulfate is used.

5.- Process for recovery of technical grade molybdenum according to claim 1, wherein the regenerating ammonium solution in step (c) has a pH value between 8.5 and 9.5.

6.- Process for recovery of technical grade molybdenum according to claim 1, wherein in step (h) calcination is performed by using temperature increase in steps.

7.- Process for recovery of technical grade molybdenum according to claim 6, wherein in step (h) calcination is performed at a temperature between 20 and 700°C.

8.- Process for recovery of technical grade molybdenum according to claim 1, wherein recovery of ammonia produced in step (i) is performed in a condenser.

9.- Process for recovery of technical grade molybdenum according to claim 1, wherein recovery of ammonia produced in step (i) is performed in a gas stripper.

Fig. 1

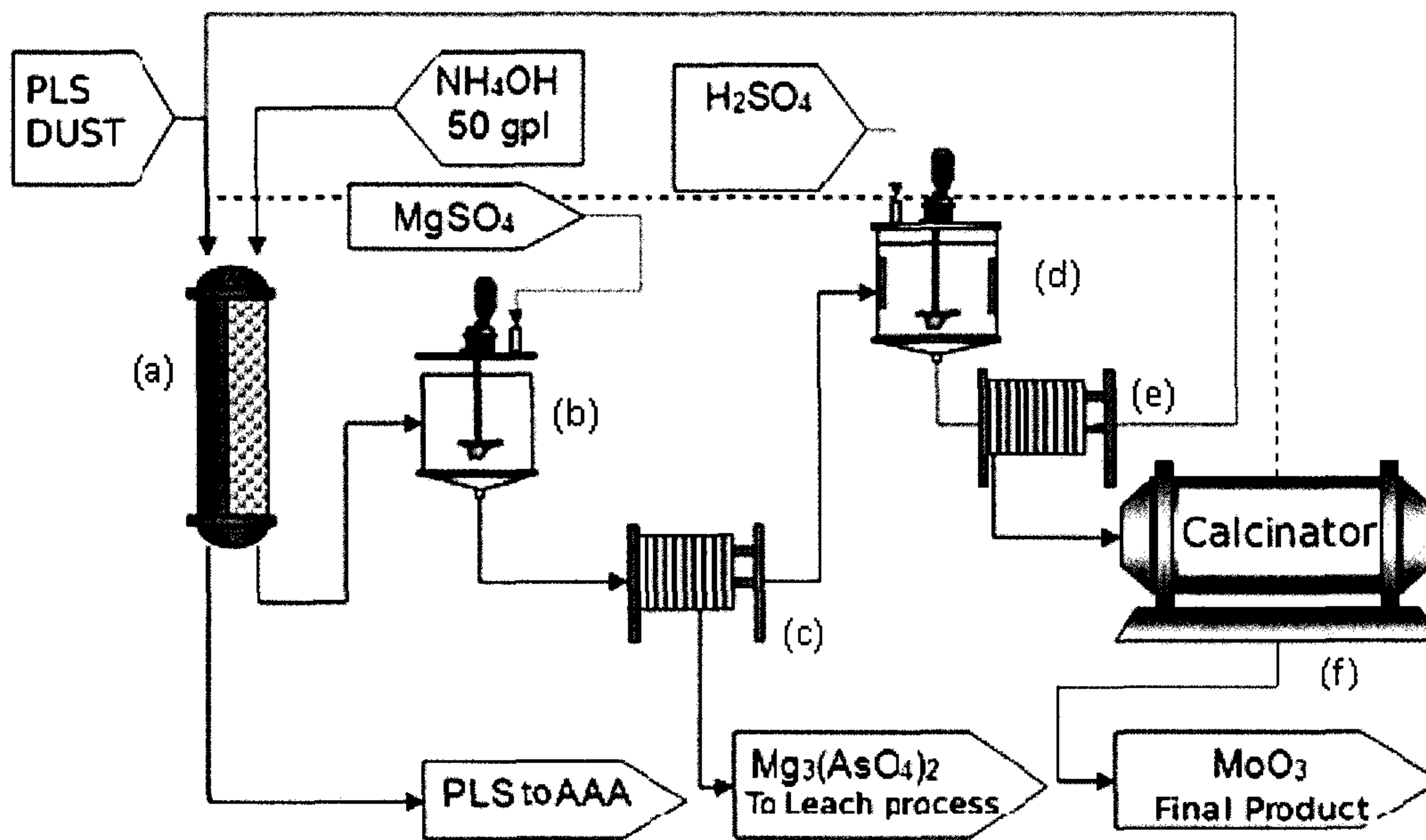


Fig. 2

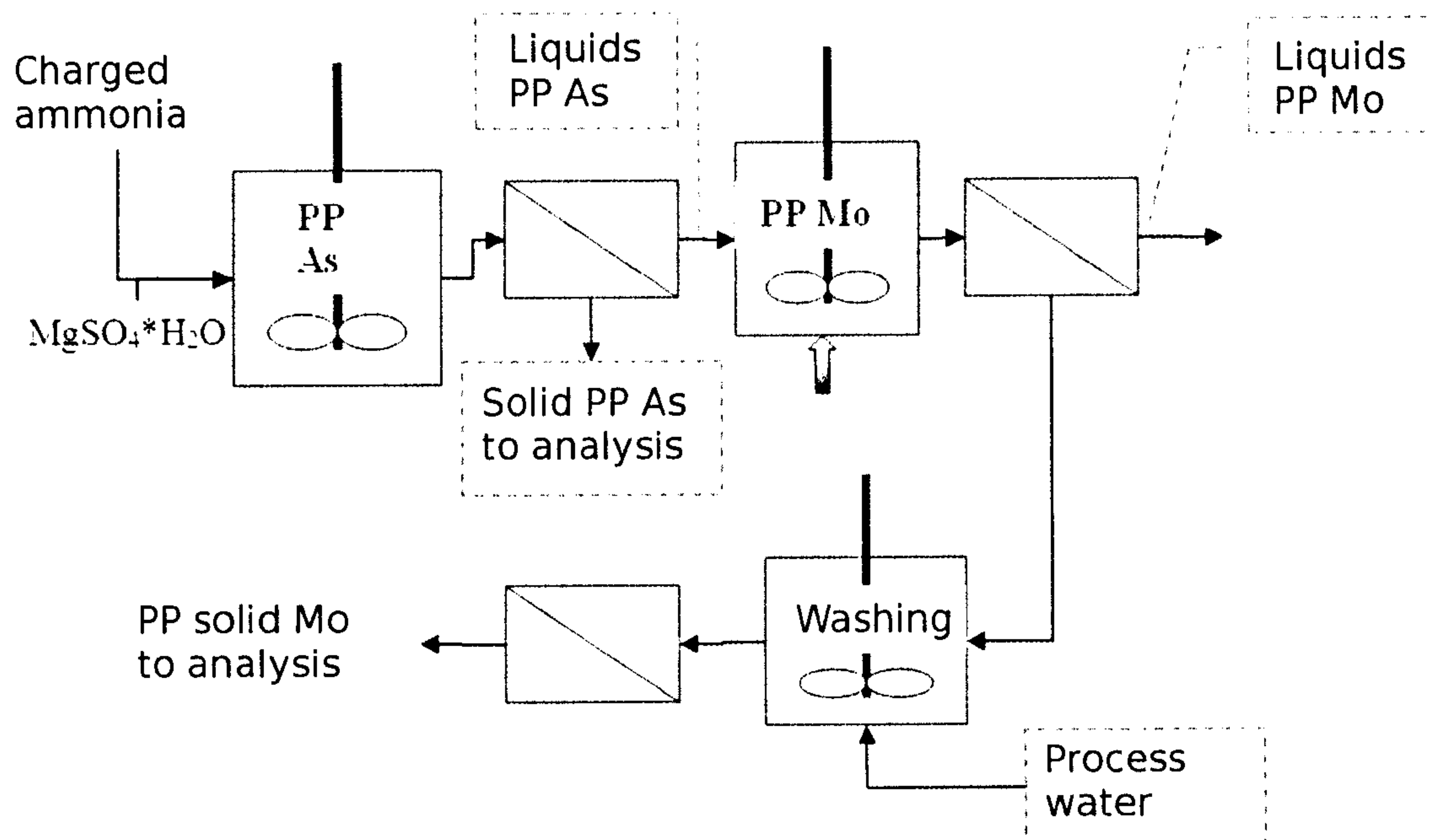


Fig. 3

