



(22) Date de dépôt/Filing Date: 2012/06/27  
 (41) Mise à la disp. pub./Open to Public Insp.: 2012/12/30  
 (62) Demande originale/Original Application: 2 781 238  
 (30) Priorité/Priority: 2011/06/30 (JP146565/2011)

(51) Cl.Int./Int.Cl. *C22B 3/42* (2006.01),  
*C22B 3/04* (2006.01), *C22B 3/44* (2006.01),  
*C25C 3/34* (2006.01), *C22B 59/00* (2006.01)

(71) Demandeur/Applicant:  
 KABUSHIKI KAISHA TOSHIBA, JP

(72) Inventeurs/Inventors:  
 MIZUGUCHI, KOJI, JP;  
 KANAMURA, SHOHEI, JP;  
 OSATO, TETSUO, JP;  
 TAKAHASHI, YUYA, JP;  
 YAITA, YUMI, JP;  
 YAMASHITA, YU, JP;

...

(74) Agent: FETHERSTONHAUGH & CO.

(54) Titre : PROCEDE DE PRODUCTION DE METAL RARE  
 (54) Title: PROCESS FOR PRODUCING RARE METAL

(57) **Abrégé/Abstract:**

According to one embodiment, a process for producing rare metals includes the steps of: recovering a first-residue solution through a primary target metal extracted by leaching a mineral resource; extracting a perrhenic acid ion contained in the first-residue solution with at least one of an anion exchange resin and a first-organic solvent; back extracting the perrhenic acid ion contained in the anion exchange resin or the first-organic solvent to a first eluant; and electrolyzing the back extracted first-eluant to collect a rhenium at a cathode.



(72) Inventeurs(suite)/Inventors(continued): FUJITA, REIKO, JP; OMORI, TAKASHI, JP; YAZAWA, TAKASHI, JP

27860-54D1

**ABSTRACT OF THE DISCLOSURE**

According to one embodiment, a process for producing rare metals includes the steps of: recovering a first-residue solution through a primary target metal extracted by leaching a mineral resource; extracting a perrhenic acid ion contained in the first-residue solution with at least one of an anion exchange resin and a first-organic solvent; back extracting the perrhenic acid ion contained in the anion exchange resin or the first-organic solvent to a first-eluant; and electrolyzing the back extracted first-eluant to collect a rhenium at a cathode.

27860-54D1

## PROCESS FOR PRODUCING RARE METAL

This is a first divisional application of Canadian Patent Application No. 2,781,238, filed June 27, 2012. It should be understood that the expression “the present invention” or the like used in this specification encompasses not only the subject matter of this  
5 first divisional application but that of the parent application and a second divisional also.

### FIELD

Embodiments described herein relate generally to a process for producing rare metal using a residue solution as raw materials, the residue solution obtained through primary target metal extracted by leaching a mineral resource.

### 10 BACKGROUND

Rhenium (Re) is a particularly rare metal among rare metals, and is used to reinforce turbine materials for aircrafts, for example.

Rare-earth metal (RE) is used as materials, such as a hydrogen storing metal alloy, rechargeable battery materials, optical glass, a powerful rare-earth permanent magnet, a  
15 fluorescent substance, and an abradant, for example.

There is a prior art disclosing that extracting rhenium metal and the rare-earth metal (neodymium, dysprosium) separately at a series of processes from the residue solution as raw material, the residue solution obtained through primary target metal extracted by leaching a mineral resource (for example, Japanese Unexamined Patent Application  
20 No. JP-A-2010-285680).

Unfortunately, the process in the prior art, if impurities such as Fe and Al are contained in the residue solution,

27860-54D1

prevent the rare-earth metals from their proper separate extraction.

The present invention was made in consideration of such a situation, introducing the step of removing the impurities in residue solution, and providing the process for producing rare metal having high robustness to solution composition.

5 In one aspect, the invention of the parent application relates to a process for producing a rare metal comprising the steps of: recovering a first-residue solution through a primary target metal extracted by leaching a mineral resource; extracting a perrhenic acid ion contained in the first-residue solution with at least one of an anion exchange resin and a first-organic solvent; back extracting the perrhenic acid ion contained in the anion exchange resin  
10 or the first-organic solvent to a first-eluant consisting of an ammonia aqueous solution; and electrolyzing the back extracted first-eluant to collect rhenium metal at a cathode.

In a further aspect, the invention of the parent application relates to the above process further comprising the steps of: recovering a second-residue solution of the perrhenic acid ion extracted from the first-residue solution; adjusting the pH of the second-residue  
15 solution within a range of pH 3 to pH 5 to generate a precipitate and then removing the precipitate; extracting a rare-earth metal ion with at least one of a cation exchange resin and a second-organic solvent from the second-residue solution from which the precipitate is removed; back extracting the rare-earth element ion contained in the cation exchange resin of the second-organic solvent to a second-eluant consisting of an ammonia aqueous solution;  
20 adding an oxalic acid in the back extracted second-eluant to precipitate a rare-earth metal oxalate; recovering the rare-earth metal oxalate and then converting the recovered rare-earth metal oxalate into a rare-earth metal oxide; and electrolyzing the rare-earth metal oxide in a molten salt to collect in a metallic state a rare-earth metal at a cathode. Suitably, either one of the cation exchange resin and the second-organic solvent has selectivity for various types of  
25 rare-earth metal ions so as to extract each separately. Suitably, the process further comprises the step of adjusting a valence of an impurity iron ion before the step of the adjusting pH of the second-residue solution within the range of pH 3 to pH 5.

27860-54D1

In a further aspect, the invention of the parent application relates to the above process further comprising the steps of: recovering a second-residue solution of the perrenic acid ion extracted from the first-residue solution; adjusting the pH of the second-residue solution within a range of pH 5 to pH 11 to generate a precipitate and then recovering the precipitate; adjusting the recovered precipitate in an aqueous solution within a range of pH 3 to pH 5 and then removing a residual-precipitate; adding an oxalic acid in the aqueous solution from which the residual-precipitate is removed to precipitate a rare-earth metal oxalate; recovering the rare-earth metal oxalate and then converting the recovered rare-earth metal oxalate into a rare-earth metal oxide; and electrolyzing the rare-earth metal oxide in a molten salt to collect in a metallic state a rare-earth metal at a cathode. Suitably, the process further comprises the step of washing the recovered precipitate in an aqueous solution adjusted to a pH of 11 or higher, before the step of adjusting the recovered precipitate in an aqueous solution within a range of pH 3 to pH 5. Suitably, the process further comprises the step of adjusting a valence of an impurity iron ion, before the step of the adjusting the second-residue solution within the range of pH 5 to pH 11. Suitably, the process further comprises the steps of extracting the rare-earth metal ions separately for each component with at least one of a cation exchange resin and a second-organic solvent having selectively for various types of rare-earth metal ions contained in the aqueous solution from which the residual-precipitate is removed; and back extracting the rare-earth metal ions contained in the cation exchange resin or the second-organic solvent to a second-eluant.

In one aspect, the invention of the first divisional application relates to a process for producing a rare metal comprising the steps of: recovering a residue solution through a primary target metal extracted by leaching a mineral resource; adjusting the pH of the residue solution within a range of pH 3 to pH 5 to generate a precipitate and then removing the precipitate; extracting a rare-earth metal ion with at least one of a cation exchange resin and an organic solvent from the residue solution from which the precipitate is removed; back extracting the rare-earth metal ion contained in the cation exchange resin or the organic solvent to an eluant consisting of an ammonia aqueous solution; adding oxalic acid in the back extracted eluant to precipitate a rare-earth metal oxalate; recovering the rare-earth metal oxalate and then converting the recovered rare-earth oxalate into a rare-earth metal

27860-54D1

oxide; and electrolyzing the rare-earth metal oxide in a molten salt to collect in a metallic state a rare-earth metal at a cathode. Suitably, either one of the cation exchange resin and the organic solvent has selectivity for various types of rare-earth metal ions to extract separately for each type of rare-earth metal ions. Suitably, the process further comprises the step of:

5 adjusting a valence of an impurity iron ion before the step of adjusting the pH of the residue solution within the range of pH 3 to pH 5.

#### BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a flow chart showing a first embodiment of the process for producing rare metal according to the present invention.

10 Fig. 2 (A) and Fig. 2 (B) are flow chart showing an extraction of various types of the rare-earth metal separately containing in the residue solution according to the first embodiment.

Fig. 3 is a flow chart having a step of adjusting a valence of an impurity Fe-ion in the residue solution according to the first embodiment.

15 Fig. 4 is a flow chart showing a second embodiment of the process for producing rare metal according to the present invention.

Fig. 5 (A) is a flow chart having a step of removing an impurity Al in the residue solution according to the second embodiment.

Fig. 5 (B) is a flow chart further having a step of removing an impurity Fe.

27860-54D1

Fig. 6 is a flow chart showing extraction of various types of the rare-earth metal separately containing in the residue solution according to the second embodiment.

#### DETAILED DESCRIPTION

(A first embodiment)

Hereafter, the embodiment of the present invention is described based on an accompanying drawing.

As shown in Fig. 1, a process for producing rare metals according to a first embodiment includes the steps of: recovering a first-residue solution through a primary target metal extracted by leaching a mineral resource (S11-S14); extracting a perrhenic acid ion ( $\text{ReO}_4^-$ ) contained in the first-residue solution with at least one of an anion exchange resin and a first-organic solvent (S15, S16); back extracting the perrhenic acid ion ( $\text{ReO}_4^-$ ) contained in the anion exchange resin or the first-organic solvent to a first-eluant (S17); and electrolyzing the back extracted first-eluant (S18) to collect a rhenium (Re) at a cathode (S19).

In the Step (S11) the mineral resource is subjected to preliminary treatment (crushing, concentrating, roasting), and then leached with an acid or alkaline solution (S12).

In the Step (S13) the primary target metal means uranium, copper, or molybdenum in this embodiment, but it is not limited to these.

27860-54D1

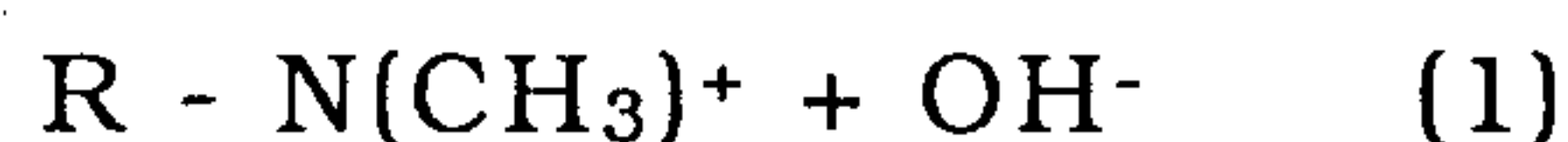
In the Step (S14) the first-residue solution contains the rare earth metal ion ( $RE^{3+}$ ), besides the perrhenic acid ion ( $ReO_4^-$ ) and further containing variety of impurity metal ion such as Fe, Al, Ca, and Mg.

In this embodiment, the rare-earth metal means the element located by the fourth to sixth period among the third group in the periodic table, such as Sc (scandium), Y (yttrium), La (lanthanum), Ce (cerium), Pr (praseodymium), Nd (neodymium), Pm (promethium), Sm (samarium), Eu (europium), Gd (gadolinium), Tb (terbium), Dy (dysprosium), Ho (holmium), Er (erbium), Tm (thulium), Yb (ytterbium), and Lu (lutetium).

These elements have the character which grows into a trivalent positive ion easily.

In the Step (S15) the anion exchange resin with which the perrhenic acid ion ( $ReO_4^-$ ) is extracted in solid phase. As shown in a following formula (1), the anion exchange resin has an ion-exchange group (fixed ion  $[-N(CH_3)^+]$  is an example) fixed to the body R, forming ionic bond with the exchangeable mobile ion currently (counter ion  $[OH^-]$  is an example).

Then if the anion exchange resin absorbs the negative ion ( $ReO_4^-$  in this case) contained in the first-residue solution, counter ion ( $OH^-$  in this case) will be emitted instead to the first-residue solution.



In the Step (S16) the first-organic solvent with which the perrhenic acid ion ( $ReO_4^-$ ) is extracted by distribution ratio.

27860-54D1

The first-organic solvent and the first-residue solution do not dissolve each other that two-phase separation is carried out. Furthermore the solubility of perrhenic acid ion ( $\text{ReO}_4^-$ ) differs between the first-organic solvent and the first-residue solution, respectively. For this reason, if the boundary motion of the perrhenic acid ion ( $\text{ReO}_4^-$ ) balanced in an equilibrium state, the perrhenic acid ion ( $\text{ReO}_4^-$ ) will be distributed to the first-organic solvent and the first-residue solution at a fixed rate.

By using the first-organic solvent with a large distribution coefficient, perrhenic acid ion ( $\text{ReO}_4^-$ ) is efficiently extractable (concentrate) from the first-residue solution.

One case only either step may be carried out among the step (S15) solid phase extraction of  $\text{ReO}_4^-$  with the anion exchange resin or the step (S16) distributed extraction of  $\text{ReO}_4^-$  with the first-organic solvent and other case both steps may be carried out continuously to promote condensing. Generally, the extraction with ion exchange resin is effective when condensing the low-concentration ion in the first-residue solution, and the extraction with an organic solvent is effective if the ion concentration is higher than the ion exchange resin's case.

In the Step (S17) back extraction to the first-eluant, the perrhenic acid ion ( $\text{ReO}_4^-$ ) contained in the anion exchange resin or the first-organic solvent distributes to the first-eluant.

Therefore, the material of the first-eluant and the method of the back extraction are different whether which step are

27860-54D1

carried out among the step (S15) solid phase extraction of  $\text{ReO}_4^-$  with the anion exchange resin or the step (S16) distributed extraction of  $\text{ReO}_4^-$  with the first-organic solvent. In case both steps (S15) (S16) are carried out continuously, the step (S17) back extraction to the first-eluant exists between (S15) and (S16), although illustration is omitted.

In the Step (S18) the electrolysis vessel holds the first-eluant containing the condensed perrhenic acid ion ( $\text{ReO}_4^-$ ) to adjust electrolytic concentration and then the electrodes inserted to impress direct-current power. If the halogen gas may generate at the anode in this case, the halogen gas generation can be controlled by adopting a DSE (Dimensionally Stable Electrodes).

The process for producing rare metals, after the steps of (S11) - (S16), further includes the step of: recovering a second-residue solution the perrhenic acid ion ( $\text{ReO}_4^-$ ) extracted from the first-residue solution (S20); adjusting a potential-hydrogen of the second-residue solution within a range of pH 3 or higher and lower than pH 5 to generate a precipitate (S21) and then removing the precipitate ( $\text{Fe}(\text{OH})_x$ ) (S22); extracting a rare-earth metal ion ( $\text{RE}^{3+}$ ) with at least one of a cation exchange resin and a second-organic solvent from the second-residue solution in which the precipitate ( $\text{Fe}(\text{OH})_x$ ) removed (S23, S24); back extracting the rare-earth metal ion ( $\text{RE}^{3+}$ ) contained in the cation exchange resin or the second-organic solvent to a second-eluant (S25); adding an oxalic acid ( $(\text{COOH})_2$ ) in the

27860-54D1

back extracted second-eluant (S26) to precipitate a rare-earth metal oxalate ( $\text{RE}_2(\text{C}_2\text{O}_4)_3$ ); recovering the rare-earth metal oxalate ( $\text{RE}_2(\text{C}_2\text{O}_4)_3$ ) (S27) and then converting into a rare-earth metal oxide ( $\text{RE}_2\text{O}_3$ ) (S28); and electrolyzing the rare-earth metal oxide ( $\text{RE}_2\text{O}_3$ ) in a molten salt (S29) to collect a rare-earth metal at a cathode (S30).

In addition, it is possible processing the first-residue solution directly in the steps of (S21) - (S30), omitting the steps (S15) - (S20) among the steps (S11) - (S20) mentioned above.

In the Step (S21) potential-hydrogen adjustment of the second-residue solution (or the first-residue solution) within a range of pH 3 or higher and lower than pH 5 by an alkali (ammonia aqueous solution etc.) supplied. The preferable potential-hydrogen range is within pH 3.5 to pH 4. If the potential-hydrogen of the second-residue solution less than pH 3 causes insufficient precipitation of impurity Fe ion for remove, and pH 5 or higher causes precipitation of the rare-earth metal ion ( $\text{RE}^{3+}$ ) for collection.

In the Step (S23) the cation exchange resin with which the rare-earth metal ion ( $\text{RE}^{3+}$ ) is extracted in solid phase. As shown in a following formula (2), the cation exchange resin has an ion-exchange group (fixed ion [ $-\text{SO}_3^-$ ] is an example) fixed to the body R, forming ionic bond with the exchangeable mobile ion currently (counter ion [ $\text{H}^+$ ] is an example).

27860-54D1

Then if the cation exchange resin absorbs the positive ion ( $\text{RE}^{3+}$  in this case) contained in the second-residue solution, counter ion ( $\text{H}^+$  in this case) will be emitted instead to the second-residue solution.



In the Step (S24) the second-organic solvent with which the rare-earth metal ion ( $\text{RE}^{3+}$ ) is extracted by distribution ratio. The second-organic solvent (or first-organic solvent) and the second-residue solution do not dissolve each other that two-phase separation is carried out. Furthermore the solubility of rare-earth metal ion ( $\text{RE}^{3+}$ ) differs between the second-organic solvent and the second-residue solution, respectively. For this reason, if the boundary motion of the rare-earth metal ion ( $\text{RE}^{3+}$ ) balanced in an equilibrium state, the rare-earth metal ion ( $\text{RE}^{3+}$ ) will be distributed to the second-organic solvent and the second-residue solution at a fixed rate.

By using the second-organic solvent with a large distribution coefficient, rare-earth metal ion ( $\text{RE}^{3+}$ ) is efficiently extractable (concentrate) from the second-residue solution.

One case only either step may be carried out among the step (S23) solid phase extraction of  $\text{RE}^{3+}$  with the cation exchange resin or the step (S24) distributed extraction of  $\text{RE}^{3+}$  with the second-organic solvent and other case both steps may be carried out continuously to promote condensing.

27860-54D1

In the Step (S25) the second-eluant carries out back extraction, the rare-earth metal ion ( $RE^{3+}$ ) contained in the cation exchange resin or the second-organic solvent distributes to the second-eluant.

Therefore, the material of the second-eluant and the method of the back extraction are different whether which step are carried out among the step (S23) solid phase extraction of  $RE^{3+}$  with the cation exchange resin and the step (S24) distributed extraction of  $RE^{3+}$  with the second-organic solvent. In case both steps (S23) (S24) are carried out continuously, the step (S25) back extraction to the second-eluant exists between (S23) and (S24), although illustration is omitted.

In the Step (S26) (S27) oxalic acid ( $(COOH)_2$ ) is added to the second-eluant in which rare-earth metal ion ( $RE^{3+}$ ) is contained, rare-earth metal oxalate ( $RE_2(C_2O_4)_3$ ) will precipitate. These precipitated rare-earth metal oxalate ( $RE_2(C_2O_4)_3$ ) is recovered by filtration.

In the Step (S28) the recovered rare-earth metal oxalate ( $RE_2(C_2O_4)_3$ ) converts into a rare-earth metal oxide ( $RE_2O_3$ ) by drying and baking.

In the Step (S29) (S30) together with salt the converted rare-earth metal oxide ( $RE_2O_3$ ) is carried out molten salt electrolysis to collect the rare-earth metal (RE) at cathode.

As such the salt used for molten salt electrolysis, it is the combination of halogenide such as chloride, fluoride, iodide of

27860-54D1

alkaline metals such as Li, Na, K, Cs, Rb and of alkaline-earth metals such as Ca, Mg, Be, Sr, Ba, Ra.

At this time, generating of the halogen gas at the anode is controlled by mixing the oxide of alkaline metals such as Li, Na, K, Cs, Rb and of alkaline-earth metals such as Ca, Mg, Be, Sr, Ba, Ra.

Fig. 2 shows the first embodiment of the process for producing rare metal wherein the residue solution contains various types of the rare-earth metal ion ( $RE^+$ ).

That is, in the back process of the step (S22) removal of iron-based precipitate ( $Fe(OH)_x$ ), the steps (S23A, S24B) either one of the cation exchange resin and the second-organic solvent having selectivity for various types of the rare-earth metal ion ( $RE^{3+}$ ) to extract separately for each component.

In the step (S23A) of Fig. 2 (A), various types of the rare-earth metal ion ( $RE^{3+}$ ) are separated for each component using different cation exchange resin which has ion selectivity. In the Step (S25) each rare-earth metal ion ( $RE^{3+}$ ) contained in different cation exchange resin respectively extracted to the second-eluant separately. Furthermore carrying out the step (S24) distributed extraction with second-organic solvent, it is possible to condense each rare-earth metal ion ( $RE^{3+}$ ).

The subsequent steps (S26) - (S30) are carried out on each second-eluant back extracted respectively.

In the step (S24B) of Fig. 2 (B), various types of the rare-earth metal ion ( $RE^{3+}$ ) are separated for each component using

27860-54D1

different second-organic solvent which has ion selectivity. In the Step (S25) each rare-earth metal ion ( $RE^{3+}$ ) contained in different second-organic solvent respectively extracted to the second-eluant separately. Furthermore, the step (S24) may be carried out in advance, for condensing rare-earth metal ions ( $RE^{3+}$ ) all together.

The subsequent steps (S26) - (S30) are carried out on each second-eluant back extracted respectively.

Fig.3 shows the first embodiment added the step (S40) valence adjustment of an impurity iron ion, before the step (S21) potential-hydrogen adjustment of the second-residue solution within the range of pH 3 or higher and lower than pH 5.

Specifically, in the step (S40) babbling the second-residue solution by oxidizers, such as air and hydrogen peroxide solution to adjust the valence of the iron ion changes into  $Fe^{3+}$  from  $Fe^{2+}$ .

(A second embodiment)

With reference to a flow chart in Fig. 4 a second embodiment of process for producing rare metals will be described. Steps S11 to S19 in the second embodiment are the same as those in the first embodiment, and description thereof will be omitted by citation of the description already given. Also in the step of (S20) or subsequent steps, described in Fig. 4, same reference numerals will be given to steps common to those described in Fig. 1, and description

27860-54D1

thereof will be omitted by citation of the above-mentioned description.

The process for producing rare metals according to second embodiment, after through the steps (S11)-(S16), further includes steps: recovering a second-residue solution the perrhenic acid ion ( $\text{ReO}_4^-$ ) extracted from the first-residue solution (S20); adjusting potential-hydrogen of the second-residue solution within a range of pH 5 or higher and lower than pH 11 to generate a precipitate (S41), and then recovering the precipitate ( $\text{RE}(\text{OH})_x$ ,  $\text{Fe}(\text{OH})_x$ ) (S42); adjusting the recovered precipitate ( $\text{RE}(\text{OH})_x$ ,  $\text{Fe}(\text{OH})_x$ ) in an aqueous solution within a range of pH 3 or higher and lower than pH 5 (S21) and then removing a residual-precipitate ( $\text{Fe}(\text{OH})_x$ ) (S22); adding an oxalic acid ( $(\text{COOH})_2$ ) in the aqueous solution ( $\text{RE}^{3+}$ ) the residual-precipitate ( $\text{Fe}(\text{OH})_x$ ) removed (S26) to precipitate a rare-earth metal oxalate ( $\text{RE}_2(\text{C}_2\text{O}_4)_3$ ); recovering the rare-earth metal oxalate ( $\text{RE}_2(\text{C}_2\text{O}_4)_3$ ) (S27) and then converting into a rare-earth metal oxide ( $\text{RE}_2\text{O}_3$ ) (S28); and electrolyzing the rare-earth metal oxide ( $\text{RE}_2\text{O}_3$ ) in a molten salt (S29) to collect a rare-earth metal (RE) at a cathode (S30).

Above mentioned the steps of (S15) - (S20) can be omitted among the steps of (S11) - (S20), and the first-residue solution can be direct processing at the steps of (S41) (S42) (S21) - (S30).

In the step (S41) potential-hydrogen adjustment of the second-residue solution (or the first-residue solution) within a

27860-54D1

range of pH 5 or higher and lower than pH 11 by alkali supplied. The preferable potential-hydrogen range is within pH 6 to pH 8. If the potential-hydrogen of the second-residue solution is the range of lower than pH 5 or pH 11 or higher causes insufficient precipitation of the rare-earth metal ion ( $\text{RE}^{3+}$ ).

In the step (S42) recovery of precipitates ( $\text{RE}(\text{OH})_x$ ,  $\text{Fe}(\text{OH})_x$ ), the precipitates contains  $\text{Fe}(\text{OH})_x$  as an impurity besides  $\text{RE}(\text{OH})_x$  as a target for recovery. Other impurities of Ca ion and Mg ion are remain in the liquid phase, and then removed.

The subsequent steps (S21) - (S30), removing Fe and then extracting a rare-earth metal (RE).

Fig. 5 shows the process for producing rare metal having a step of removing aluminum of impurities.

It is assumed where aluminum of impurities is mixed in the recovered precipitate ( $\text{RE}(\text{OH})_x$ ,  $\text{Fe}(\text{OH})_x$ ) at the step (S42).

In Fig. 5 (A), the process for producing rare metals, before the step of (S21), further includes the steps of: washing the recovered precipitate ( $\text{RE}(\text{OH})_x$ ,  $\text{Fe}(\text{OH})_x$ ) in an aqueous solution adjusted a potential-hydrogen pH 11 or higher (S43), to remove an eluted aluminum (S44).

In Fig. 5 (B), the process for producing rare metal, further includes the steps of: adjusting a valence of an impurity iron ion (S40), before the step of (S41) adjusting potential-hydrogen

27860-54D1

of the second-residue solution within the range of pH 5 or higher and lower than pH 11.

In the step (S43), although the potential-hydrogen adjusted pH 11 or higher, it is more prefer the potential-hydrogen adjusted pH14 or higher. If the potential-hydrogen adjusted lower than pH11, it may become insufficient for dissolving and removing of aluminum of the impurities contained in the recovered precipitate.

Fig. 6 shows the process for producing rare metals according to the second embodiment, before the step of the adding an oxalic acid (S26); further comprising the step of: extracting the rare-earth metal ion ( $RE^{3+}$ ) separately for each component with at least one of a cation exchange resin and a second-organic solvent having selectivity for various types of the rare-earth metal ion ( $RE^{3+}$ ) contained in the aqueous solution in which the residual-precipitate ( $Fe(OH)_x$ ) removed (S23A, S23B); and back extracting the rare-earth metal ion ( $RE^{3+}$ ) contained in the cation exchange resin or the second-organic solvent to the second-eluant (S25).

In the step (S23A), various types of the rare-earth metal ion ( $RE^{3+}$ ) dissolved in the solution are separated for each component using different cation exchange resin which has ion selectivity. In the Step (S25) each rare-earth metal ion ( $RE^{3+}$ ) contained in different cation exchange resin respectively, extracted to the second-eluant separately. Furthermore carrying out the step (S24) distributed extraction with second-

27860-54D1

organic solvent, it is possible to condense each rare-earth metal ion ( $\text{RE}^{3+}$ ).

In the step (S24B), various types of the rare-earth metal ion ( $\text{RE}^{3+}$ ) dissolved in the solution are separated for each component using different second-organic solvent which has ion selectivity. In the Step (S25) each rare-earth metal ion ( $\text{RE}^{3+}$ ) contained in different second-organic solvent respectively, extracted to the second-eluant separately. Furthermore, the step (S24) may be carried out in advance, for condensing rare-earth metal ions ( $\text{RE}^{3+}$ ) all together.

The subsequent steps (S26) - (S30) are carried out on each back extracted second-eluant respectively.

While certain embodiments have been described, these embodiments have been presented by way of example only, and are not intended to limit the scope of the inventions. Indeed, the novel process and system described herein may be embodied in a variety of other forms; furthermore, various omissions, substitutions and changes in the form of the methods and systems described herein may be made without departing from the claimed scope of the inventions.

27860-54D1

CLAIMS:

1. A process for producing a rare metal comprising the steps of:  
recovering a residue solution through a primary target metal extracted by leaching a mineral resource;  
5 adjusting the pH of the residue solution within a range of pH 3 to pH 5 to generate a precipitate and then removing the precipitate;  
extracting a rare-earth metal ion with at least one of a cation exchange resin and an organic solvent from the residue solution from which the precipitate is removed;  
back extracting the rare-earth metal ion contained in the cation exchange resin  
10 or the organic solvent to an eluant consisting of an ammonia aqueous solution;  
adding oxalic acid in the back extracted eluant to precipitate a rare-earth metal oxalate;  
recovering the rare-earth metal oxalate and then converting the recovered rare-earth oxalate into a rare-earth metal oxide; and  
15 electrolyzing the rare-earth metal oxide in a molten salt to collect in a metallic state a rare-earth metal at a cathode.
2. The process according to claim 1, wherein either one of the cation exchange resin and the organic solvent has selectivity for various types of rare-earth metal ions to extract separately for each type of rare-earth metal ions.
- 20 3. The process according to claim 1, further comprising the step of:  
adjusting a valence of an impurity iron ion before the step of adjusting the pH of the residue solution within the range of pH 3 to pH 5.

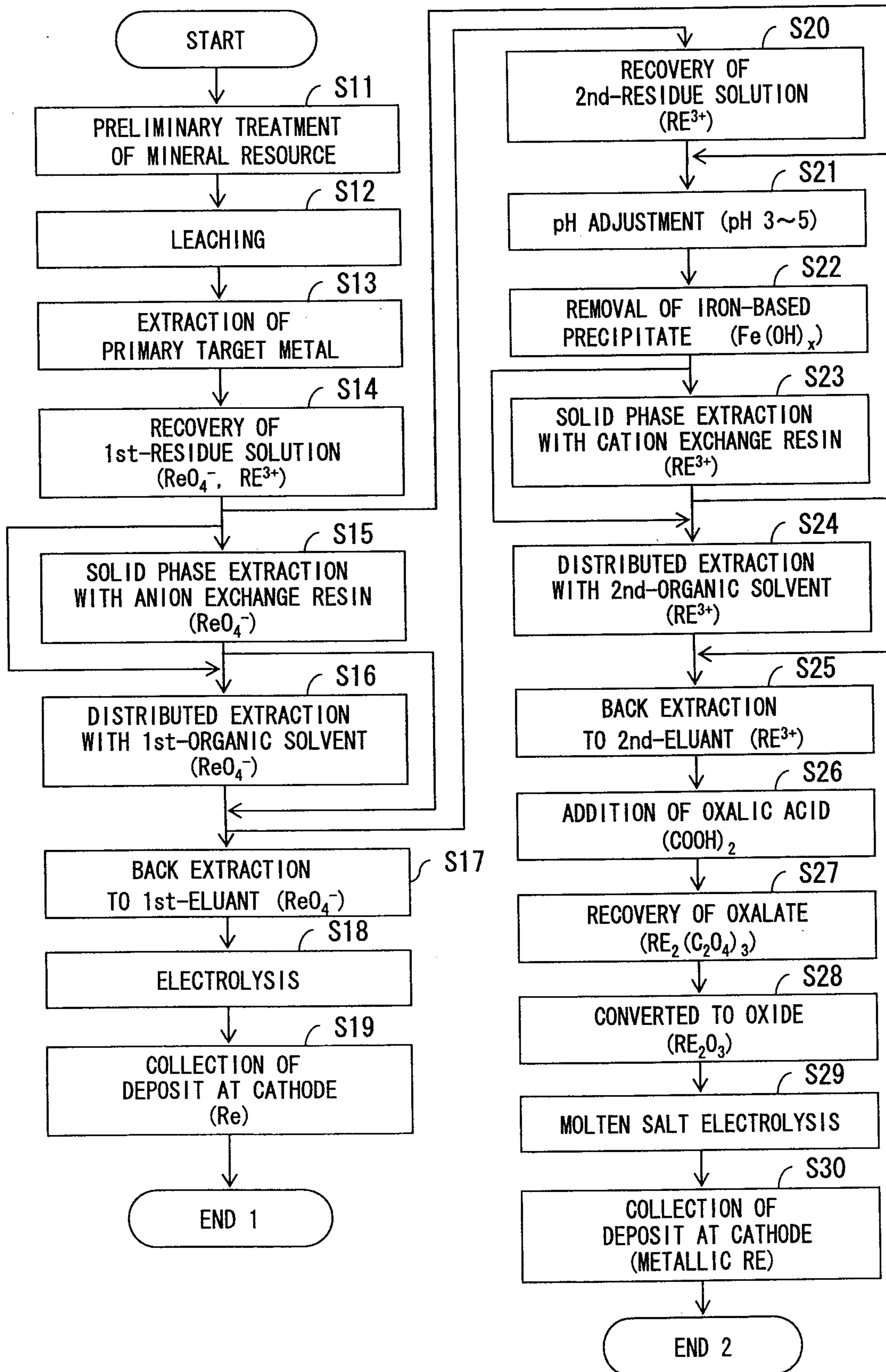


FIG. 1

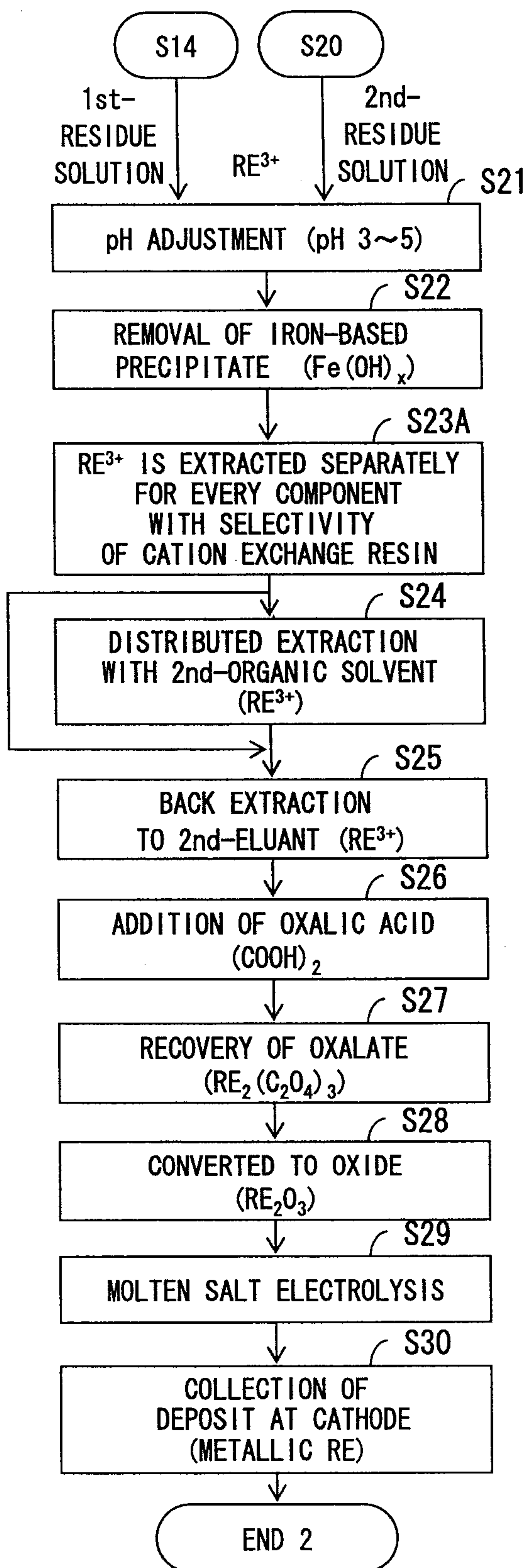


FIG. 2A

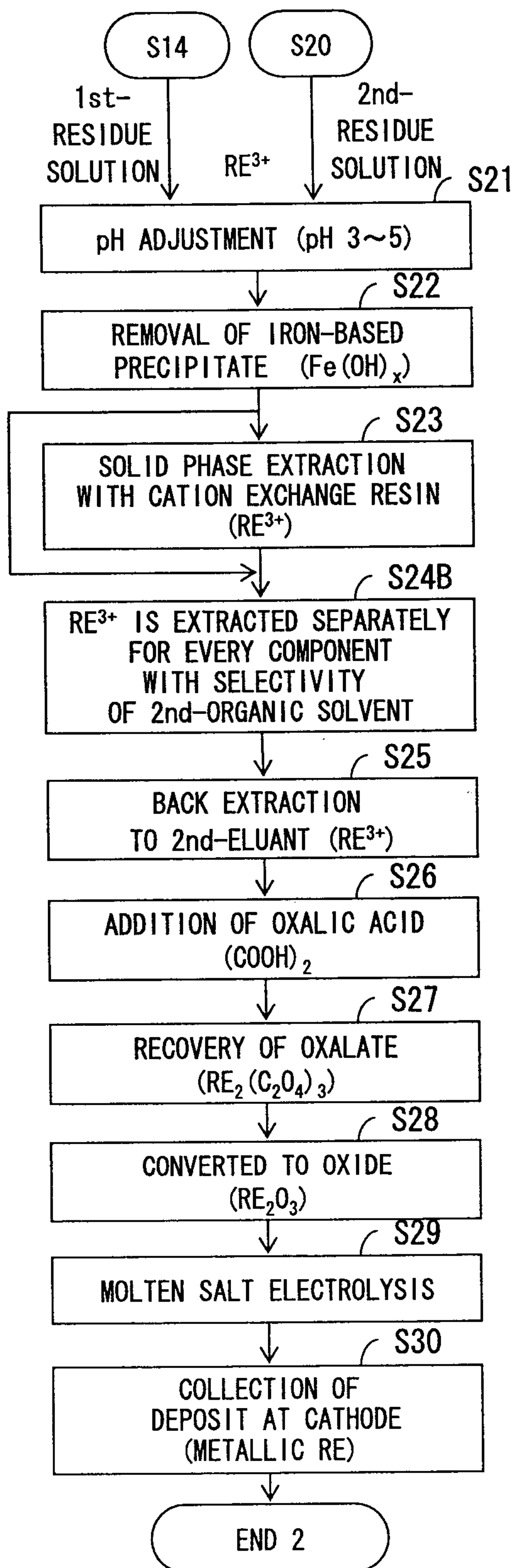


FIG. 2B

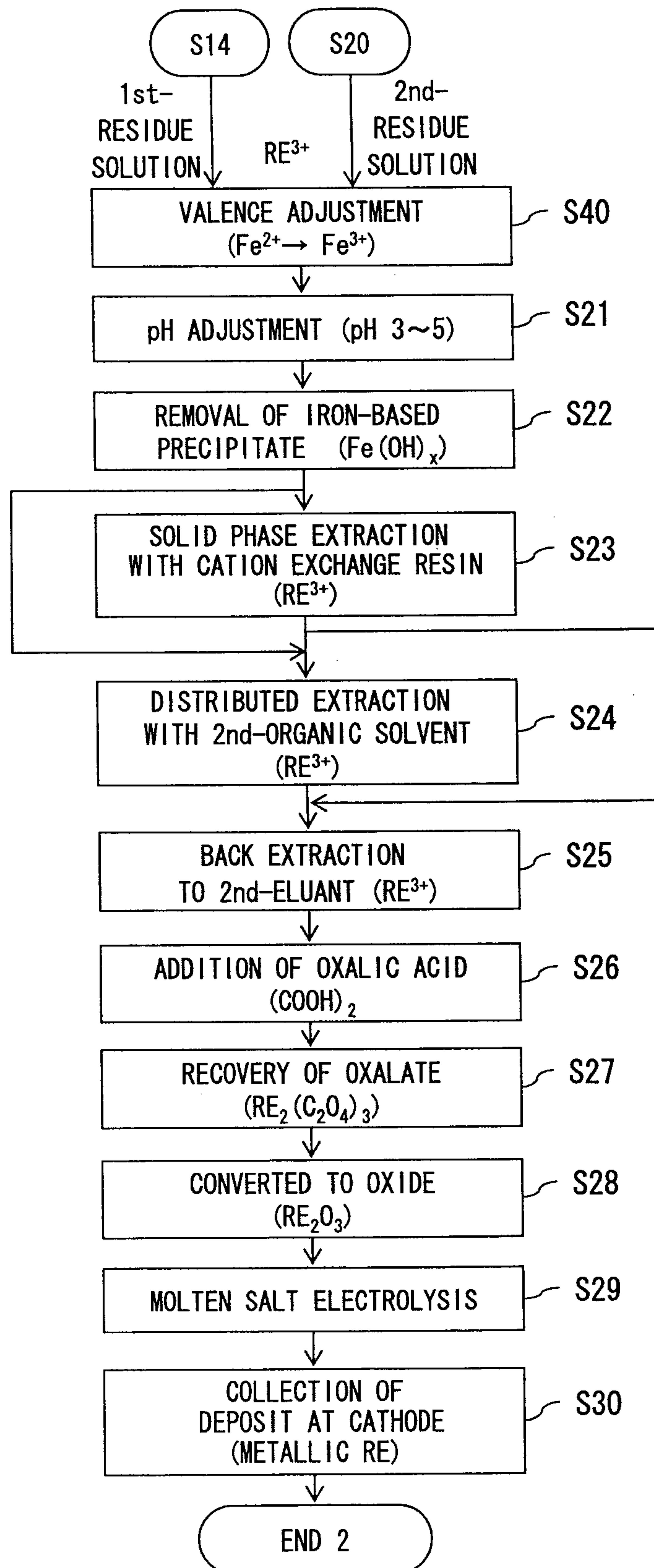


FIG. 3

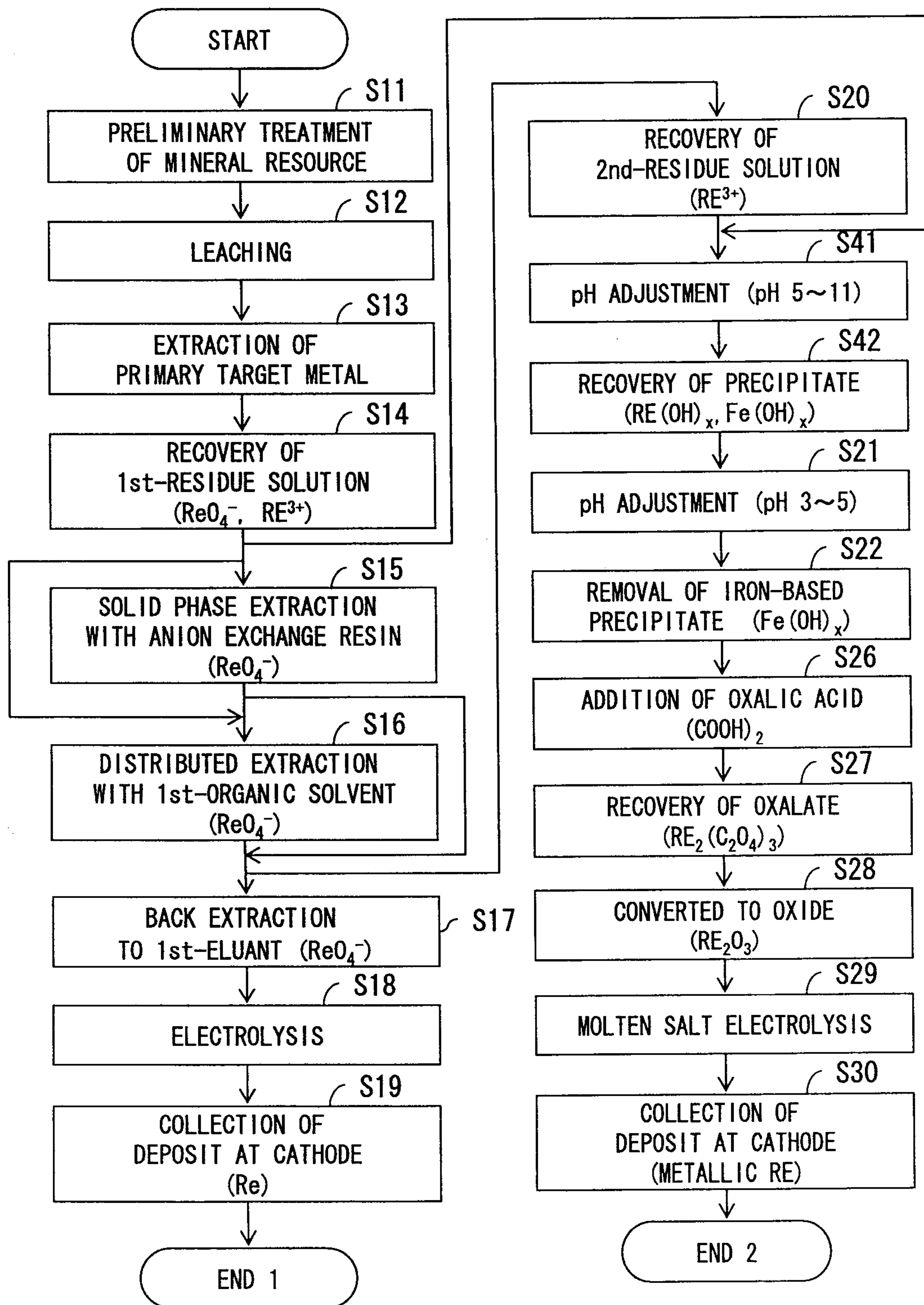


FIG. 4

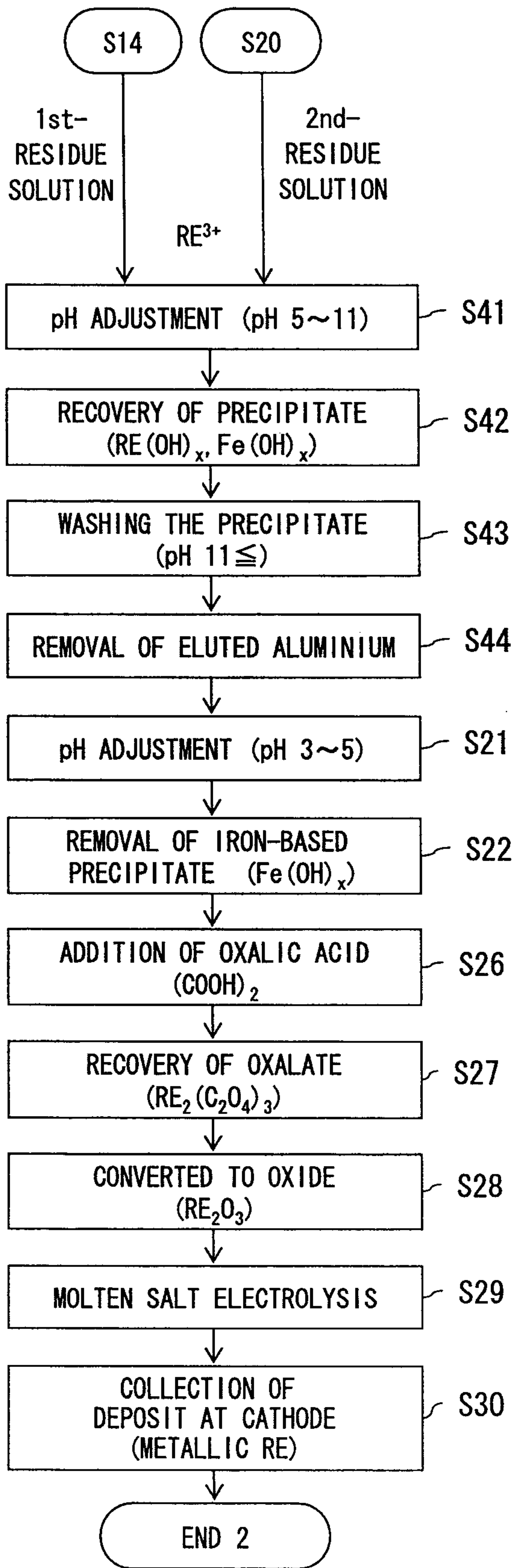


FIG. 5A

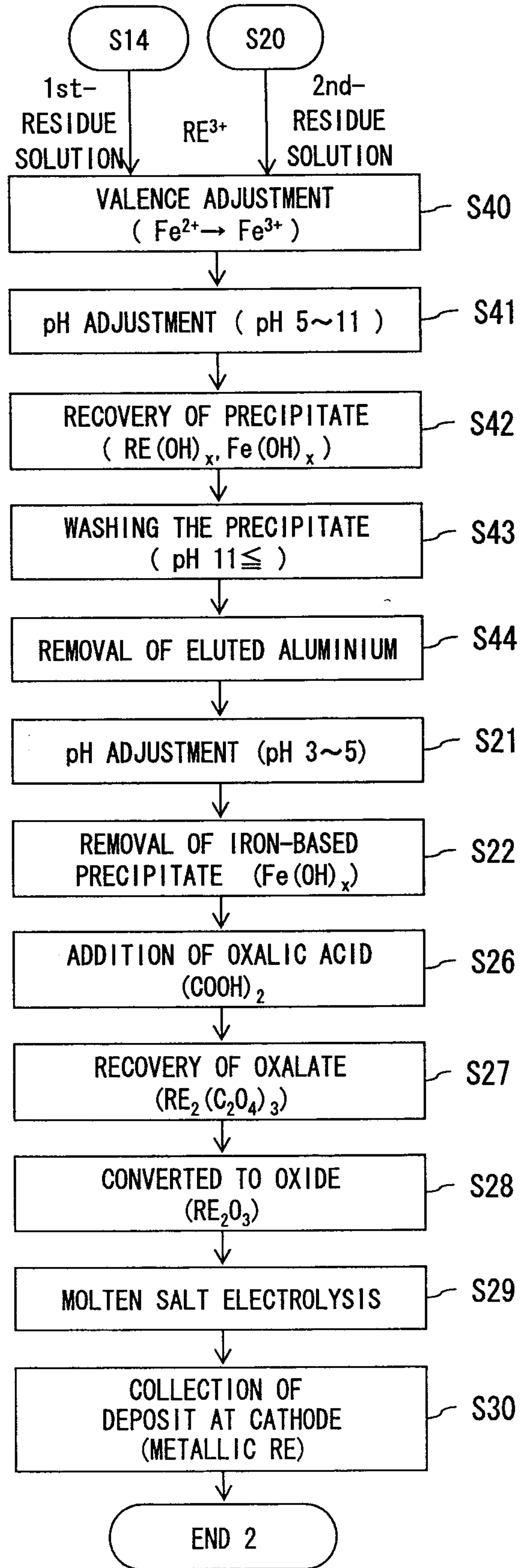


FIG. 5B

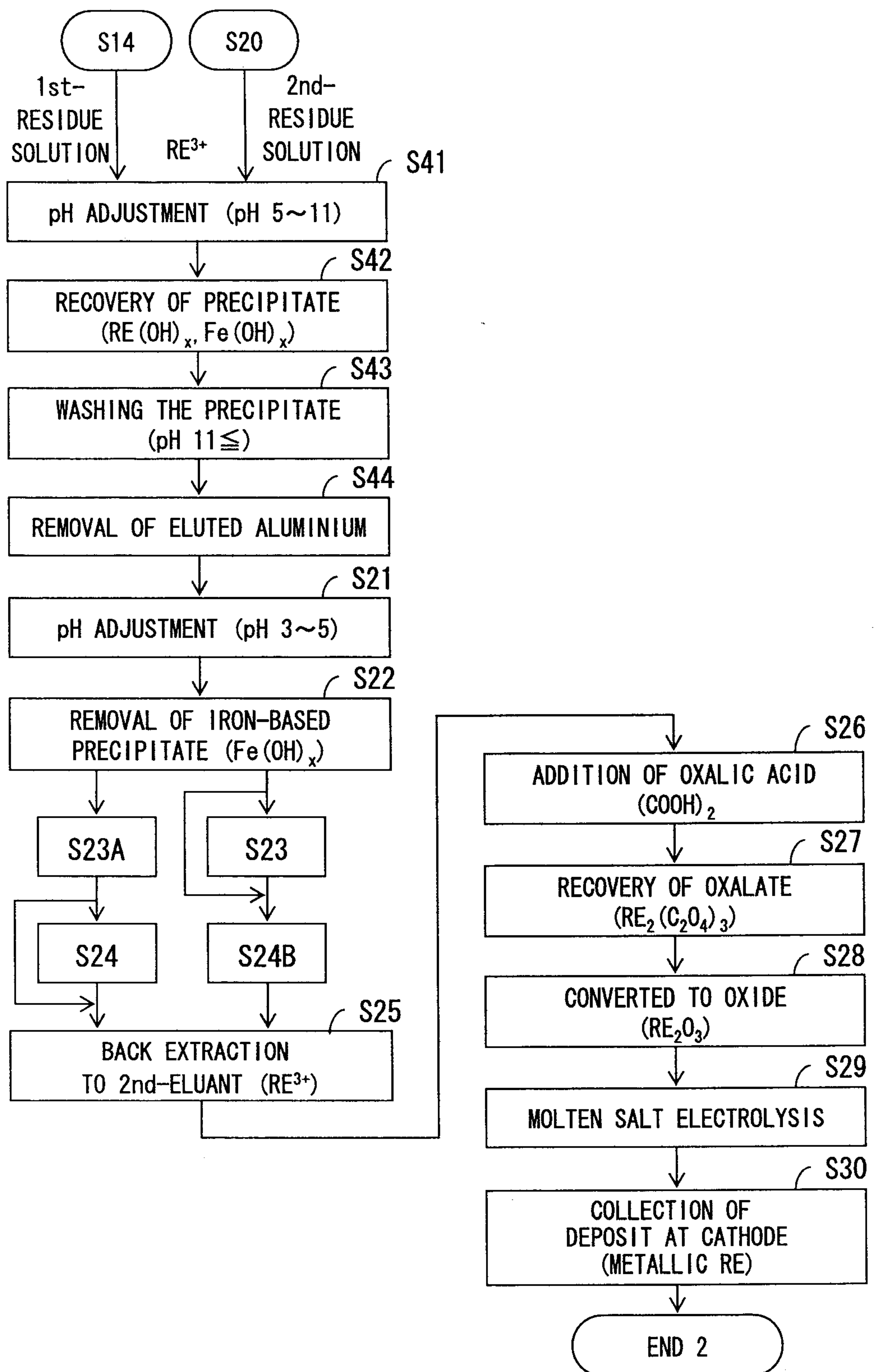


FIG. 6