

(19)



(11)

**EP 3 406 762 B1**

(12)

**EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention of the grant of the patent:  
**17.06.2020 Bulletin 2020/25**

(51) Int Cl.:  
**C23F 11/14** <sup>(2006.01)</sup>      **C10G 21/16** <sup>(2006.01)</sup>  
**C10G 21/20** <sup>(2006.01)</sup>      **C10G 75/02** <sup>(2006.01)</sup>

(21) Application number: **16895498.0**

(86) International application number:  
**PCT/JP2016/082311**

(22) Date of filing: **31.10.2016**

(87) International publication number:  
**WO 2017/163475 (28.09.2017 Gazette 2017/39)**

(54) **METHOD FOR REDUCING CORROSIVE IONS IN AROMATIC COMPOUND EXTRACTION SOLVENT**

VERFAHREN ZUR REDUZIERUNG VON KORROSIVEN IONEN IN EINEM LÖSUNGSMITTEL ZUR EXTRAKTION VON AROMATISCHEN VERBINDUNGEN

PROCÉDÉ DE RÉDUCTION D'IONS CORROSIFS DANS UN SOLVANT D'EXTRACTION DE COMPOSÉ AROMATIQUE

(84) Designated Contracting States:  
**AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR**

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(43) Date of publication of application:  
**28.11.2018 Bulletin 2018/48**

(56) References cited:  
**GB-A- 1 134 582**      **JP-A- H07 180 073**  
**JP-A- H08 225 488**      **JP-A- 2001 199 978**  
**JP-A- 2004 211 195**      **JP-A- 2009 078 197**  
**JP-A- 2009 078 198**      **US-A- 3 466 345**  
**US-A- 4 191 615**      **US-A1- 2006 043 340**

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**Description**

## Technical Field

5 **[0001]** The present invention relates to a method for reducing corrosive ions such as chloride ions, sulfate ions, and sulfite ions in an aromatic compound extraction solvent, the method being capable of preventing corrosion in a system where a solvent for extracting aromatic compounds such as benzene, toluene, and xylene from a hydrocarbon stream circulates in petroleum refineries, coal chemical plants, and the like.

## 10 Background Art

**[0002]** In petroleum refining and in hydrocarbon refining at coal chemistry plants, there is a perennial problem in that corrosive ions such as chloride ions, sulfate ions, and sulfite ions originating from various salts present in raw materials cause equipment corrosion.

15 **[0003]** In this regard, a technique of adding an alkali to scavenge corrosive ions is a possible solution. However, salts produced by adding an alkali can adhere/precipitate and possibly cause serious troubles such as deposit buildup and clogging in equipment.

**[0004]** Patent Literature 1 discloses a method for preventing corrosion caused by chloride in a condensation system of an oil refining process by using a plurality of amines such as monoethanolamine in combination. Patent Document 2  
20 discloses that metal corrosion is prevented by introducing a quaternary ammonium compound such as choline hydroxide (2-hydroxyethyltrimethylammonium hydroxide) into a fluid that may come into contact with the inside of petroleum refining equipment and the like to prevent generation of hydrogen chloride.

**[0005]** US 3 466 345 A is directed at a method for solvent extracting aromatic hydrocarbons wherein the preferred solvent comprises sulfolane and the typical aromatic hydrocarbons to be recovered include benzene, toluene, and xylene.

25 **[0006]** US 4 191 615 A describes that in order to prevent corrosion of the apparatus in extraction and extractive distillation processes where N-substituted morpholine is employed as the selective solvent, an additive is added to the solvent which additive consists of (a) phosphoric acid, (b) a salt thereof, (c) a vanadium compound, (d) a molybdenum compound or (e) a mixture of two or more of these compounds, the addition being effected in an amount of about 0.005 to 0.02% by weight of the morpholine solvent.

30 **[0007]** G 1 134 582 describes that sulfolane-type solvents containing corrosive contaminants are purified by contact with an aqueous solution containing an alkaline compound and with an alkali-resistant liquid, which is sparingly soluble in water and which dissolves the sulfolane-type solvent more readily than water, separating a solvent phase containing alkali-resistant liquid from the aqueous phase and subsequently recovering the sulfolane-type solvent from the solvent phase.

35 **[0008]** US 2006/043340 A1 describes that a quaternary ammonium compound is used as a metal corrosion inhibitor.

## Citation List

## Patent Literature

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**[0009]**

PTL1: JP 7-180073 A

PTL2: JP 2004-211195 A

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## Summary of Invention

## Technical Problem

50 **[0010]** However, with conventional methods, although chloride ions derived from salts contained in petroleum and the like are reduced, it is difficult to completely remove them. Therefore, it is not uncommon that chlorides, chloride ions, sulfate ions, and sulfite ions are present in hydrocarbon mixtures such as naphtha obtained by refining petroleum and the like, and there is still the problem of corrosion by corrosive ions in a system where such a hydrocarbon mixture is used as a raw material.

55 **[0011]** Meanwhile, a catalyst is used in a method for obtaining an aromatic compound such as benzene, toluene, or xylene by reforming a hydrocarbon mixture such as naphtha. The catalyst is either used continuously or continuously recovered from the reaction product composition and regenerated, but the catalytic activity deteriorates due to long-term use, and therefore a catalyst regeneration treatment is carried out. Chlorine compounds are often used in the catalyst

regeneration treatment, and thus the reformed hydrocarbon fed from the aromatic compound generator to the extractor may contain not only chloride ions derived from the raw-material hydrocarbon mixture but also chloride ions derived from the chlorine compound used in the catalyst regeneration.

5 **[0012]** Moreover, an aromatic hydrocarbon oil that is a reformed hydrocarbon and the like are subjected to an aromatic compound extraction treatment involving an extraction solvent. In this extraction treatment, the aromatic hydrocarbon oil is dissolved in an extraction solvent, and the aromatic compound is extracted and separated by distillation. Then, a feed stock aromatic hydrocarbon oil is newly supplied to the lean-extraction solvent, and distillation is performed again. Thus, the extraction solvent is recycled in the extractor from the viewpoint of cost reduction, prevention of environmental contamination, and the like.

10 **[0013]** As a result, corrosive ions contained in the successively fed raw-material aromatic hydrocarbon oil build up in the recycled extraction solvent. In conjunction with a small amount of water present in the extraction solvent, an increased concentration of corrosive ions in the extraction solvent causes rapid corrosion of the extractor. Even when an alkali is added to neutralize acid that produces corrosive ions in the extraction solvent as is known in the art, salts build up in the circulation system of the extraction solvent. Also, to date, there has been no technical concept of scavenging corrosive ions by adding an alkali.

15 **[0014]** Thus, an increase of the corrosive ion concentration in the extraction solvent is a problem specific to the extraction solvent of the circulation system, and an approach that provides a greater corrosion suppressing effect is required than in a system where the solvent is used only once and disposed of.

20 **[0015]** The use of a method as described in Patent Literature 1 to address the problem in the circulation system requires a plurality of amines to be optimally combined for neutralization and precipitation and, moreover, the amines are likely to be discharged from the circulation system as volatile components due to distillation, thus deteriorating the corrosion suppressing effect. Depending on the conditions, commonly used alkanolamines and the like are likely to be discharged from the circulation system as volatile components due to distillation and lack the ability to scavenge corrosive ions.

25 **[0016]** The present invention has been made under such circumstances, and an object of the present invention is to provide a method for reducing corrosive ions in an aromatic compound extraction solvent, by which the corrosive ions in the aromatic compound extraction solvent are conveniently and efficiently reduced, in order to suppress corrosion of an extractor caused by corrosive ions contained in an aromatic compound extraction solvent of a circulation system.

### 30 Solution to Problem

**[0017]** The present invention is based on the finding that by scavenging corrosive ions in an aromatic compound extraction solvent in a circulation system as a non-volatile salt and concentrating the extraction solvent containing this salt in a facility, the salt concentration in the system can be lowered and, moreover, by discharging the concentrated salt, the corrosive ions in the circulating extraction solvent can be conveniently and efficiently reduced.

35 **[0018]** That is to say, the present invention is defined in the claims.

### Advantageous Effects of Invention

40 **[0019]** According to the present invention, corrosive ions in an aromatic compound extraction solvent of a circulation system can be conveniently and efficiently reduced.

**[0020]** Accordingly, corrosion of an extractor caused by corrosive ions contained in an aromatic compound extraction solvent of a circulation system can be suppressed, enabling an aromatic compound to be efficiently extracted.

### 45 Brief Description of Drawing

**[0021]** [Fig. 1] Fig. 1 is a schematic system diagram of one example of an aromatic compound extractor.

### Description of Embodiments

50 **[0022]** The method for reducing corrosive ions in an aromatic compound extraction solvent of the present invention is a method applied to a circulation system where the aromatic compound extraction solvent circulates, and chloride ions, sulfate ions, and sulfite ions, i.e., corrosive ions, are reduced. The method includes the step of forming a non-volatile salt by adding a corrosive ion scavenger to react the corrosive ions with the corrosive ion scavenger, and the step of removing the salt by concentrating the solvent containing the salt.

55 **[0023]** Thus, by forming the corrosive ions in the aromatic compound extraction solvent in the circulation system into a non-volatile salt and concentrating the extraction solvent containing the non-volatile salt, the corrosive ions in the solvent that cause corrosion of an extractor can be conveniently and efficiently reduced.

[Circulation system]

**[0024]** The method of the present invention is applied to a circulation system where the aromatic compound extraction solvent contains at least one kind selected from the group consisting of chloride ions, sulfate ions, and sulfite ions that are corrosive ions. An example is a system where a solvent is recycled in the process of extracting an aromatic compound from an aromatic hydrocarbon oil in the refining of a hydrocarbon oil such as petroleum.

**[0025]** The aromatic hydrocarbon oil is, for example, an oil that contains an aromatic compound obtained by reformation, and specifically that contains benzene, toluene or xylene as an aromatic compound, and a mixture of any of these.

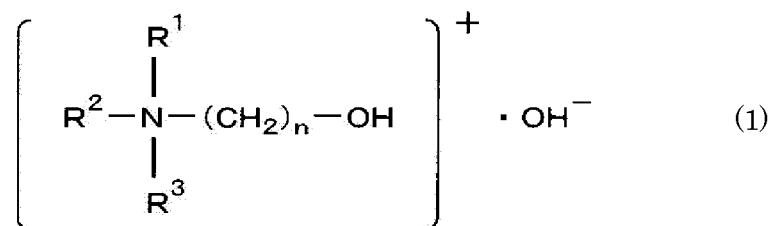
[Aromatic compound extraction solvent]

**[0026]** An aromatic compound extraction solvent commonly used in the refining of a hydrocarbon oil can be used, and examples include sulfolane, diethylene glycol, triethylene glycol, tetraethylene glycol, N-methylpyrrolidone, dimethyl sulfoxide, morpholine, N-formylmorpholine, methyl carbamate, diglycolamine, furfural, phenol, and the like. One of these solvents may be used singly, or two or more may be used in combination. Among these, sulfolane is suitably used. Sulfolane may gradually undergo thermal decomposition during use, and may contain sulfuric acid as an impurity and possibly produce sulfate ions and sulfite ions, but sulfolane does not become problematic because the method of the present invention is also capable of reducing sulfate ions and sulfite ions derived from sulfolane.

[Corrosive ion scavenger]

**[0027]** The corrosive ion scavenger is a compound capable of forming a salt with corrosive ions, and a corrosive ion scavenger not volatilizing in a circulation system to which the corrosive ion scavenger is added is used. The corrosive ion scavenger is strongly basic because the salt formed with corrosive ions is a neutral salt, and the corrosion of an extractor can be further suppressed.

**[0028]** The corrosive ion scavenger is a quaternary ammonium compound represented by general formula (1) below. Due to the concern of an increased ash content in the solvent of the circulation system and from the handleability viewpoint, the inorganic alkali to be added is a quaternary ammonium compound as referred to above.



**[0029]** In formula (1),  $R^1$  to  $R^3$  are each independently a hydrocarbon group having 1 to 4 carbon atoms, and  $n$  is an integer of 1 to 10. Specific examples of  $R^1$  to  $R^3$  include linear or branched alkyl groups and the like, such as a methyl group, an ethyl group, a n-propyl group, an isopropyl group, a n-butyl group, an isobutyl group, a sec-butyl group, and a t-butyl group.

**[0030]** Specific examples of such quaternary ammonium compounds include hydroxymethyltrimethylammonium hydroxide, hydroxymethyltriethylammonium hydroxide, choline hydroxide, 2-hydroxyethyltriethylammonium hydroxide, 3-hydroxypropyltrimethylammonium hydroxide, and the like. One of these may be used singly, or two or more may be used in combination.

**[0031]** Preferably,  $R^1$  to  $R^3$  are each independently a hydrocarbon group having 1 to 3 carbon atoms, and  $n$  is an integer of 1 to 4. When having such a low molecular weight, the quaternary ammonium compound has excellent solubility in water and exhibits a sufficient effect as a corrosive ion scavenger even in a small amount, and thus the extractor corrosion suppressing effect, which is the effect of the present invention, is further increased. Among these, choline hydroxide (also referred to as choline), wherein  $R^1$  to  $R^3$  are all methyl groups and  $n$  is 2, is particularly preferable.

**[0032]** In terms of handling, such a quaternary ammonium compound in general is preferably used as an aqueous solution. The concentration thereof is not particularly limited, and is preferably 5 to 50 mass%.

**[0033]** Since ammonia and other amines are weakly basic, salts formed by these alkalis and corrosive ions are acidic salts, and even a small amount of water, if present, causes corrosion. On the other hand, since the quaternary ammonium compound as described above is strongly basic, the salt formed by corrosive ions is a neutral salt and is unlikely to cause corrosion even in the presence of water.

**[0034]** Moreover, the salt formed by the quaternary ammonium compound and corrosive ions has deliquescent prop-

erties and fluidity, and is thus unlikely to adhere and is free from the risk of deposit buildup and clogging inside the equipment of the circulation system.

5 [0035] The salt formed by the corrosive ion scavenger of the present invention and corrosive ions has a high boiling point and is non-volatile in the circulation system. Therefore, in the distillative refining process for the aromatic hydrocarbon oil or the like, the salt is unlikely to evaporate and enter the extracted aromatic compound, and the salt can be separated from the extraction solvent by distillative concentration and can be removed as a distillation residue.

[0036] The corrosive ion scavenger is a quaternary ammonium compound as described above. While a sufficient corrosive ion scavenging effect can be exerted solely by the corrosive ion scavenger, another agent such as ammonia or a neutralizing amine may be used in combination in order to further increase the corrosive ion scavenging effect.

10 [0037] Examples of the neutralizing amine include monoethanolamine, cyclohexylamine, morpholine, diethylethanolamine, monoisopropanolamine, 3-methoxypropylamine, 2-amino-2-methyl-1-propanol, and the like.

[0038] Concerning the amount of the corrosive ion scavenger to be added, if the corrosive ion concentration in the extraction solvent in the circulation system can be predicted or measured, the frequency and the concentration of the corrosive ion scavenger to be added can be determined based on the corrosive ion concentration. The corrosive ion concentration can be measured with an absorptiometer, an ion chromatograph, a capillary electrophoresis analyzer, or the like.

15 [0039] Concerning the concentration of the corrosive ion scavenger, from the viewpoint of the effect of addition, preferably the corrosive ion scavenger is added so as to be 0.5 to 2 molar equivalents of the corrosive ion concentration, and from the viewpoint of obtaining a more stable corrosive ion scavenging effect, more preferably 1 to 2 molar equivalents.

20 [0040] As means for indirectly detecting the corrosive ion concentration, the metal ion concentration in the extraction solvent in the circulation system may be measured. When corrosion of the extractor progresses with an increase in the corrosive ion concentration in the circulating extraction solvent, the metal ion concentration in the circulation system increases. Accordingly, it is also possible to detect an increase of the corrosive ion concentration by way of an increase of the metal ion concentration. The metal ion concentration can be measured with an absorptiometer, an inductively coupled plasma atomic emission spectrometer (ICP-AES), an inductively coupled plasma mass spectrometer (ICP-MS), or the like.

25 [0041] In this case, it is preferable to find in advance a correlation between the measurement-target metal ion concentration and the corrosive ion concentration in the circulation system. Thus, the frequency and the concentration of the corrosive ion scavenger to be added can be determined based on the metal ion concentration of the circulation system.

30 [0042] Specifically, the measurement-target metal ion is preferably an iron ion that is likely to be eluted due to equipment corrosion.

[0043] By using the method of the present invention as described above as a method for reducing corrosive ions in an extraction solvent when extracting an aromatic compound in a circulation system, corrosion of an extractor caused by corrosive ions contained in the extraction solvent of the circulation system can be effectively suppressed, thus enabling efficient extraction to be performed.

35 [0044] Below, equipment and a treatment mechanism to which the method of the present invention is applied will now be described by way of one example. Fig. 1 shows a schematic system diagram of one example of an aromatic compound extractor to which the method of the present invention is applied.

[0045] In the system flow shown in Fig. 1, a raw-material aromatic hydrocarbon oil that is continuously or intermittently fed from a feed 1 is subjected to a desalting treatment in a chloride trap 2, then mixed with an aromatic compound extraction solvent in an extraction column 3 to extract aromatic compounds such as benzene, toluene, and xylene. Components insoluble in the solvent are withdrawn from the column top and washed in a washing column 4, and then a raffinate 9 is separated.

40 [0046] On the other hand, the aromatic compounds extracted into the extraction solvent are withdrawn from the bottom of the extraction column 3 together with the extraction solvent. Thereafter, the aromatic compounds and the extraction solvent are separated by distillation in a stripper column 5 and a recovery column 6 to obtain the aromatic compounds.

[0047] Here, a corrosive ion scavenger is added as an aqueous solution from an injection facility 10 provided elsewhere in the extraction solvent circulation system, such as in the column top system of the stripper column 5.

45 [0048] The extraction solvent is distilled again for a regeneration treatment in the recovery column 6, subjected to a dehydration treatment in a process water stripper 8, returned to the extraction column 3, and recycled.

[0049] The recovered extraction solvent from the recovery column 6 contains a non-volatile salt produced by reaction between the corrosive ion scavenger and the corrosive ions in the extraction solvent. The recovered extraction solvent containing such a salt is concentrated by distillation in a bypass regenerator 7. By separating and removing a concentration residue and returning a volatilized fraction back to the recovery column 6, a regenerated solvent with reduced corrosive ions is fed to the circulation system.

50 [0050] The corrosive ion scavenger may be added elsewhere in the circulation system. For example, in the above apparatus, the corrosive ion scavenger can be added from the injection facility 10 provided in the column top system of the stripper column 5 that is a subsequent stage relative to the aromatic compound extraction column 3.

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**[0051]** The temperature of the extraction solvent circulation system including the regenerator 7 is 180°C or lower from the viewpoint of preventing volatilization of the corrosive ion scavenger and promoting the corrosive ion scavenging effect thereof in the regenerator 7.

**[0052]** As described above, the salt formed by the corrosive ion scavenger and the corrosive ions is a salt that is non-volatile in the circulation system, and is therefore not discharged on the extract side by, for example, distillation performed for extracting aromatic compounds and can be separated as a distillation residue of solvent concentration in a specific place such as the regenerator 7 provided in the circulation system for solvent regeneration. Moreover, the salt may be removed out of the circulation system by providing a means for discharging remaining residues containing the salt.

Examples

**[0053]** Below, the present invention will now be described in more detail, but the present invention is not limited to the following Examples.

[Test 1] Confirmatory test at laboratory level

(Test 1-1)

**[0054]** First, 100 mL of xylene, 96 mL of sulfolane, and 4 mL of pure water were introduced into a 500 mL round-bottom flask. Hydrochloric acid and an aqueous solution of choline hydroxide (choline) were added thereto to prepare a sample solution having a chloride ion concentration of 100 mg/L and a choline concentration of 340 mg/L.

**[0055]** This sample solution was distilled to remove water. The residual liquid (sulfolane and xylene) remaining in the round-bottom flask was extracted twice with 200 mL of pure water.

(Test 1-2)

**[0056]** A sample solution was prepared that had the same chloride ion concentration and choline concentration as the sample solution of Test 1-1 and to which monoethanolamine (MEA) was added so as to have a monoethanolamine concentration of 172 mg/L and, otherwise, distillation and extraction were performed in the same manner as in Test 1-1.

(Test 1-3, comparative)

**[0057]** A sample solution having the same chloride ion concentration as the sample solution of Test 1-1 to which no choline hydroxide (choline) was added was prepared, and otherwise, distillation and extraction were performed in the same manner as in Test 1-1.

(Test 1-4, comparative)

**[0058]** A sample solution having the same chloride ion concentration and MEA concentration as the sample solution of Test 1-2 to which no choline hydroxide (choline) was added was prepared, and otherwise, distillation and extraction were performed in the same manner as in Test 1-2.

**[0059]** Concerning the extracts of the first and second extractions in the above tests, the chloride ion, choline hydroxide, and MEA concentrations were measured with a capillary electrophoresis analyzer. The measurement results thereof are shown in Table 1.

Table 1

Test No.	1-1	1-2	1-3	1-4	
Amount added (mg/L)					
	Cl <sup>-</sup>	100	100	100	100
	Choline	340	340	-	-
	MEA	-	172	-	172

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(continued)

Cl <sup>-</sup> in extract (mg/L)					
	First	61	109	2.6	<1.0
	Second	11.2	12.9	<1.0	3.7
	Total	72.2	121.9	2.6	3.7

**[0060]** As can be understood from the results shown in Table 1, when choline hydroxide was added (Test 1-1), the chloride ion concentration in the extract was 20 times or higher than when no choline hydroxide was added (Test 1-3), confirming an improved corrosive ion scavenging effect in the aromatic compound extraction solvent.

**[0061]** When choline hydroxide was added (Test 1-1), the chloride ion concentration in the extract was at least 15 times higher than when only MEA was added (Test 1-4), confirming that the corrosive ion scavenging effect in the aromatic compound extraction solvent was superior.

**[0062]** Moreover, when choline and MEA were added (Test 1-2), the chloride ion concentration in the extract was high, and it can be said that the corrosive ion scavenging effect in the aromatic compound extraction solvent is further improved.

[Test 2] Test at actual equipment level

**[0063]** The following test was conducted in the equipment shown in Fig. 1 under a condition where an aromatic hydrocarbon oil containing benzene, toluene, and xylene and sulfolane (having a chloride ion concentration of 70 mg/L) were in circulation.

**[0064]** An aqueous solution of choline hydroxide (choline) was added once a week to a place near the column-top outlet of the stripper column 5. The amount of choline hydroxide added was equimolar to the chloride ions in sulfolane. The concentration of chloride ions was measured by performing extraction with 50 mL of pure water twice, followed by ion chromatography.

**[0065]** After the equipment was operated for a predetermined number of days, 1 L of sulfolane was recovered from the bottom of the recovery column 6, and the chloride ion concentration in this sulfolane was measured. The measurement results are shown in Table 2.

Table 2

Days of operation (days)	1	7	14	20	30	35
Cumulative concentration of choline (mg/L)	267	533	800	1067	1333	1600
Cl <sup>-</sup> in recovered sulfolane (mg/L)	101	88	71	62	52	46

**[0066]** As can be understood from the results shown in Table 2, it was confirmed that the chloride ion concentration in sulfolane recovered from the bottom of the recovery column 6 decreased as the cumulative concentration of choline hydroxide increased. It was also confirmed that choline remained in the recovered sulfolane.

**[0067]** The rate of chloride ion buildup in the bottom of the regenerator 7 was 60 mg/(L·day), i.e., increased about 3 times by adding choline hydroxide relative to 23 mg/(L·day) attained when no choline hydroxide was added.

**[0068]** Accordingly, it can be said that a favorable corrosive ion scavenging effect can be obtained due to choline hydroxide in actual equipment as well.

Reference Signs List

**[0069]**

- 1 Feed
- 2 Chloride trap
- 3 Extraction column
- 4 Washing column
- 5 Stripper column
- 6 Recovery column
- 7 Regenerator
- 8 Process water stripper

- 9 Raffinate  
 10 Injection facility (Place where corrosive ion scavenger is added)

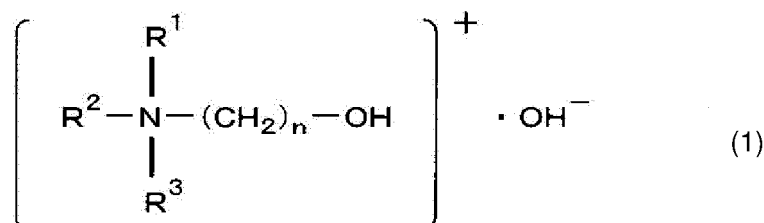
5 **Claims**

1. A method for reducing corrosive ions in an aromatic compound extraction solvent in a circulation system where the aromatic compound extraction solvent circulates,  
 the corrosive ions being at least one kind selected from the group consisting of chloride ions, sulfate ions, and sulfite ions,  
 the method comprising:

a step of forming a non-volatile salt by adding a corrosive ion scavenger to react the corrosive ions with the corrosive ion scavenger,  
 a step of concentrating the solvent comprising the salt, wherein the concentration is performed in a regenerator for the aromatic compound extraction solvent and the temperature of the extraction solvent circulation system including the regenerator is 180°C or lower, and  
 a step of removing the salt,

**characterized in that**

the corrosive ion scavenger is a quaternary ammonium compound represented by general formula (1) below:



wherein R<sup>1</sup> to R<sup>3</sup> are each independently a hydrocarbon group having 1 to 4 carbon atoms, and n is an integer of 1 to 10.

2. The method for reducing corrosive ions in an aromatic compound extraction solvent according to claim 1, wherein the aromatic compound extraction solvent is one or more selected from the group consisting of sulfolane, diethylene glycol, triethylene glycol, tetraethylene glycol, N-methylpyrrolidone, dimethyl sulfoxide, morpholine, N-formylmorpholine, methyl carbamate, diglycolamine, furfural, and phenol.
3. The method for reducing corrosive ions in an aromatic compound extraction solvent according to claim 1 or 2, wherein one or more selected from the group consisting of ammonia and neutralizing amines are also used in the circulation system.
4. The method for reducing corrosive ions in an aromatic compound extraction solvent according to any one of claims 1 to 3, wherein the corrosive ion scavenger is added in a frequency and a concentration determined based on a corrosive ion concentration in the aromatic compound extraction solvent in circulation.
5. The method for reducing corrosive ions in an aromatic compound extraction solvent according to claim 4, wherein the corrosive ion scavenger is added so as to be 0.5 to 2 molar equivalents relative to the corrosive ion concentration.
6. The method for reducing corrosive ions in an aromatic compound extraction solvent according to any one of claims 1 to 3, wherein the corrosive ion scavenger is added in a frequency and a concentration determined based on a metal ion concentration in the aromatic compound extraction solvent in circulation.
7. The method for reducing corrosive ions in an aromatic compound extraction solvent according to claim 6, wherein the metal ion is an iron ion.
8. The method for reducing corrosive ions in an aromatic compound extraction solvent according to any one of claims



1 to 7, wherein the aromatic compound contains one or more selected from the group consisting of benzene, toluene, and xylene.

9. Use of the method for reducing corrosive ions in an aromatic compound extraction solvent according to any one of claims 1 to 8 in a method for extracting an aromatic compound.

### Patentansprüche

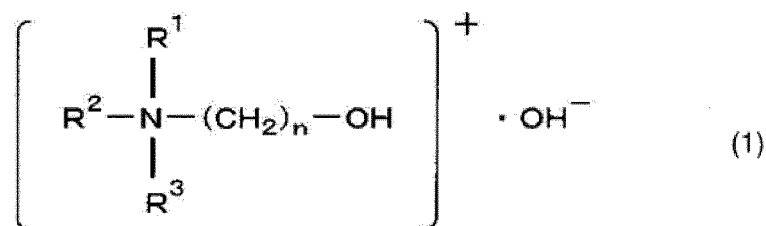
1. Verfahren zur Reduzierung von korrosiven Ionen in einem Lösungsmittel zur Extraktion von aromatischen Verbindungen in einem Zirkulationssystem, in dem das Lösungsmittel zur Extraktion von aromatischen Verbindungen zirkuliert,

die korrosiven Ionen sind wenigstens eine Sorte ausgewählt aus der Gruppe bestehend aus Chloridionen, Sulfationen und Sulfitionen, das Verfahren umfasst:

einen Schritt des Bildens eines nichtflüchtigen Salzes durch Zugabe eines Fängers für korrosive Ionen, um die korrosiven Ionen mit dem Fänger für korrosive Ionen reagieren zu lassen, einen Schritt des Konzentrierens des das Salz umfassenden Lösungsmittels, wobei die Konzentration in einem Regenerator für das Lösungsmittel zur Extraktion von aromatischen Verbindungen erfolgt und die Temperatur des in dem System zirkulierenden Lösungsmittels zur Extraktion einschließlich des Regenerators 180°C oder niedriger ist, und einen Schritt des Entfernens des Salzes,

**dadurch gekennzeichnet, dass**

der Fänger für das korrosive Ion eine quartäre Ammoniumverbindung dargestellt durch die folgende allgemeine Formel (1) ist:



wobei  $R^1$  und  $R^3$  jeweils unabhängig eine Kohlenwasserstoffgruppe sind, die 1 bis 4 Kohlenstoffatome aufweist, und  $n$  eine ganze Zahl von 1 bis 10 ist.

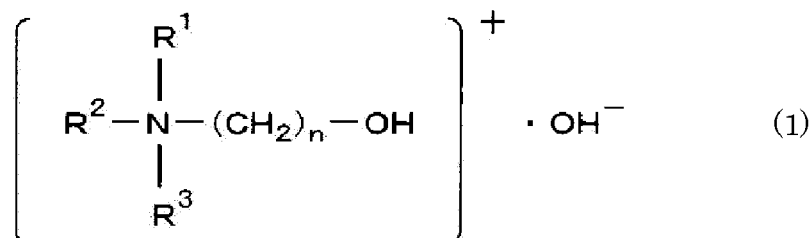
2. Verfahren zur Reduzierung von korrosiven Ionen in einem Lösungsmittel zur Extraktion von aromatischen Verbindungen nach Anspruch 1, wobei das Lösungsmittel zur Extraktion von aromatischen Verbindungen eines oder mehrere ausgewählt aus der Gruppe bestehend aus Sulfolan, Diethylenglycol, Triethylenglycol, Tetraethylenglycol, N-Methylpyrrolidon, Dimethylsulfoxid, Morpholin, N-Formylmorpholin, Methylcarbammat, Diglycolamin, Furfural und Phenol ist.
3. Verfahren zur Reduzierung von korrosiven Ionen in einem Lösungsmittel zur Extraktion von aromatischen Verbindungen nach Anspruch 1 oder 2, wobei eines oder mehrere ausgewählt aus der Gruppe bestehend aus Ammoniak und neutralisierenden Aminen ebenfalls in dem Zirkulationssystem verwendet werden.
4. Verfahren zur Reduzierung von korrosiven Ionen in einem Lösungsmittel zur Extraktion von aromatischen Lösungsmitteln nach einem der Ansprüche 1 bis 3, wobei der Fänger für das korrosive Ion in einer Frequenz und einer Konzentration, die auf der Grundlage einer Konzentration des korrosiven Ions in dem Lösungsmittel zur Extraktion von aromatischen Verbindungen in der Zirkulation bestimmt werden, zugegeben wird.
5. Verfahren zur Reduzierung von korrosiven Ionen in einem Lösungsmittel zur Extraktion von aromatischen Verbindungen nach Anspruch 4, wobei der Fänger für das korrosive Ion zugegeben wird, um relativ zu der Konzentration des korrosiven Ions 0,5 bis 2 molare Äquivalente aufzuweisen.

6. Verfahren zur Reduzierung von korrosiven Ionen in einem Lösungsmittel zur Extraktion von aromatischen Verbindungen nach einem der Ansprüche 1 bis 3, wobei der Fänger für das korrosive Ion in einer Frequenz und einer Konzentration, die auf der Grundlage einer Konzentration eines Metallions in dem Lösungsmittel zur Extraktion von aromatischen Verbindungen in der Zirkulation bestimmt werden, zugegeben wird.
7. Verfahren zur Reduzierung von korrosiven Ionen in einem Lösungsmittel zur Extraktion von aromatischen Verbindungen nach Anspruch 6, wobei das Metallion ein Eisenion ist.
8. Verfahren zur Reduzierung von korrosiven Ionen in einem Lösungsmittel zur Extraktion von aromatischen Verbindungen nach einem der Ansprüche 1 bis 7, wobei die aromatische Verbindung eines oder mehrere ausgewählt aus der Gruppe bestehend aus Benzol, Toluol und Xylol enthält.
9. Verwendung des Verfahrens zur Reduzierung von korrosiven Ionen in einem Lösungsmittel zur Extraktion von aromatischen Verbindungen nach einem der Ansprüche 1 bis 8 in einem Verfahren zur Extraktion einer aromatischen Verbindung.

### Revendications

1. Procédé pour réduire des ions corrosifs dans un solvant d'extraction de composé aromatique dans un système de circulation où circule le solvant d'extraction de composé aromatique, les ions corrosifs étant d'au moins un type choisi dans l'ensemble constitué par les ions chlorure, les ions sulfate, et les ions sulfite, le procédé comprenant :

une étape de formation d'un sel non volatil par addition d'un piègeur d'ions corrosifs pour que les ions corrosifs réagissent avec le piègeur d'ions corrosifs,  
 une étape de concentration du solvant comprenant le sel, dans laquelle la concentration est effectuée dans un régénérateur pour le solvant d'extraction de composé aromatique et la température du système de circulation de solvant d'extraction comprenant le régénérateur est de 180°C ou moins, et  
 une étape d'élimination du sel,  
**caractérisé en ce que** le piègeur d'ions corrosifs est un composé d'ammonium quaternaire représenté par la formule générale (1) ci-dessous :



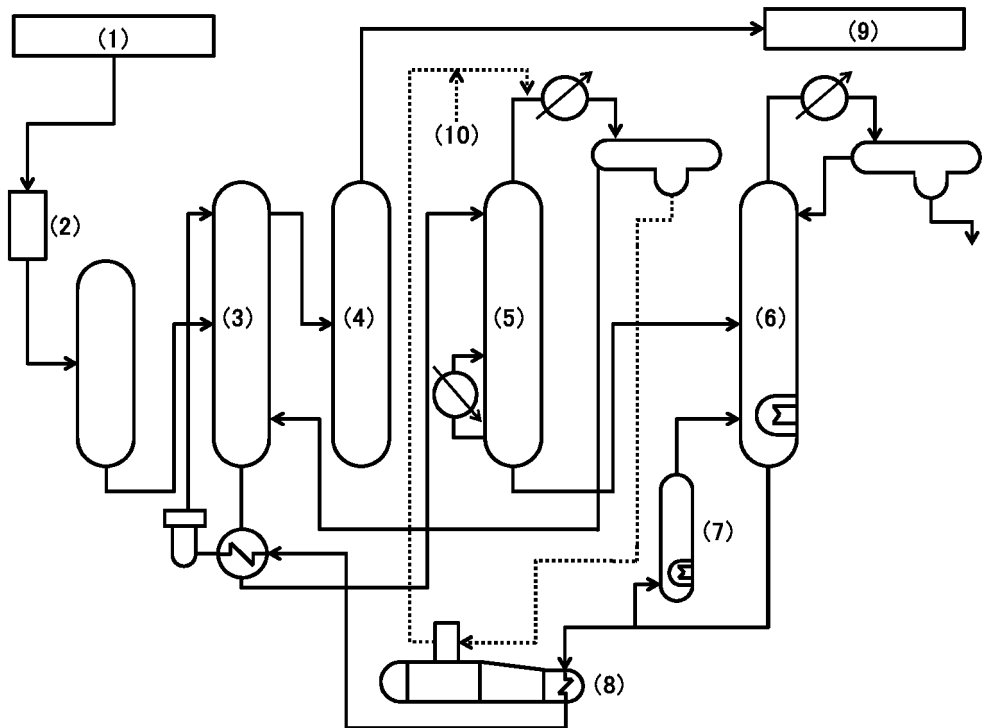
dans laquelle chacun de R<sup>1</sup> à R<sup>3</sup> est indépendamment un groupe hydrocarboné ayant 1 à 4 atomes de carbone, et n est un entier de 1 à 10.

2. Procédé pour réduire des ions corrosifs dans un solvant d'extraction de composé aromatique selon la revendication 1, dans lequel le solvant d'extraction de composé aromatique est un ou plusieurs choisis dans l'ensemble constitué par le sulfolane, le diéthylèneglycol, le triéthylèneglycol, le tétraéthylèneglycol, la N-méthylpyrrolidone, le diméthylsulfoxyde, la morpholine, la N-formylmorpholine, le carbamate de méthyle, la diglycolamine, le furfural, et le phénol.
3. Procédé pour réduire des ions corrosifs dans un solvant d'extraction de composé aromatique selon la revendication 1 ou 2, dans lequel un ou plusieurs choisis dans l'ensemble constitué par l'ammoniac et les aminés neutralisantes sont également utilisés dans le système de circulation.
4. Procédé pour réduire des ions corrosifs dans un solvant d'extraction de composé aromatique selon l'une quelconque des revendications 1 à 3, dans lequel le piègeur d'ions corrosifs est ajouté à une fréquence et à une concentration déterminées sur la base de la concentration d'ions corrosifs dans le solvant d'extraction de composé aromatique

en circulation.

- 5
- 6
- 10
- 15
- 20
- 25
- 30
- 35
- 40
- 45
- 50
- 55
5. Procédé pour réduire des ions corrosifs dans un solvant d'extraction de composé aromatique selon la revendication 4, dans lequel le piègeur d'ions corrosifs est ajouté de façon à être à 0,5 à 2 équivalents molaires par rapport à la concentration d'ions corrosifs.
  6. Procédé pour réduire des ions corrosifs dans un solvant d'extraction de composé aromatique selon l'une quelconque des revendications 1 à 3, dans lequel le piègeur d'ions corrosifs est ajouté à une fréquence et à une concentration déterminées sur la base de la concentration d'ions métalliques dans le solvant d'extraction de composé aromatique en circulation.
  7. Procédé pour réduire des ions corrosifs dans un solvant d'extraction de composé aromatique selon la revendication 6, dans lequel l'ion métallique est un ion fer.
  8. Procédé pour réduire des ions corrosifs dans un solvant d'extraction de composé aromatique selon l'une quelconque des revendications 1 à 7, dans lequel le composé aromatique contient un ou plusieurs choisis dans l'ensemble constitué par le benzène, le toluène, et le xylène.
  9. Utilisation du procédé pour réduire des ions corrosifs dans un solvant d'extraction de composé aromatique selon l'une quelconque des revendications 1 à 8 dans un procédé pour extraire un composé aromatique.

Fig. 1



**REFERENCES CITED IN THE DESCRIPTION**

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**Patent documents cited in the description**

- US 3466345 A [0005]
- US 4191615 A [0006]
- US 2006043340 A1 [0008]
- JP 7180073 A [0009]
- JP 2004211195 A [0009]