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Solvent and Method for Removal of an Acid Gas from a Fluid Stream

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Remias et al.

(54) SOLVENT AND METHOD FOR REMOVAL OF AN ACID GAS FROM A FLUID STREAM

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- (58) Field of Classification Search None

See application file for complete search history.

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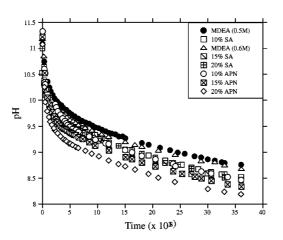
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(57) ABSTRACT

A solvent for removal of an acid gas from a fluid stream includes a promoter amine with a pKa of between 6.5 and 10.5 and a tertiary amine with a pKa of between 8.5 and 10.5.

15 Claims, 5 Drawing Sheets



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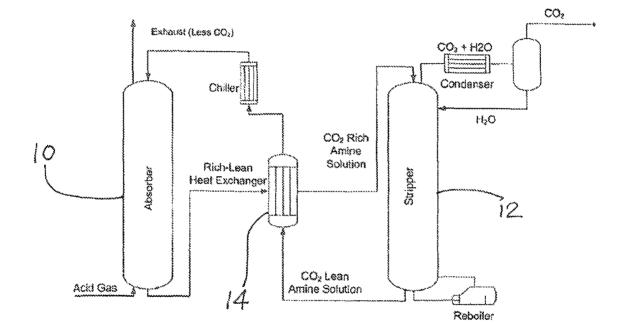
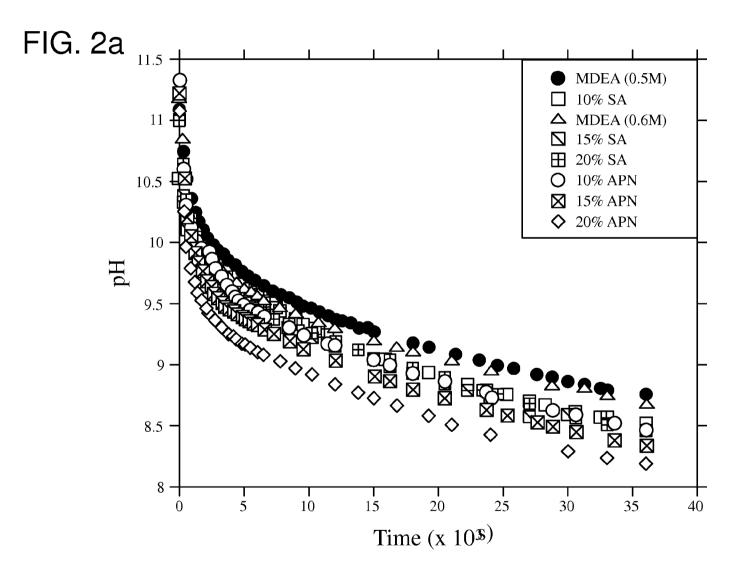
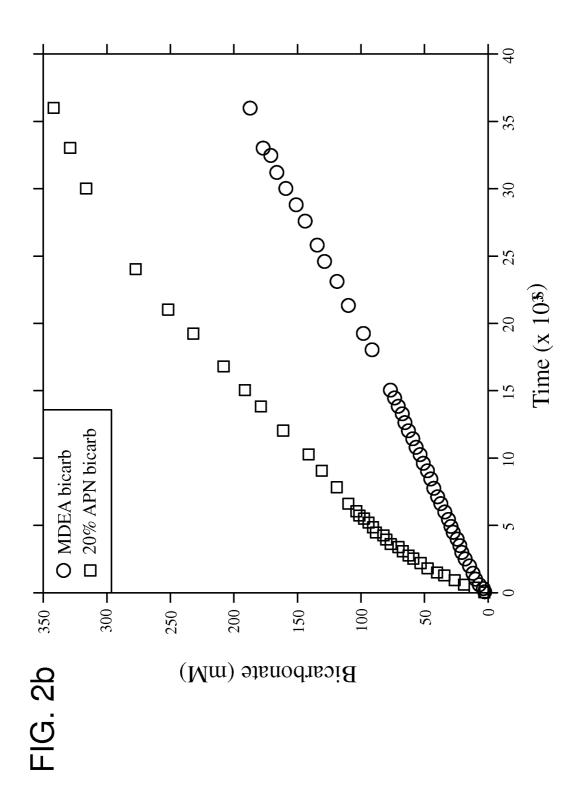
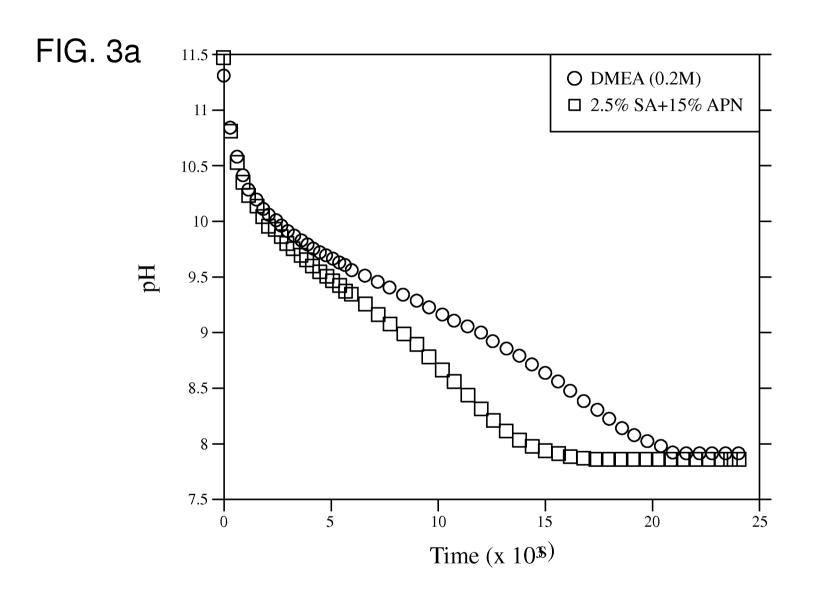
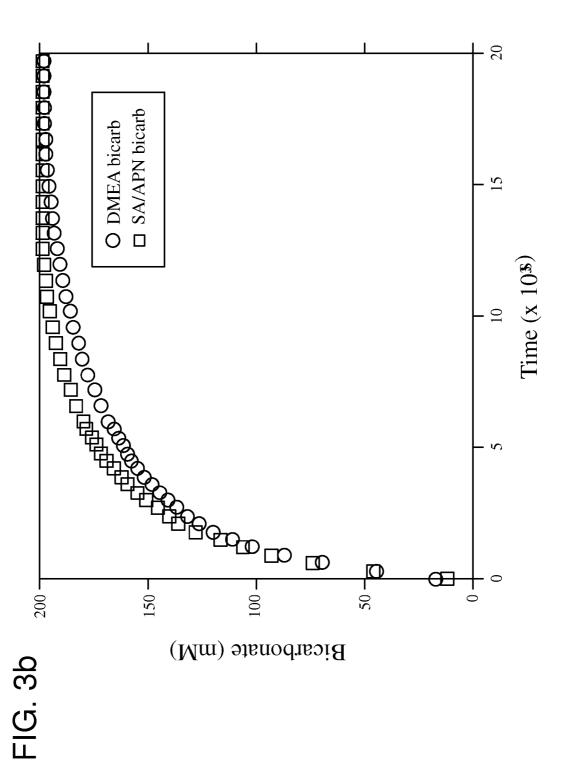


FIG. 1









SOLVENT AND METHOD FOR REMOVAL OF AN ACID GAS FROM A FLUID STREAM

TECHNICAL FIELD

This document relates to the removal of CO₂ and other acid gases from a fluid stream and more particularly to a solvent of promoter and tertiary amines used for separating an acid gas from that fluid stream.

BACKGROUND

The cleanup of acid gasses or sour gas, such as CO₂ in particular, from natural gas and in oil refining has been an extensively practiced technology. The industrial removal of 15 CO₂ from natural gas dates back to the 1930's. In the 21st century, due to the potential impact of anthropogenic CO₂ emissions on the climate, post-combustion CO₂ capture has gained tremendous attention. While several technologies exist for the removal of acid gasses, one of the most 20 commonly employed practices is the use of aqueous amines. Of these amines, tertiary amines are often used for natural gas applications due to their low energy of regeneration. For post-combustion CO2 capture applications primary and secondary amines tend to be in part favored by their faster rate 25 at the low driving force condition. Regardless of the application, the mass transfer rate in the absorber column dictates the size of the column (capital cost) used and, consequently, has a substantial impact on the overall process cost. An overall process depicting a thermal swing process is pre- 30 sented in FIG. 1. An aqueous amine solution is circulated between the absorber 10 and stripper 12. The CO₂ containing gas enters the bottom of the absorber where it contacts the aqueous amine absorbent removing it from the gas stream. The liquid solution, CO_2 rich amine solution, is then 35 passed through a heat exchanger 14 to improve efficiency before being heated to a higher temperature in the stripper 12. The stripper 12 removes the CO_2 as a gas from the amine solution to produce a lean, or CO₂ deficient solution. The lean solution is returned to the absorber 10 by way of the 40 as carbon dioxide from a fluid stream such as a flue gas heat exchanger to repeat the process.

In order to minimize system capital (absorber cost) it is important to maximize the overall mass transfer rate for the scrubber system as there is a direct correlation between the two. Primary (RNH₂) and secondary (R₂NH) amines are 45 capable of achieving a high mass transfer rate per unit due to the direct chemical reaction with CO₂ to form a carbamate. However, they show a lower than desirable capacity. It requires 2 moles of amine to capture 1 mole of CO₂ since the carbamate is then negatively charged another amine must 50 absorb the proton formed. Furthermore, due to the high enthalpy of absorption from the formation of the carbamate the regeneration energy is high. Tertiary amines (R_3N) show a significant decrease in mass transfer rates due to the inability to react with CO2 directly; however, they show 55 significantly lower heats of regeneration. The described process seeks to exploit the advantages of both systems without inheriting the limitations exhibited by either. More specifically, by promoting the CO₂ capture reaction rate the absorber will be smaller and reduce the process capital. The 60 intrinsic lower energy of regeneration for the tertiary amine solvent minimizes operating costs.

While gas sweetening applications represent the most immediate opportunity for application of the described invention, post-combustion CO_2 capture could represent a 65 large potential application of the described technology. The market driver for this application will be the regulation of

CO2 emissions due to concern about its environmental impact towards global climate change or a need for CO₂ for utilization purposes such as enhanced oil recovery (EOR). Carbon dioxide capture and sequestration (CCS) from large stationary sources such as fossil fuel combusting electricity generators represents one method to reduce the increase in atmospheric CO2 levels. The challenges of post-combustion CO₂ capture include the fact that flue gas from utility boilers is at near atmospheric pressure and the concentration of CO_2 ¹⁰ in the flue gas is relatively low at 12-14%. Another technical hurdle is the energy requirements for the CO₂ capture/ desorption devices to regenerate absorber reagents. Generally speaking, the energy required for CO₂ capture and sequestration using MEA is estimated to reduce a PC plant's output by about 30 percent, which equates to a very substantial 60-80% increase in the cost of electricity. The ability to capture and store CO₂ more efficiently will be highly valued by utilities.

In broad terms the described method seeks to add a promoter amine, in the form of a substituted primary and/or secondary amine to a tertiary amine to form a solvent. The promoters serve to increase the overall mass transfer of the acid gas into the absorption solvent. The promoters are designed to have particularly low volatility without contributing significant viscosity to the solution. The described promoters achieve this attribute without being an ionic compound which can negatively impact mass transfer. The low volatility and low viscosity are achieved by using alternate functional groups in addition to the amine functional group that reacts with the CO2 molecule. Low volatility is important to reduce amine loss in the CO_2 capture process. This property is often achieved by using alcohol groups in addition to the amine group. However, the alcohol groups are hydrogen bond donors which add more solution viscosity due to intermolecular bonding.

SUMMARY

A solvent is provided for the removal of an acid gas such stream. The solvent comprises (a) a promoter amine selected from a group of amines consisting of primary amines with a pKa between 6.5 and 10.5, secondary amines with a pKa between 6.5 and 10.5 and mixtures thereof and (b) a tertiary amine with a pKa of between 8.5 and 10.5. More particularly the solvent includes 0.01-15.0 weight percent promoter amine, 10.0-65.0 weight percent tertiary amine and 34.44-89.99 weight percent water.

The promoter amine includes at least one non-volatilizing functional group selected from a group consisting of $-SO_2$, -OP(OR)₃, --CN, --OPR₃, --OR and --COOR and mixtures thereof where R=-H or alkyl. In one embodiment the promoter amine is 3-N-sulfonylamine (SA). In one embodiment the promoter is N-methyltetrahydrothiophen-3-amine 1,1-dioxide. In one embodiment the promoter is 2,2'-sulfonyldiethanamine. In one embodiment the promoter is 3.3'sulfonyldipropaneamine. In one embodiment the promoter is 4,4'-sulfonyldibutanenamine. In one embodiment the promoter is 2-aminoethyl methyl sulfone. In one embodiment the promoter amine is 3-aminopropionitrile (APN). In one embodiment the promoter is 4-aminobutanenitrile. In one embodiment the promoter is 5-aminopentanenitrile. In one embodiment the promoter is 6-aminohexanenitrile. In one embodiment the promoter is 3-(methylamino)propanenitrile. In one embodiment the promoter amine is diethyl (2-aminoethyl)phosphonate (EtP2). In one embodiment the promoter is diethyl [2-(methylamino)ethyl]phosphonate. In one

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embodiment the promoter is diethyl (3-aminopropyl)phosphonate. In one embodiment the promoter is diethyl (4-aminobutyl)phosphonate. In one embodiment the promoter is diethyl (5-aminopentyl)phosphonate. In one embodiment the promoter is diethyl (6-aminohexyl)phosphonate. In one embodiment the promoter is 2-(tert-butoxy) ethan-1-amine. In one embodiment the promoter is N-methyl-2-[(2-methyl-2-propanyl)oxy]ethanamine. In one embodiment the promoter amine is a mixture of two or three amines selected from a group consisting of 3-N-sulfonylamine (SA), 3-aminopropionitrile (APN) and diethyl 2-aminoethanephosphonate (EtP2). The tertiary amine is selected from a group of tertiary amines consisting of methyldiethanolamine (MDEA), triethanolamine (TEA), N,N,-dialkylethanolamine, N,N,N'N'-tetraalky-1,8-naphthalenediamine, N,N,-dialkylbenzylamine, 1,4-dialkylpiperazine, N,N,N',N'tetraalkyl-1,6-hexanediamine, N,N,N',N'-tetraalkyl-1,5-pentanediamine, N,N,N',N'-tetraalkyl-1,4-butanediamine, N,N, N',N'-tetraalkyl-1,3-propanediamine, N,N,N',N'-tetraalkyl- 20 N,N,N',N'-tetrakis(2-hydroxyethyl) 1,2-ethanediamine, N,N,N'N'N"ethylenediamine, N,N,N'N'N"pentaalkyldiethylenetriamine, pentaalkyldipropylaminetriamine, N,N,-

N,N,N',N'-tetraalkylbis²⁵ dialkylcyclohexylamine, (aminoethyl)ether, N,N,-dimethyl-2(2-aminoethoxy) ethanol, where alkyl represents any methyl, ethyl, propyl, butyl isomer, and mixtures thereof. In one particularly useful embodiment the promoter amine comprises a mixture of 3-N-sulfonylamine (SA) and 3-aminopropionitrile (APN) while the tertiary amine is methyldiethanolamine (MDEA). More particularly the solvent comprises about 0.1 weight percent 3-N-sulfonylamine (SA), about 0.25 weight percent 3-aminopropionitrile (APN) and between about 2.0 and 35 about 65.0 weight percent methyldiethanolamine (MDEA).

In accordance with an additional aspect, a method is provided for removing an acid gas from a fluid stream. That method comprises contacting the fluid stream with a solvent for the removal of acid gas from the fluid stream. That $_{40}$ solvent includes a promoter amine with a pKa of between about 6.5 and 10.5 and a tertiary amine with a pKa of between 8.5 and 10.5. Other details of the solvent are the same as described above.

Additional steps of the method include binding the acid 45 gas in the fluid stream to the solvent to form an acid gas-solvent complex, separating the acid gas solvent complex from the fluid stream, regenerating the solvent by releasing the acid gas and recycling the regenerated solvent for contacting the fluid stream.

In the following description there is shown and described preferred embodiments of the solvent and method for removing an acid gas from a fluid stream such as, for example, CO_2 from flue gas. As it will be realized, these solvents and methods are capable of other different embodi- 55 ments and their several details are capable of modification in various, obvious aspects. Accordingly, the drawings and descriptions will be regarded as illustrative in nature and not as restrictive.

BRIEF DESCRIPTION OF THE DRAWINGS

The accompanying drawings incorporated herein and forming a part of the specification, illustrate several aspects of the present invention and together with the description 65 serve to explain certain principles of the invention. In the drawings:

FIG. 1 is a schematical illustration of a process for removing acid gas from a fluid stream utilizing a solvent and thermal swing regeneration.

FIG. 2a is a graphical illustration of the pH-drop of a 0.5 M MDEA solution with various concentrations of solvent promoters.

FIG. 2b is a graphical illustration of bicarbonate formation via CO₂ hydration in 0.5 M MDEA and 0.5 M MDEA with 20% APN.

FIG. 3a is a graphical illustration of the pH-drop of a 0.2 M DMEA solution and 0.2 M DMEA solution with solvent promoters.

FIG. 3b is a graphical illustration of the bicarbonate formation via CO₂ hydration in 0.2 M DMEA and 0.2 M 15 DMEA with a 2.5% and 15% APN mixture.

Reference will now be made in detail to the present preferred embodiment of the invention, examples of which are illustrated in the accompanying drawings.

DETAILED DESCRIPTION

A solvent or solvent system for removal of an acid gas from a fluid stream comprises a promoter amine with a pKa of between 6.5 and about 10.5 and a tertiary amine with a pKa of between 8.5 and 10.5. More specifically the solvent includes about 0.01-15.0 weight percent promoter amine, about 10.0-65.0 weight percent tertiary amine and about 34.44-89.99 weight percent water.

The promoter amine useful in the solvent system includes a primary amine and/or a secondary amine with at least one non-volatilizing functional group selected from a group consisting of -SO₂, -OP(OR)₃, -CN, -OPR₃, -OR and —COOR and mixtures thereof where R=—H or alkyl. For purposes of this document alkyl refers to branched and unbranched alkyl compounds with between 1 and 10 carbon atoms. In one embodiment the promoter amine is 3-Nsulfonylamine (SA). In one embodiment the promoter is N-methyltetrahydrothiophen-3-amine 1,1-dioxide. In one embodiment the promoter is 2,2'-sulfonyldiethanamine. In one embodiment the promoter is 3,3'-sulfonyldipropaneamine. In one embodiment the promoter is 4,4'-sulfonyldibutanenamine. In one embodiment the promoter is 2-aminoethyl methyl sulfone. In one embodiment the promoter amine is 3-aminopropionitrile (APN). In one embodiment the promoter is 4-aminobutanenitrile. In one embodiment the promoter is 5-aminopentanenitrile. In one embodiment the promoter is 6-aminohexanenitrile. In one embodiment the promoter is 3-(methylamino)propanenitrile. In one embodiment the promoter amine is diethyl (2-aminoethyl) phosphonate (EtP2). In one embodiment the promoter is diethyl [2-(methylamino)ethyl]phosphonate. In one embodiment the promoter is diethyl (3-aminopropyl)phosphonate. In one embodiment the promoter is diethyl (4-aminobutyl) phosphonate. In one embodiment the promoter is diethyl (5-aminopentyl)phosphonate. In one embodiment the promoter is diethyl (6-aminohexyl)phosphonate. In one embodiment the promoter is 2-(tert-butoxy)ethan-1-amine. In one embodiment the promoter is N-methyl-2-[(2-methyl-2-propanyl)oxy]ethanamine. In one embodiment the pro-60 moter amine is a mixture of two or three amines selected from a group consisting of 3-N-sulfonylamine (SA), 3-aminopropionitrile (APN) and diethyl 2-aminoethanephosphonate (EtP2).

The tertiary amine is selected from a group of tertiary amines consisting of methyldiethanolamine (MDEA), triethanolamine (TEA), N,N,-dialkylethanolamine, N,N,N',N'tetraalky-1,8-naphthalenediamine, N,N,-dialkylbenzylam-

1,4-dialkylpiperazine, N,N,N',N'-tetraalkyl-1,6ine. hexanediamine, N,N,N',N'-tetraalkyl-1,5-pentanediamine, N.N.N'.N'-tetraalkyl-1,4-butanediamine, N.N.N'.N'-tetraalkyl-1,3-propanediamine, N,N,N',N'-tetraalkyl-1,2-ethanediamine, N,N,N',N'-tetrakis(2-hydroxyethyl)ethylenedi- 5 amine, N,N,N'N'N"-pentaalkyldiethylenetriamine, N,N, N'N'N"-pentaalkyldipropylaminetriamine, N.N.dialkylcyclohexylamine, N,N,N',N'-tetraalkylbis (aminoethyl)ether, N,N,-dimethyl-2(2-aminoethoxy) ethanol, where alkyl represents any methyl, ethyl, propyl, 10 butyl isomer, and mixtures thereof. One particularly useful solvent embodiment comprises a promoter amine mixture of 3-N-sulfonylamine (SA) and 3-aminopropionitrile (APN) in combination with a tertiary amine of methyldiethanolamine (MDEA). More specifically the solvent comprises about 0.1 15 weight percent 3-N-sulfonylamine (SA), about 0.25 weight percent 3-aminopropionitrile (APN) and between about 2.0 and about 65.0 weight percent methyldiethanolamine (MDEA). The remainder is water.

Also disclosed is a method of removing an acid gas from ²⁰ a fluid stream. That method comprises contacting the fluid stream with a solvent for removal of the acid gas. As described above, the solvent includes a promoter amine with a pKa of between 6.5 and 10.5 and a tertiary amine with a pKa of between 8.5 and 10.5. The method further includes ²⁵ the step of binding the acid gas in the fluid stream to the solvent to form an acid gas-solvent complex. In addition the method includes separating the acid gas solvent complex from the fluid stream. Further the method includes regenerating the solvent by releasing the acid gas. Finally the ³⁰ method includes recycling the regenerated solvent for contacting the fluid stream.

The following Example is provided to further illustrate the solvent and related method for removal of an acid gas such as CO_2 from a fluid stream such as flue gas. While ³⁵ representative of the solvent and method it should be appreciated that this Example is not to be considered as limiting in scope.

EXAMPLE 1

The current solvent system/process relates to a more efficient removal of carbon dioxide from a gaseous stream containing carbon dioxide and/or other acidic gases. In particular, the method for separating carbon dioxide from a 45 gas mixture uses a combination of less basic primary amines (pKa < 10.5) containing functional groups that provide a decrease in vapor pressure and viscosity in the presence of a more basic tertiary amine (pKa > 8.5).

The combination of amines provides an overall increase 50 in the mass transfer rate to more closely resemble that of a primary amine, but maintains the low heat of regeneration exhibited by tertiary amines. The primary amines have the ability to react directly with CO_2 in the gas stream to form an initial carbamate which serves as a promoter (transfer 55 agent) for the more basic tertiary amine to form bicarbonate where the CO_2 is now considered absorbed.

For purposes of example, the solvent system consists of 1-20 mol % of a primary amine containing a non-volatilizing functional group, SO₂, OP(OR)₃, or CN with pKa ranging 60 between 6.5 and 10.5 and a tertiary amine, methylydiethanolamine (MDEA), triethanolamine (TEA), dimethylethanolamine (DMEA) for examples, with pKa ranging between 8.5 and 10.5. The functionalized primary amines have lower or similar vapor pressures vs. the traditional primary amines 65 such as MEA. A low vapor pressure for the amine is critical to reduce the potential for atmospheric emissions from the

absorber stack or contamination of the natural gas stream produced. Table 1 shows the low vapor pressure for compounds with representative functional groups compared to MEA. These same functional groups also aid in decreasing the viscosity of the solution due to the lack of participating in an extended H-bonding network.

TABLE 1

	The vapor pressure of example amines with non-hydrogen bonding functional groups		
	Functional group	Example compound	Vapor pressure (Pa)*
_	НО	Monoethanolamine (MEA)	55
	SO2	Sulfonylamine (SA)	0
	CN	3-aminopropionitrile	89
	OP(OR) ₃	Aminoethyl-dimethoxyphosphine oxide	16

*Vapor pressure predicted using ACD/I-Lab at 25° C.

Addition of 3-N-sulfonylamine (SA), 3-aminopropionitrile (APN), or a combination of the two to a tertiary amine solution (0.2 M-0.5 M) resulted in an up to 300% increase in relative rate of CO_2 hydration (FIG. 2). As shown in FIG. 2a, the relative rate of CO₂ hydration increases with increased amounts of solvent promoters added. However, it was observed that different promoters, based on pK_a , show different maximum levels of promotion. For example, SA shows no increase in promotion above ~3-5 mol % of tertiary amine concentration, while APN maintains its promoter effects up to at least 20 mol % based on tertiary amine concentration. The effectiveness of the solvent promoter was judged on its ability to increase the relative rate of bicarbonate formation from CO₂ and H₂O (FIG. 2b). It was observed that addition of 20 mol % APN to a 0.5M MDEA solution gives significant enhancement of the relative rate of bicarbonate formation. The rate enhancement and increased bicarbonate formation is not a factor of the summation of the two amines acting independently (data not shown). If the 40 amount of bicarbonate formed by APN independently is added to the amount of bicarbonate formed by MDEA independently then the total amount of bicarbonate is less than that of when the two are combined in one solution. This strongly suggests that APN is acting as a promoter for carbon capture by MDEA.

The solvent promoters show promising results in MDEA solutions. In order to understand the scope of these promoters, various solvents were tested. It was observed that the different promoters showed a varying amount of activity in other solvents. For example, the optimal mixture used for the best enhancement in MDEA (2.5% SA and 15% APN) showed a ~150% increase in relative rate in DMEA solutions (FIG. 3). While some solvents showed more significant results than others, it shows the versatility of using less basic primary amines as promoters for more basic tertiary amines.

The foregoing has been presented for purposes of illustration and description. It is not intended to be exhaustive or to limit the embodiments to the precise form disclosed. Obvious modifications and variations are possible in light of the above teachings. For example, while certain promoter amines and tertiary amines are identified above as being useful in the present solvent and method, these are to be considered illustrative of possible promoter and tertiary amines that may be used rather than limiting. Similarly, while this document references CO_2 capture from a flue gas, the method is suited for other applications where acid gas capture from a fluid stream is desired. All such modifications 10

and variations are within the scope of the appended claims when interpreted in accordance with the breadth to which they are fairly, legally and equitably entitled.

What is claimed:

1. A solvent for removal of an acid gas from a fluid stream, 5 comprising:

a promoter amine comprising a mixture of N-sulfonylamine (SA) and 3-aminopropionitrile (APN), wherein SA comprises 3-5 mol % and APN comprises 1-20 mol %; and

a tertiary amine with a pKa of between 8.5 and 10.5.

2. The solvent of claim **1** including 0.01 to 15.0 weight percent promoter amine, 10.0 to 65.0 weight percent tertiary amine and 34.99 to 89.99 weight percent water.

3. The solvent of claim 1, wherein said tertiary amine is 15 selected from a group of tertiary amines consisting of methyldiethanolamine (MDEA), triethanolamine (TEA), N,N,-dialkylethanolamine, N,N,N',N'-tetraalky-1,8-naphthalenediamine, N,N,-dialkylbenzylamine, 1,4-dialkylpiperazine, N.N.N'.N'-tetraalkvl-1.6-hexanediamine, N.N.N', 20 N'-tetraalkyl-1,5-pentanediamine, N,N,N',N'-tetraalkyl-1,4butanediamine, N,N,N',N'-tetraalkyl-1,3-propanediamine, N,N,N',N'-tetraalkyl-1,2-ethanediamine, N,N,N'N'-tetrakis (2-hydroxyethypethyl) ethylenediamine, N,N, N',N"-pentaalkyldiethylenetriamine, N.N.N',N',N"-pentaalkyldipropy- 25 laminetriamine, N,N,-dialkylcyclohexylamine, N,N,N',N'tetraalkylbis (aminoethyl)ether, N,N,-dimethyl-2(2aminoethoxy)ethanol, where alkyl represents any methyl, ethyl, propyl, butyl isomer, and mixtures thereof.

4. The solvent of claim **1**, wherein said tertiary amine is 30 selected from a grou of tertiary amines consisting of methyldiethanolamine (MDEA), triethanolamine (TEA), dimethylethanolamine (DMEA), N,N,-dimethylbenzylamine, diethylmonoethanolamine, dipropylmonoethanolamine, 1,4-dimethylpiperazine, N,N,N',N'-tetramethyl-1,6-hexanedi-35 amine, N,N,N',N'-tetrakis(2-hydroxyethyl)ethylenediamine, N,N,N',N'-tetramethylethylenediamine, N,N,N',N'-tetra

5. The solvent of claim **1**, wherein said tertiary amine is methyldiethanolamine (MDEA).

6. The solvent of claim **5**, wherein said solvent comprises about 0.1 weight percent N-sulfonylamine (SA), about 0.25

weight percent aminopropionitrile (APN) and between about 2.0 and about 65.0 weight percent methyldiethanolamine and a remainder is water.

7. The solvent of claim 1, wherein said acid gas is carbon dioxide.

8. A method of removing an acid gas from a fluid stream, comprising:

- contacting said fluid stream with a solvent for removal of said acid gas from said fluid stream, and said solvent including:
- (a) a promoter amine comprising a mixture of N-sulfonylamine and 3-aminopropionitrile, wherein N-sulfonylamine comprises 3-5 mol % and 3-aminopropionitrile comprises 1-20 mol %; and
- (b) a tertiary amine with a pKa of between 8.5 and 10.5.

9. The method of claim **8**, including providing said solvent with 0.01 to 15.0 weight percent promoter amine, 10.0 to 65.0 weight percent tertiary amine and 34.99 to 89.99 weight percent water.

10. The method of claim 8, including using a tertiary amine selected from a group of tertiary amines consisting of methyldiethanolamine (MDEA), triethanolamine (TEA), dimethylethanolamine (DMEA), N,N,-dimethylbenzylamine, diethylmonoethanolamine, dipropylmonoethanolamine, 1,4-dimethylpiperazine, N,N,N',N'-tetramethyl-1,6-hexanediamine, N,N,N',N'-tetrakis(2-hydroxyethyl)ethylenediamine, N,N,N',N'-pentamethyldiethylenetriamine, N,N, N',N'-tetramethylethylenediamine, N,N,N',N'tetramethylpropane-1,3-diamine, N,N,N',N'tetramethylbutane-1,4-diamine, N,N,N',N'-tetramethyl-1,5pentanediamine, and mixtures thereof.

11. The method of claim **8**, including methyldiethanolamine (MDEA) as said tertiary amine.

12. The method of claim 8, further including binding acid gas in said fluid stream to said solvent to form an acid gas-solvent complex.

13. The method of claim 12, including separating said acid gas solvent complex from said fluid stream.

14. The method of claim 13, including regenerating said solvent by releasing said acid gas.

15. The method of claim **14**, including recycling said regenerated solvent for contacting said fluid stream.

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