

Use of Hot N-Methyl-2-pyrrolidone (NMP) from Solvent Extraction Operation to Supply Heat to Strip H₂S from NMP-Scrubbing Solution

The present invention relates to the use of hot N-methyl-2-pyrrolidone (NMP) from a solvent extraction operation to supply heat to strip H₂S from an NMP-scrubbing solution. More particularly, this invention relates to an improved process for scrubbing H₂S from a gas with NMP to form an H₂S-rich solution which is then heated and sent to a stripping zone to remove the H₂S from the NMP wherein the improvement comprises using hot NMP recovered from extracted hydrocarbon oil to heat the H₂S-rich NMP solution before same is sent to said stripping zone. Still more particularly, in a process for removing H₂S from a hydrofiner tail gas wherein said H₂S-containing gas is passed into a scrubbing zone wherein it is contacted with liquid NMP to remove most of the H₂S from the gas to form an H₂S-rich NMP solution, heating said H₂S-rich NMP solution and passing said hot solution to a stripping zone to remove most of the H₂S from the NMP to form an H₂S-lean NMP solution, extracting a hydrocarbon oil with said H₂S-lean NMP solution and recovering hot NMP from said hot extracted oil; the improvement which comprises heating said H₂S-rich NMP solution to the temperature required for stripping same by indirectly contacting same, in heat exchange relationship, with at least a portion of said hot, liquid NMP recovered from said extracted oil.

It is known from US—A—3738086 to remove SO₂ from an SO₂-containing gas by scrubbing the said gas with NMP to form a NMP-SO₂ solution which is thereafter subjected to indirect heat exchange to raise its temperature so that SO₂ is subsequently liberated from the heated solution in a distillation vessel; the indirect heat exchange being with hot SO₂-containing gas and with hot NMP leaving the distillation vessel and wherein the hot NMP obtains its heat from the hot gas and from a boiler.

It is known from US—A—3324627 to use NMP to scrub or absorb H₂S from various H₂S-containing gas streams thereby forming an H₂S-rich NMP solution. It is known from US—A—3120993 and 3492788 and GB—A 1153786 to regenerate the NMP by heating up the H₂S-rich NMP solution and stripping off the H₂S in a stripping zone, cooling the so-regenerated NMP and recycling it back into the scrubber. It is also known in the art to use NMP for hydrocarbon extraction processes such as the BTX (benzene, toluene, xylene) process for separating paraffins from gasoline fractions and for lube oil extraction processes. In all of the hydrocarbon extraction processes employing NMP as the extraction solvent, it is economically necessary to recover the solvent. This is generally done by a combination of flash evaporation and distillation, with gas stripping used to remove residual amounts of NMP from

the extracted hydrocarbon, as disclosed in, e.g. GB—A—1573259. The solvent-free hydrocarbon is then often sent to a hydrofiner for final purification by contact with hydrogen and a catalyst wherein polar compounds containing sulfur, nitrogen and oxygen, as well as color bodies, and unstable compounds, etc., are removed. The gas exiting from a hydrofiner is generally relatively rich in hydrogen. By way of illustrative, but non-limiting example, a typical tail gas from a lube oil hydrofiner will comprise about 71% mole hydrogen, 13% methane, 5% ethane, 5% C₃+ alkanes and 6% H₂S. Thus, it can be seen that this gas contains considerable quantities of H₂S. This gas may be used in another hydrofiner in order to consume more of the hydrogen or it may be sent directly to a scrubber in another part of the refinery to remove the H₂S by scrubbing with an amine such as MEA (monoethanolamine). The H₂S is then sent to a sulfur recovery plant and the scrubbed gas can be used as fuel or for other purposes.

It would be advantageous to use this H₂S-containing tail gas for gas stripping in a lube oil or other extraction solvent recovery system on a once-through basis and ultimately use the spent gas from the solvent recovery system as fuel in the solvent recovery furnaces. However, in order to accomplish this, the H₂S must be removed from the gas prior to its use in the solvent stripping operation in order to avoid excessive metal corrosion in the solvent recovery system.

It is undesirable to scrub the gas with caustic soda solution, because the high levels of hydrogen sulfide in the gas would require excessive quantities of caustic and also because of the pollution problem caused by the disposal of large amounts of spent caustic. Scrubbing the gas with MEA, DEA (diethanolamine) or "Catacarb" (hot potassium carbonate solution) is feasible, but this would introduce a whole new system of scrubbing and regeneration, pumps, solution storage, filters, etc., as well as introducing potential NMP solvent contamination to the H₂S extraction process. Therefore, it would be advantageous if a way could be found to strip hydrogen sulfide from these gases in an economical manner and in a way such that the H₂S gas scrubbing and NMP regeneration process is integrated with the solvent recovery operation or section of an NMP solvent extraction process, particularly if a way could be found to do this without having to introduce any additional energy or contaminants in the system which is required as taught by the prior art.

The present invention provides a process for removing H₂S from an H₂S-containing gas stream, comprising passing said gas stream into a scrubbing zone wherein it is contacted with liquid N-methyl-2-pyrrolidone (NMP) to remove most of the H₂S from said gas stream to form an

H₂S-rich NMP solution, heating said H₂S-rich NMP solution by indirect heat exchange with a hot stream of fluid and passing the resulting heated solution to a gas stripping zone to remove most of the H₂S from said solution to form an H₂S-lean NMP solution, wherein the process comprises the following combination of features: (a) the H₂S-lean NMP solution is combined with hot liquid NMP obtained by recovering NMP from a mixture comprising hydrocarbon oil and NMP, formed by solvent extraction of a hydrocarbon oil with NMP, to form a hot combined solution; (b) a portion of the combined solution is employed as the said hot stream of fluid which constitutes the sole heat source for heating the said H₂S-rich NMP solution before entering the stripping zone; and (c) after passing in indirect heat exchange relationship with the H₂S-rich NMP solution, the said portion of the combined solution is recycled to the scrubbing zone.

The NMP solvent (N-methyl-2-pyrrolidone) may contain minor amounts of water ranging from about 0.1 up to 10 wt.% or more and may also contain minor amounts (i.e., 0.1 to 10 wt.%) of other components such as the extracted hydrocarbon oil. The gas may be any non-aqueous, H₂S-containing gas that is relatively inert with respect to the NMP. The gas to be scrubbed will generally contain from about 0.5 to 15 volume % H₂S and may come from any source. However, in a preferred embodiment the gas will be a tail gas from a hydrofiner. In a particularly preferred embodiment of this invention, the process of this invention will be integrated with a lube oil extraction process employing NMP as the extraction solvent and using gas stripping in the solvent recovery section of the extraction process, wherein tail gas from a hydrofiner, containing excessive amounts of H₂S, is scrubbed with NMP recovered from the lube oil extraction process. The scrubbed tail gas is then used as once-through stripping gas in the lube oil extraction process solvent recovery operation, with at least a portion of said spent stripping gas passed through the stripping zone of the process of this invention to strip H₂S from the H₂S-rich NMP solution.

Brief Description of the Drawings

The attached drawing is a flow diagram of a preferred embodiment of an H₂S scrubbing process employing the improvement of the instant invention.

Detailed Description

Referring to the drawing, a mixture of stripping gas and condensed NMP solvent at a temperature ranging from about 148.9 to 204.4°C (300 to 400°F) and a gauge pressure ranging from about 379 to 516 kPa (55 to 75 psig), obtained from the solvent recovery section (not shown) of a lube oil extraction plant employing NMP as the extraction solvent and

wherein said solvent recovery includes gas stripping, is passed via line 10 to solvent drum 12 wherein the spent stripping gas is disengaged from the hot NMP. At least a portion of the disengaged stripping gas is passed to low pressure stripping zone 40 via line 11 and then sent via line 14 to a gas cooler and flash zone and then to a furnace wherein it is burned as fuel. The hot NMP is passed via line 16 to pump 18 wherein it is pumped up to a gauge pressure ranging from about 1379 to 2068 kPa (200 to 300 psig) and more preferably from about 1379 to 1724 kPa (200 to 250 psig) and passed into line 20. Most of the hot solvent is then passed to a lube oil extraction tower (not shown) via line 22 while a slip stream comprising from about 5 to 15 wt.% of the total solvent coming from pump 18 is withdrawn via line 24 and passed through heat exchanger 26 wherein the hot NMP heats up an H₂S-rich NMP solution taken from the bottom of scrubbing zone 30. As a result of passing through heat exchanger 26 to the NMP is cooled to a temperature ranging from about 37.8 to 93.3°C (100 to 200°F) and is then passed through exchanger 28 via line 25 wherein it is cooled down to a temperature ranging from about 32.2 to 65.6°C (90 to 150°F) and more preferably from about 37.8 to 51.7°C (100 to 125°F). The cool NMP is then passed via line 29 into the top of scrubbing zone 30 which is a simple absorption column and may be provided with either packing or trays. The relatively cool NMP passes down through scrubbing zone 30 wherein it countercurrently contacts the gas to be used for stripping NMP from the extracted oil. This gas is a hydrofiner tail gas containing from about 0.5 to 15 volume % H₂S and is passed into the bottom of scrubbing zone 30 via line 32. In scrubbing zone 30, the NMP absorbs most of the H₂S from the gas to produce a scrubbed gas of low H₂S content (i.e., from about 10 to 2,000 ppm) and an H₂S-rich NMP solution which is removed from the bottom of zone 30 via line 36. The scrubbed gas at a temperature ranging from about 23.9 to 93.3°C (75 to 200°F) and at a gauge pressure of from about 689 to 2068 kPa (100 to 300 psig), is passed via line 34 to the solvent recovery section (not shown) of the lube oil extraction plant wherein it is used to strip residual amounts of NMP from the extracted oil. If complete removal of the H₂S is desired, the scrubbed gas may first be sent to further clean-up by known methods such as caustic scrubbing or zinc oxide treatments. The NMP passing down through scrubbing zone 30 is heated up to a very minor degree by the heat of absorption of the H₂S. The H₂S-rich NMP solution containing up to about 5 wt.% H₂S is taken from the bottom of zone 30 and passed to heat exchanger 26 via line 36. In heat exchanger 26 the H₂S-rich NMP solution is heated up to a temperature of from about 135.0 to 171.1°C (275 to 340°F) and more preferably from about 148.9 to 165.6°C (300 to 330°F) and passed

to stripping zone 40 via line 38 wherein it countercurrently contacts stripping gas introduced into zone 40 via line 11.

The H_2S is stripped out of solution as gas and removed from zone 40 via line 14 mixed with the stripping gas and then sent to a cool flash zone to condense and remove any NMP present in the gas. The H_2S -reduced NMP solution containing less than about 0.001 wt.% and more preferably less than about 0.0005 wt.% H_2S flows out of the bottom of zone 40 into solvent drum 12 via line 46 wherein it is combined with the condensed extraction solvent entering therein via line 10. The combined mixture is removed from drum 12 via line 16 and recycled back to both the extraction zone and the H_2S gas scrubbing zone. The NMP-containing stripping gas exiting zone 40 via line 14 after going to cool flash zone to condense and remove NMP and water present in the hot gas is then sent to lube oil extraction furnaces (not shown) wherein it is burned as fuel. The invention is further illustrated by reference to the following example.

Example

Referring to the figure, a combined stream of spent stripping gas comprising hydrogen and light hydrocarbon vapors, along with condensed NMP solvent containing about 3.8 wt.% water and 1.0 wt.% oil, at a temperature of 176.7°C (350°F) and a gauge pressure of 276 kPa (40 psig), is passed from the solvent recovery section (not shown) of a lube oil extraction plant employing NMP as the extraction solvent via line 10 into dry solvent surge drum 12 wherein the spent stripping gas disengages from the solvent solution and passes, via line 11, into the bottom of stripping zone 40 wherein it countercurrently contacts an H_2S -rich NMP solution in stripping zone 40 to strip most of the H_2S out of said solution, with the stripped NMP being passed to drum 12 via line 46 as H_2S -lean NMP. In zone 40, each mole of liquid H_2S -rich NMP contacts 2 moles of stripping gas. Drum 12 is at a temperature and gauge pressure of 176.7°C (350°F) and 276 kPa (40 psig), respectively and the spent stripping gas leaving the drum and entering stripping zone 40, via line 11, contains an equilibrium amount of gaseous NMP and water, which is determined by the conditions in drum 12. Hot NMP solution is withdrawn from 12 via line 16 wherein it passes through pump 18 and is pumped up to a gauge pressure of 1619 kPa (235 psig) and recycled back to the lube oil extraction tower (not shown) via lines 20 and 22. About 6.9 wt.% of the NMP passing through line 20 is withdrawn via line 24 and passed through heat exchanger 26 wherein it is cooled to 57.2°C (135°F) and from there via line 25 to heat exchanger 28 wherein it is further cooled to 37.8°C (100°F). The cool NMP solution at 37.8°C (100°F) and 1240 kPa gauge (180 psig) is passed via line 29 to the top of scrubbing tower 30 which is

provided with packing or trays. Tower 30 operates at a gauge pressure of 1240 kPa (180 psig) and 37.8°C (100°F). Tail gas from a hydrofiner at a temperature of 35.0°C (95°F) and a gauge pressure of 1240 kPa (180 psig) comprising mostly hydrogen along with minor amounts of light hydrocarbon vapors and containing 5 wt.% H_2S is passed into scrubber 30 via line 32 wherein it countercurrently contacts the cool NMP. The ratio of NMP to gas is about 1 to 1 on a molar basis. The NMP scrubs most of the H_2S from the tail gas to produce a scrubbed gas having less than 0.1 volume % H_2S which is then removed from the top of scrubber 30 via line 34 and sent to the solvent recovery section (not shown) of the lube oil extraction plant wherein it is used as stripping gas to remove residual amounts of NMP solvent from the extracted lube oil raffinate and extract. The H_2 -rich NMP solution is withdrawn from the bottom of scrubber 30 via line 36 and passed to heat exchanger 26 via line 36 wherein it is heated up to 165.6°C (330°F) by the hot NMP withdrawn from dry solvent drum 12 via line 16, pump 18 and lines 20 and 24. The hot, H_2S -rich NMP solution, at a temperature of 165.6°C (330°F) and a gauge pressure of about 276 kPa (40 psig), is then passed into stripping zone 40 which operates at a gauge pressure of 276 kPa (40 psig) wherein most of the H_2S is removed from the NMP to produce an H_2S -reduced NMP solution containing less than about 0.025 wt.% NMP which is then recycled back to drum 12 via line 46 wherein it is combined with the NMP solution entering said drum via line 10. The combined NMP in drum 12 contains an equilibrium amount of H_2S of about 0.02 wt.%. It should be noted that, in this example, all of the scrubbed tail gas used as stripping gas in the lube oil solvent recovery operation is recovered and passed through zone 40. On exiting zone 40, the H_2S -containing gas is cooled and flashed to remove NMP and water from the gas and then sent to lube oil extraction furnaces wherein it is burned as fuel.

Temperatures in °F are converted to °C by subtracting 32 and then dividing by 1.8.

Pressures in pounds per square inch gauge (psig) are converted to the gauge equivalent in kg/cm² by multiplying by 0.07031.

Claims

1. A process for removing H_2S from an H_2S -containing gas-stream comprising passing said gas stream (32) into a scrubbing zone (30) wherein it is contacted with liquid N-methyl-2-pyrrolidone (NMP) to remove most of the H_2S from said gas stream to form an H_2S -rich NMP solution (36), heating said H_2S -rich NMP solution in (26) by indirect heat exchange with a hot stream of fluid and passing the resulting heated solution (38) to a gas stripping zone (40) to remove most of the H_2S from said solution to form an H_2S -lean NMP solution (46), the process be-



to stripping zone 40 via line 38 wherein it countercurrently contacts stripping gas introduced into zone 40 via line 11.

The H_2S is stripped out of solution as gas and removed from zone 40 via line 14 mixed with the stripping gas and then sent to a cool flash zone to condense and remove any NMP present in the gas. The H_2S -reduced NMP solution containing less than about 0.001 wt.% and more preferably less than about 0.0005 wt.% H_2S flows out of the bottom of zone 40 into solvent drum 12 via line 46 wherein it is combined with the condensed extraction solvent entering therein via line 10. The combined mixture is removed from drum 12 via line 16 and recycled back to both the extraction zone and the H_2S gas scrubbing zone. The NMP-containing stripping gas exiting zone 40 via line 14 after going to cool flash zone to condense and remove NMP and water present in the hot gas is then sent to lube oil extraction furnaces (not shown) wherein it is burned as fuel. The invention is further illustrated by reference to the following example.

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ing characterized by the following combination of features: (a) the H_2S -lean NMP solution is combined in (12) with hot liquid NMP (10) obtained by recovering NMP from a mixture comprising hydrocarbon oil and NMP, formed by solvent extraction of a hydrocarbon oil with NMP, to form a hot combined solution (16); (b) a portion (24) of the combined solution is employed as the said hot stream of fluid which constitutes the sole heat source for heating in (26) the said H_2S -rich NMP solution before entering the stripping zone (40); and (c) after passing in indirect heat exchange relationship (26) with the H_2S -rich NMP solution, the said portion of the combined solution is recycled (25, 29) to the scrubbing zone (30).

2. The process of claim 1 characterized in that said scrubbed gas (34) is used to strip NMP from said extracted oil.

3. The process of claim 2 characterized in that said scrubbed gas, after being used to strip NMP from extracted oil, is passed (via 11) to said gas stripping zone (40) to remove most of the H_2S from said H_2S -rich NMP solution.

4. The process of claim 3 characterized in that said gas, after removing the H_2S from the H_2S -rich solution, is burned as fuel.

5. A process according to any one of claims 1 to 4 characterized by comprising the step of recycling (via 22) a portion of said combined solution back to said extraction zone.

6. The process of any one of claims 1 to 5 characterized in that said hydrocarbon oil is a lube oil stock.

7. The process of any one of claims 1 to 6 characterized in that said H_2S -containing gas (32) is a hydrofiner tail gas.

8. The process of any one of claims 1 to 7 characterized in that the hot liquid NMP (10) obtained by recovering NMP from the mixture comprising hydrocarbon oil and NMP is accompanied by spent stripping gas from the NMP recovery operation, and the spent stripping gas is passed through the gas stripping zone (40).

Patentansprüche

1. Verfahren zur Entfernung von H_2S aus einem H_2S enthaltenden Gasstrom, bei dem der Strom (32) in eine Waschzone (30) geleitet wird, in der er mit flüssigem N-Methyl-2-pyrrolidon (NMP) zur Entfernung des meisten H_2S aus dem Gasstrom unter Bildung einer H_2S -reichen NMP-Lösung (36) kontaktiert wird, die H_2S -reiche NMP-Lösung (in 26) durch indirekten Wärmeaustausch mit einem heißen Fluidstrom erhitzt wird und die resultierende erhitzte Lösung (38) in eine Gasabtriebszone (40) geleitet wird, um den meisten H_2S unter Bildung einer H_2S -armen NMP-Lösung (46) zu entfernen, gekennzeichnet durch die folgende Kombination von Merkmalen:

(a) die H_2S -arme NMP-Lösung wird (in 12) mit heißem Flüssigen NMP (10), das durch Gewinnung von NMP aus einer Mischung von

Kohlenwasserstofföl und NMP, die durch Lösungsmittelextraktion eines Kohlenwasserstofföls mit NMP hergestellt worden ist, erhalten worden ist, unter Bildung einer heißen kombinierten Lösung (16) kombiniert;

(b) ein Teil (24) der kombinierten Lösung wird als heißer Fluidstrom, der die einzige Wärmequelle für das Erhitzen (in 26) der H_2S -reichen NMP-Lösung vor Eintritt in die Abtriebszone (40) darstellt, verwendet; und

(c) nach dem indirekten Wärmeaustausch (26) mit der H_2S -reichen NMP-Lösung wird dieser Teil der kombinierten Lösung in die Waschzone (30) zurückgeführt (25, 29).

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß das gewaschene Gas (34) dazu verwendet wird, NMP aus dem extrahierten Öl zu entfernen.

3. Verfahren nach Anspruch 2, dadurch gekennzeichnet, daß das gewaschene Gas nach Verwendung zur Entfernung von NMP aus dem extrahierten Öl (über 11) in die Gasabtriebszone (40) geleitet wird, um den meisten H_2S aus der H_2S -reichen NMP-Lösung zu entfernen.

4. Verfahren nach Anspruch 3, dadurch gekennzeichnet, daß das Gas nach der Entfernung von H_2S aus der H_2S -reichen Lösung als Brennstoff verbrannt wird.

5. Verfahren nach jedem der Ansprüche 1 bis 4, dadurch gekennzeichnet, daß ein Teil der kombinierten Lösung (über 22) in die Extraktionszone zurückgeführt wird.

6. Verfahren nach jedem der Ansprüche 1 bis 5, dadurch gekennzeichnet, daß das Kohlenwasserstofföl ein Schmieröl ist.

7. Verfahren nach jedem der Ansprüche 1 bis 6, dadurch gekennzeichnet, daß das H_2S enthaltende Gas (32) ein Hydrofiningendgas ist.

8. Verfahren nach jedem der Ansprüche 1 bis 7, dadurch gekennzeichnet, daß das heiße flüssige NMP (10), das durch Gewinnung von NMP aus der Mischung aus Kohlenwasserstofföl und NMP erhalten worden ist, von verbrauchtem Abtriebsgas aus der NMP-Gewinnungsstufe begleitet wird und das verbrauchte Abtriebsgas durch die Gasabtriebszone (40) geleitet wird.

Revendications

1. Procédé pour éliminer H_2S d'un courant de gaz contenant du H_2S , consistant à faire passer ce courant (32) dans une zone de lavage (30) dans laquelle il est mis en contact avec de la N-méthyl-2-pyrrolidone (NMP) liquide pour éliminer la plus grande partie du H_2S du courant gazeux pour former une solution de NMP (36) riche en H_2S , à chauffer ladite solution de NMP riche en H_2S (en 26) par échange de chaleur indirect avec un courant chaude de fluide et à faire passer la solution chauffée obtenue (38) dans une zone d'épuration des gaz (40) pour éliminer la plus grande partie du H_2S de cette solution pour former une solution de NMP (46) pauvre en H_2S , procédé caractérisé par la combinaison suivante de caractéristiques: (a) la



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solution de NMP pauvre en H_2S est combinée (en 12) avec de la NMP liquide chaude (10) obtenue en récupérant la NMP d'un mélange comprenant une huile hydrocarbonée et de la NMP, formé par une extraction aux solvants d'une huile hydrocarbonée avec de la NMP, pour former une solution combinée chaude (16); (b) une portion (24) de la solution combinée est employée en tant que courant chaud de fluide, qui constitue la source de chaleur unique pour chauffer (en 26) ladite solution de NMP riche en H_2S avant son introduction dans la zone d'épuration (40); et (c) après passage dans une relation d'échange de chaleur indirect (26) avec la solution de NMP riche en H_2S , ladite portion de la solution combinée est recyclée (25, 29) vers la zone d'épuration (3).

2. Procédé selon la revendication 1, caractérisé en ce que le gaz lavé (34) est utilisé pour séparer la NMP de l'huile extraite.

3. Procédé selon la revendication 2, caractérisé en ce que le gaz lavé, après avoir été utilisé pour séparer la NMP de l'huile extraite, est envoyé (par l'intermédiaire de 11) à la zone d'épuration des gaz (40) pour éliminer de la solution de NMP riche en H_2S la plus grande quantité de H_2S .

4. Procédé selon la revendication 3, caractérisé en ce que le gaz, après élimination du H_2S de la solution riche en H_2S , est brûlé en tant que combustible.

5. Procédé selon l'une quelconque des revendications 1 à 4, caractérisé en ce qu'il comprend l'étape consistant à recycler (par l'intermédiaire de 22) une portion de la solution combinée, renvoyée à la zone d'extraction.

6. Procédé selon l'une quelconque des revendications 1 à 5, caractérisé en ce que l'huile hydrocarbonée est une huile usuelle lubrifiante.

7. Procédé selon l'une quelconque des revendications 1 à 6, caractérisé en ce que le gaz (32) contenant du H_2S est un gaz de queue d'une unité d'hydrotraitement.

8. Procédé selon l'une quelconque des revendications 1 à 7, caractérisé en ce que la NMP liquide chaude (10) obtenue en récupérant la NMP d'un mélange comprenant une huile hydrocarbonée et de la NMP est accompagnée d'un gaz d'épuration usé provenant de l'opération de récupération de la NMP, et que le gaz d'épuration usé est envoyé à travers la zone d'épuration des gaz (40).

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