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(54) Title: A METHOD OF REMOVING NITROUS OXIDE

(57) Abstract: Hydroxylamine is formed in a reactor through a partial hydrogenation of nitric oxide gas (NO) with hydrogen gas (H₂) in an aqueous medium with nitrogen gas (N₂) as an inert gas. The formation of the hydroxylamine forms nitrous oxide gas (N₂O). The gases and the water vapor are flowed away from the reactor in a vent gas stream enabling recycling of the gases. The N₂O is removed from the vent gas stream to reduce flammability. Once the N₂O is removed, the NO, H₂, and N₂ are recycled and re-used in the reactor to form additional hydroxylamine. The N₂O removed from the vent gas stream can be commercially sold or economically discarded.

A METHOD OF REMOVING NITROUS OXIDE

FIELD OF THE INVENTION

[001] The present invention generally relates to a method of removing nitrous oxide. More specifically, the present invention relates to a method of using pressure swing adsorption and a nitrous oxide adsorbent material for removing the nitrous oxide in a vent gas formed from a synthesis of hydroxylamine and recycling remaining gases in the vent gas.

BACKGROUND OF THE INVENTION

[002] Nitrous oxide gas, (N_2O), is produced as a by-product in a synthesis of hydroxylamine. Hydroxylamine may be used to form a caprolactam that can be used to form nylon 6. Nylon 6 is an important polymer that is used throughout the world in carpets, apparel, upholstery, auto parts, and in many other products.

[003] Typically, hydroxylamine is formed from partially hydrogenating nitric oxide gas (NO) with hydrogen gas (H_2) in an aqueous medium including nitrogen gas (N_2) as an inert gas. Mixing the NO with the H_2 and the N_2 forms hydroxylamine in addition to a variety of by-products. The by-products include N_2O . The N_2O , in addition to unused N_2 , H_2 , and NO along with water vapor (H_2O), may be evacuated from the hydroxylamine as a moist gas mixture that may be disposed of or recycled. The gas mixture may also include contaminants including methane, carbon dioxide (CO_2), and carbon monoxide (CO). The gas mixture may be recycled along with H_2 and NO to reduce overall consumption of H_2 and NO.

[004] However, if the gas mixture and the H_2 and NO are recycled, a level of the N_2O can accumulate in the gas mixture to dangerous levels. If the level of N_2O in the gas mixture becomes too high, the gas mixture becomes flammable and unsafe to handle. If the level of N_2O reaches a predetermined upper limit, the gas mixture is discarded, resulting in loss of H_2 , and NO remaining in the gas mixture. Removal of N_2O from the gas mixture would preferably prevent premature disposal of the gas mixture along with H_2 and NO thereby allowing recycling of the H_2 and NO and reducing overall consumption.

[005] Adsorption can provide a more efficient and economic means for separating gases than use of cryogenic distillation, absorption, or membrane-based systems. Adsorption can be used to separate a gas from the gas mixture that includes at least two gases that have different adsorption characteristics toward an adsorbent material.

[006] Typically, adsorption generally includes a synchronized cycling of a pressure or temperature of one or a plurality of adsorbent beds. The adsorbent bed includes the adsorbent material that preferentially adsorbs one or more gases present in the gas mixture. Pressure swing adsorption (PSA) generally includes a series of steps that change the pressure and/or flow direction of the gas mixture to achieve separation of a preferred gas from a series of non-preferred gases. The number and nature of the steps involved in PSA may vary based on separation objectives.

[007] More specifically, PSA typically includes flowing the gas mixture from a feed inlet of the adsorbent bed to a discharge end of the adsorbent bed, at an adsorption pressure. A gas in the gas mixture that has strong adsorption characteristics preferentially adsorbs onto the adsorbent material, while a gas with weak adsorption characteristics continues to flow through the adsorbent bed. As such, the gas mixture flowing out of the discharge end of the adsorbent bed is substantially depleted of the gas preferentially adsorbed onto the adsorbent material.

[008] As the gas with the strong adsorption characteristics flows from the feed inlet to the discharge end, the gas with the strong adsorption characteristics accumulates on the adsorbent material closest to the feed inlet. As the accumulation proceeds, the adsorbent material closest to the feed inlet becomes saturated first, followed by saturation of the entire adsorbent material in the direction of flow of the gas mixture.

[009] Eventually, once the entire adsorbent material becomes saturated, the gas with the strong adsorption characteristics breaks through the discharge end. Usually, the flow of the gas mixture is stopped before breakthrough occurs. Once the flow of the gas mixture is stopped, the gas with the weak adsorption characteristics may be removed without excessively desorbing the gas adsorbed onto the adsorbent material. After the adsorbent pressure is reduced, the adsorbent bed is further depressurized and the gas adsorbed onto the adsorbent material is removed from the bed. The adsorbent bed is then purged and re-pressurized to the adsorption pressure with the gas having the weak adsorption characteristics. Once this occurs, the adsorbent bed may be reused.

[0010] The adsorbent materials that may be used with PSA are dependent on the gas to be adsorbed, the gas mixture itself, and other factors that are well known to those skilled in the art. In general, suitable adsorbent materials include zeolite molecular sieves, silica gel, activated carbon, and activated alumina. For certain applications, specialized adsorbent materials can be used.

[0011] The efficiency of PSA depends on a variety of parameters including pressures, temperatures, volumes, and flow rates of gases in PSA systems, time of the synchronized pressure cycling, types, sizes, and shapes of the adsorbent materials, dimensions of the adsorbent beds, and compositions of feed, product, purge, and other intermediate gas streams. Variations in these parameters can influence the cost and productivity of the PSA systems.

[0012] Conventional PSA systems can remove N_2O from the gas mixture. However, conventional PSA methods known in the art potentially form flammable gas mixtures of N_2O , H_2 , and NO and many adsorbents do not exhibit a high selectivity for the N_2O as compared to the H_2O in the gas mixture. As such, there remains an opportunity to develop a PSA method that effectively separates N_2O from H_2O in the gas mixture such that N_2O can be removed from the gas mixture using a minimum number of PSA systems and the N_2 , H_2 , and NO can be efficiently recycled.

BRIEF SUMMARY OF THE INVENTION AND ADVANTAGES

[0013] The present invention provides a method of removing nitrous oxide from a vent gas. In a first embodiment of the method, the vent gas includes the nitrous oxide and at least one other gas. The method includes the step of flowing the vent gas through a nitrous oxide adsorbent material. The vent gas is flowed through the nitrous oxide adsorbent material such that the nitrous oxide is adsorbed onto the nitrous oxide adsorbent material. The method also includes the step of flowing nitrogen through the nitrous oxide adsorbent material to displace the other gas. The method further includes desorbing the nitrous oxide from the nitrous oxide adsorbent material.

[0014] In a second embodiment of the method, the method includes the step of synthesizing hydroxylamine thereby producing the vent gas. The second embodiment also includes the steps of flowing the vent gas through the nitrous oxide adsorbent material such that the nitrous oxide is adsorbed onto the nitrous oxide adsorbent material, flowing the nitrogen through the nitrous oxide adsorbent material to displace

the other gas, and desorbing the nitrous oxide from the nitrous oxide adsorbent material, as in the first embodiment.

[0015] In a third embodiment of the method, the method includes the step of adsorbing the nitrous oxide from a vent gas onto a nitrous oxide adsorbent material. The third embodiment also includes the step of flowing nitrogen into the nitrous oxide adsorbent material. The third embodiment further includes the step of desorbing the nitrous oxide from the nitrous oxide adsorbent material.

[0016] Accordingly, an effective method of removing nitrous oxide from vent gases is established. Flowing the nitrogen preferably displaces any of the vent gas, e.g., gas including hydrogen and other flammable gasses, from the nitrous oxide adsorbent material and allows the nitrous oxide to be removed from the nitrous oxide adsorbent material such that flammability is reduced.

BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWINGS

[0017] Other advantages of the present invention will be readily appreciated, as the same becomes better understood by reference to the following detailed description when considered in connection with the accompanying drawings wherein:

[0018] Figure 1 is schematic view of an embodiment of the present invention generally illustrating a method wherein first, second, and third vent gases flow through a reactor, a removal unit, a first pressure swing adsorption (PSA) system, and a second PSA system;

[0019] Figure 2 is a schematic view of an embodiment of the present invention that is exemplary of the first PSA system that includes two first adsorbent beds;

[0020] Figure 3 is a schematic view of an embodiment of the present invention that is exemplary of the first and second PSA systems; and

[0021] Figure 4 is a schematic view of an embodiment of the present invention that is exemplary of the second PSA system that includes two second adsorbent beds.

DETAILED DESCRIPTION OF THE PRESENT INVENTION

[0022] A method of removing nitrous oxide gas (N_2O) from a vent gas including the N_2O and at least one other gas is provided. It is to be understood that the vent gas may include any gas or gas stream including the N_2O . The vent gas, hereafter, will be referred to as a third vent gas (3), as the method of the present invention preferably utilizes first, second, and third vent gases (1, 2, 3). However, it is to be understood that the first and the second vent gases (1, 2) need not be utilized and are optional.

[0023] A first embodiment of the present invention includes the step of flowing the third vent gas (3) through a nitrous oxide adsorbent material (67). Typically, there are void spaces between the nitrous oxide adsorbent material (67), which will also be described in greater detail below. The third vent gas (3) is flowed through the nitrous oxide adsorbent material (67) such that the N_2O is adsorbed onto the nitrous oxide adsorbent material (67). The first embodiment also includes the step of flowing nitrogen gas (N_2) (12) through the nitrous oxide adsorbent material (67) to displace the other gas and possibly reduce or prevent formation of flammable compositions resulting from intermingling of gasses. The first embodiment still further includes the step of desorbing the N_2O from the nitrous oxide adsorbent material (67). It is also contemplated that the first embodiment may include the step of flowing the nitrogen gas (12) through the nitrous oxide adsorbent material (67) at a reduced pressure, to displace the nitrous oxide retained in the void spaces of the nitrous oxide adsorbent material (67).

[0024] The third vent gas (3) includes at least one other gas. Typically, the other gas includes, but is not limited to, hydrogen gas (H_2), the N_2O , nitric oxide gas (NO), N_2 , and water vapor (H_2O), in addition to other gases such as methane, and trace gases such as carbon dioxide (CO_2) and carbon monoxide (CO). It is contemplated that the N_2O present in the third vent gas (3) may be formed as a by-product from a synthesis of hydroxylamine. The methane, CO_2 and CO may also be present as a result of the H_2 or NO that may be present in the third vent gas (3) possibly as a consequence of side-reactions in the synthesis of hydroxylamine.

[0025] Typically, the N_2O is present in the third vent gas (3) in an amount of from 3 to 15, and more typically of from 5 to 11, mol %. Also, the H_2 is typically present in the third vent gas (3) in an amount of at least 40 mol % and more typically of at least 60 mol %. Further, the NO and the N_2 are typically each present in the third vent gas (3) in an amount of less than 30 and more typically of less than 25, mol %, each. Still further, the H_2O may be present in the third vent gas (3) in any amount and typically in an amount that saturates the third vent gas (3) at a temperature of from 30-60 °C at a pressure of greater than 2 bar absolute. In one embodiment, the pressure is 29 psia. Typically, methane may be present in the third vent gas (3) in an amount of from 0.1 to 3.0 mol % while CO_2 and CO may be present in an amount of from 10 to 100 parts per one million parts of the third vent gas (3), each.

[0026] Hydroxylamine may be formed in a reactor (26) from partially hydrogenating NO with H₂ in an aqueous medium including N₂ as an inert gas. Mixing the NO with the H₂ and the N₂ forms hydroxylamine in addition to a variety of by-products. The by-products include N₂O. The N₂O, in addition to unused N₂, H₂, and NO along with H₂O, may be evacuated from the reactor (26) as the first vent gas (1), first introduced above. The first vent gas (1) may also include contaminants including methane, CO₂ and CO. The H₂O may be removed from the first vent gas (1) thereby forming the second and third vent gases (2, 3), discussed in greater detail below. Ultimate removal of the N₂O from the third vent gas (3) reduces safety and environmental hazards associated with flammability. Once removed, the N₂O can be used as needed in other applications, commercially sold, or economically discarded. In the instant invention, the N₂O that is removed from the vent gas may be of any purity. More specifically, after removal and when isolated, the N₂O may be measured to determine a level of impurities present as compared to a total amount of the N₂O being measured. In one embodiment, the N₂O is removed in approximately 50 percent purity, i.e., the N₂O removed includes approximately 50% of N₂O molecules and approximately 50% of molecules of other gasses which are considered impurities. In another embodiment, the N₂O is removed in approximately 25 to 30 percent purity. Additionally, the N₂O may be removed in any percentage relative to the original amount of N₂O present in the vent gas. Specifically, the method of the instant invention may remove up to 100% of the N₂O present in the vent gas, and thereby exhibit complete efficiency in removal of the N₂O. However, depending on the parameters of the method, the efficiency may not be complete and may be less than 100%.

[0027] The first embodiment of the present invention may be completed using a variety of adsorption techniques including, but not limited to, pressure swing adsorption (PSA) techniques, any other adsorption techniques known in the art, and combinations thereof. Preferably, the steps of the first embodiment are completed utilizing PSA techniques.

[0028] However, before the aforementioned steps of the first embodiment are performed, it is contemplated that the first vent gas (1) may be treated, as first introduced above. Preferably, if the first vent gas (1) includes the H₂O, the first vent gas (1) may be treated to remove the H₂O in two contents including a first content (4)

and a second content (5), respectively. Preferably, the first content is removed using a removal unit (27), thereby forming the second vent gas (2) that includes some residual H₂O. Preferably, the second content (5) is removed using a first PSA system (29), thereby forming the third vent gas (3) that includes a minimal amount of H₂O. In one embodiment, a minimal amount of H₂O preferably includes an amount of H₂O present in the third vent gas (3) of from 1 to 1,000, more preferably of from 10 to 100, even more preferably of from 10 to 50, still more preferably of from 15 to 40, and most preferably of from 20 to 25, parts of H₂O per one million parts of the third vent gas (3). In another embodiment, the amount of H₂O present in the third vent gas (3) is preferably of from 1 to 5,000, more preferably of from 5 to 500, and most preferably of from 10 to 100, parts of H₂O per one million parts of the third vent gas (3). It is to be understood that the amount of H₂O present in the third vent gas (3) may depend on product specifications of the nitrous oxide.

[0029] The first content (4) of the H₂O may be substantially removed from the first vent gas (1) through use of the removal unit (27), first introduced above. In a preferred embodiment of the present invention, the removal unit (27) is in fluid communication with the reactor (26). The first vent gas (1) may be flowed from the reactor (26) into the removal unit (27). The removal unit (27) may include any apparatus that functions to remove the first content of the H₂O from the first vent gas (1). Suitable examples of the first removal unit (27) include, but are not limited to, coolers, condensers, refrigerated dryers, gas scrubbers, and combinations thereof. Preferably, the first removal unit (27) is a refrigerated dryer and removes the first content (4) of the H₂O from the first vent gas (1) as liquid water. It is desirable to remove H₂O from the first vent gas (1) because a low content of the H₂O in the first vent gas (1) allows for a more effective adsorption of the N₂O onto the nitrous oxide adsorbent material (67). The first vent gas (1) preferably flows into the first removal unit (27) at a pressure of from 20 to 90, more preferably of from 25 to 50, and most preferably of from 30 to 40, psia, at a temperature of from 25 to 60°C.

[0030] If the first removal unit (27) includes the refrigerated dryer, the refrigerated dryer may include a shell and a refrigeration system disposed within the shell. The refrigeration system may be generally defined by a housing including a moisture separator (43), a sump, a discharge conduit, and a drain to evacuate the liquid water from the refrigerated dryer.

[0031] An evaporator may also be included in the housing of the refrigerated dryer. The first vent gas (1), flowing through the housing, may pass through the evaporator and be cooled to effect the removal of the first content (4) of the H₂O from the first vent gas (1) thereby forming the second vent gas (2), as first introduced above. The first vent gas (1) may be cooled to a dew point such that the first content (4) of the H₂O in the first vent gas (1) condenses to form the liquid water. In one embodiment, the first vent gas (1) is preferably cooled to a dew point of from 5 to 30°C, more preferably of from 8 to 16°C, and most preferably of from 10 to 14°C. In another embodiment, the first vent gas (1) is preferably cooled to a dew point of from 0 to 30°C, more preferably of from 5 to 20°C, and most preferably of from 5 to 15°C.

[0032] After being cooled to the dew point, the first vent gas (1) may subsequently flow through the moisture separator (43). The moisture separator (43) preferably removes the first content of the H₂O from the refrigerated dryer. The liquid water may flow from the moisture separator (43) through the discharge conduit to the sump. The sump preferably pumps the liquid water to the drain and evacuates the liquid water from the refrigerated dryer.

[0033] If the second vent gas (2) is formed, the second vent gas (2) preferably exits the removal unit (27) through a removal outlet. The removal outlet may be in fluid communication with an inlet (33) of the first PSA system (29). The first PSA system (29) may be used to remove the second content (5) of the H₂O and form the third vent gas (3), as seen in Figures 1 and 2. The second vent gas (2) preferably enters the first PSA system (29) through the inlet (33) and exits through a first discharge end (35), e.g., a first discharge valve. Preferably, the first PSA system (29) includes two first discharge ends (35).

[0034] The first PSA system (29) also preferably includes a first adsorbent bed (30). It is contemplated that the first PSA system (29) may include a plurality of first adsorbent beds (30), as seen in Figure 2. More preferably the first PSA system (29) includes two first adsorbent beds (30). Preferably, the first adsorbent bed (30) includes a water adsorbent material (31) that preferentially adsorbs H₂O as compared to other gases in the second vent gas (2). The first PSA system (29) also preferably includes a dry gas product tank (32), fluid movers (not shown,) valves, and instrumentation (not shown) that may be selected by one skilled in the art.

[0035] Once in the first PSA system (29), the second vent gas (2) preferably flows into the first adsorbent bed (30) at a first adsorption pressure, and for a first pressurization time, from the inlet (33). Preferably, the first adsorption pressure includes a pressure of from 20 to 90, more preferably of from 25 to 50, and most preferably of from 30 to 40, psia.

[0036] As the second vent gas (2) flows through the first adsorbent bed (30), the H₂O preferably adsorbs onto and saturates the water adsorbent material (31) closest to the inlet (33) for a first adsorption time. Preferably, the water adsorbent material (31) includes, but is not limited to, hydrophilic adsorbents, hydrophobic adsorbents, polymeric adsorbents, activated carbon, carbon molecular sieve adsorbents, activated alumina, silica gel, zeolites including those with type A, X, Y, ZSM-5, silicalite, mordenite, desiccants, and combinations thereof. More preferably, the water adsorbent material (31) includes various grades of silica gel and alumina. Most preferably, the water adsorbent material (31) includes a silica gel commercially available from Grace Davison of Columbia, MD, under the trade name Davison 03. The silica gel has a high selectivity for the H₂O over the N₂, the H₂, NO, and N₂O thereby increasing an efficiency of removing the H₂O from the second vent gas (2). However, it is contemplated that the silica gel may also adsorb some of the N₂O from the second vent gas (2). If this occurs, the N₂O may be separated from the H₂O with additional adsorption and desorption techniques. The other gases such as CO₂ may also be removed from the second vent gas (2) using any adsorption method known in the art.

[0037] As the H₂O preferably adsorbs onto the water adsorbent material (31), the other gases in the second vent gas (2) such as H₂, NO, N₂O, and N₂, preferably flow out of the first discharge end (35) of the first adsorbent bed (30) and the third vent gas (3) is formed, as first introduced above. After leaving the discharge end (35) of the first adsorbent bed (30), the third vent gas (3) preferably continues to flow (3) into the dry gas product tank (32) preferably in fluid communication with the first discharge end (35) of the first adsorbent bed (30).

[0038] If the water adsorbent material (31) closest to the inlet (33) becomes saturated, the H₂O preferably adsorbs onto the water adsorbent material (31) immediately next to the saturated water adsorbent material (31) in a direction towards the first discharge end (35). If all of the water adsorbent material (31) from the inlet (33) to the first

discharge end (35) becomes saturated, the H₂O may breakthrough the first adsorbent bed (30). Preferably, flow of the second vent gas (2) is stopped before breakthrough of the H₂O from the first adsorbent bed (30).

[0039] Once the flow of the second vent gas (2) is stopped, the first adsorbent bed (30) is preferably counter-currently depressurized to a pressure below that first adsorbent pressure for a first blowdown time. It is to be understood that "counter-currently" includes a flow of a gas in an opposite direction of a flow of the second vent gas (2) (e.g., within the first adsorbent bed (30)).

[0040] If the third vent gas (3) flows into the dry gas product tank (32), the third vent gas (3) may be used to further counter-currently de-pressurize and/or re-pressurize and purge the first adsorbent bed (30), at a first desorption pressure and a purge flow rate, to assist a desorption of the H₂O from the water adsorbent material, thereby regenerating the first adsorbent bed (30). Specifically, the first discharge end (35) and inlet (33) may be closed and a valve (36) and the purge valve (37) may be opened to allow the third vent gas (3) to flow through and out of the first adsorbent bed (30) and into the moisture separator (43). If the third vent gas (3) is used to further counter-currently de-pressurize and/or re-pressurize and purge the first adsorbent bed (30), the first desorption pressure preferably includes a pressure of from 0.5 to 30, and more preferably from 1 to 15, psia. It is also contemplated that a variety of intermediate con-current depressurization steps and purges may be utilized to improve formation of the third vent gas (3). It is contemplated that the first adsorbent bed (30) may be purged with additional nitrogen (12) to improve formation of the third vent gas (3).

[0041] Preferably, after entering the dry gas product tank (32), the third vent gas (3) enters into a second PSA system (65). Preferably, the second PSA system (65) is similar to the first PSA system (29) including similar fluid movers, valves and instrumentation that are known in the art. However, it is also contemplated that the second PSA system (65) may be different from the first PSA system (29). The second PSA system (65) preferably includes the second adsorbent bed (66), and it is contemplated that the second PSA system (65) may include a plurality of second adsorbent beds (66). More preferably, the second PSA system (65) includes two second adsorbent beds (66), as seen in Figure 4. Preferably, the second adsorbent bed (66) includes the nitrous oxide adsorbent material (67). However, it is to be understood that the nitrous oxide adsorbent material (67) may be disposed in any

vessel known in the art. The second PSA system (65) also may include a light product or N₂O lean product tank (71), an N₂O product tank (72), a recycle tank (73), and fluid movers, valves, and instrumentation that are known in the art.

[0042] Preferably, the third vent gas (3) enters the second PSA system (65) through an inlet valve (A) at a second adsorption pressure and for a second pressurization time, and exits through a second discharge end (B), e.g., a second discharge valve. In one embodiment, the second adsorption pressure preferably includes a pressure of from 10 to 75, more preferably of from 15 to 40, and even more preferably of from 15 to 30, psig. Most preferably, in this embodiment, the second adsorption pressure is 15 psig. In another embodiment, the second adsorption pressure preferably includes a pressure of from 25 to 90, more preferably of from 30 to 55, and even more preferably of from 30 to 45, psia. In this embodiment, the second adsorption pressure is most preferably 30 psia.

[0043] As the third vent gas (3) flows through the second adsorbent bed (66), the N₂O preferably adsorbs onto and saturates the nitrous oxide adsorbent material (67) closest to the inlet valve (A) for a second adsorption time. The nitrous oxide adsorbent material (67) preferentially adsorbs N₂O over H₂, NO, N₂, other gases and trace components present in the third vent gas (3). The first embodiment of the present invention includes the step of flowing the third vent gas (3) through the nitrous oxide adsorbent material (67), e.g., through the void spaces, such that the N₂O adsorbs onto the nitrous oxide adsorbent material. It is contemplated that the first embodiment may also include the step of flowing the third vent gas (3) through the void spaces of the nitrous oxide adsorbent material.

[0044] Preferably, the nitrous oxide adsorbent material (67) includes, but is not limited to, hydrophilic adsorbents, hydrophobic adsorbents, polymeric adsorbents, activated carbon, carbon molecular sieve adsorbents, activated alumina, silica gel, zeolites including those with type A, X, Y, ZSM-5, silicalite, mordenite, and combinations thereof. More preferably the nitrous oxide adsorbent material (67) includes 5A zeolite, ZSM-5, and LiX. Most preferably, the nitrous oxide adsorbent material (67) includes a hydrophobic zeolite molecular sieve currently commercially available from UOP LLC of Des Plaines Il, under the trade name of HISIV-3000. Use of the HISIV-3000 allows for efficient adsorption and removal of the N₂O from the third vent gas (3).

[0045] If the N_2O preferably adsorbs onto the nitrous oxide adsorbent material (67), the other gases in the third vent gas (3) such as H_2 , NO , and N_2 , other gases and trace components, known as a multi-component gas, preferably flow out of the second discharge end (B) of the second adsorbent bed (66). Preferably, the multi-component gas flowing out of the second discharge end (B) flows into the light product tank (71). It is also contemplated that the multi-component gas may be used to purge the second adsorbent bed (66) for an N_2O lean purge time.

[0046] If the nitrous oxide adsorbent material (67) closest to the inlet valve (A) becomes saturated, the N_2O preferably adsorbs onto nitrous oxide adsorbent material (67) immediately next to the saturated nitrous oxide adsorbent material (67) in a direction towards the second discharge end (B). If all of the nitrous oxide adsorbent material (67) from the inlet valve (A) to the second discharge end (B) becomes saturated, the N_2O may breakthrough. Preferably, flow of the third vent gas (3) is stopped before breakthrough of the N_2O .

[0047] The first embodiment of the present invention also includes the step of flowing N_2 (12) through the nitrous oxide adsorbent material (67) to displace the other gas. Preferably, the nitrous oxide adsorbent material (67) is isolated from the flow of the third vent gas (3) and/or effluent gas during depressurization and prior to and/or during the steps of flowing the N_2 (12) through the nitrous oxide adsorbent material (67). Preferably, the N_2 (12) is flowed into the second adsorbent bed (66) through a first gas valve (C) at the second adsorption pressure, for an N_2 pressurization time, and at an N_2 pressurization rate (also known as an N_2 purge rate). Most preferably, the first embodiment includes the step of flowing the N_2 (12) counter-currently to the flow of the third vent gas (3) in the second adsorbent bed (66) after the nitrous oxide adsorbent material (67) is isolated from the flow of the third vent gas (3) and after depressurization of the second adsorbent bed (66). Preferably, the N_2 (12) is flowed to displace any third vent gas (3) and/or any nitrous oxide in the void spaces of the nitrous oxide adsorbent material (67), away from the second discharge end (B) of the second adsorbent bed (66). Without intending to be bound by any particular theory, it is believed that through displacing the third vent gas (3) in the void spaces, the N_2O may be desorbed from the nitrous oxide adsorbent material (67) without forming a flammable mixture with the residual third vent gas (3). If the third vent gas (3) in the void spaces is displaced away from the second discharge end (B), a first effluent,

leaving inlet valve (A) or a fifth gas valve (E), is preferably produced. This first effluent may be recycled to back into the third vent gas (3) or sent to the recycle tank (73).

[0048] The first embodiment also includes the step of desorbing the N_2O from the nitrous oxide adsorbent material (67). It is contemplated that, after the flow of the N_2 is stopped, a pressure of the second adsorbent bed (66) may be lowered through counter-current depressurization to a second desorption pressure for a second blowdown time to desorb the N_2O at a desorption rate and for a second desorption time, thereby producing a second effluent. The second effluent preferably flows out of the second adsorbent bed (66) via a third gas valve (F) and into the N_2O product tank (72) via appropriate fluid movers (76), valves, and instrumentation that are well known in the art. The second effluent may include amounts of less than or equal to 100 mol % N_2O , less than or equal to 90 mol % N_2 , less than or equal to 5 mol % NO, and less than or equal to 5 mol % H_2 . In one embodiment, the second effluent includes of from 7 to 100 mol % N_2O , 50 to 90 mol % N_2 , 1 to 5 mol % NO, and 1 to 5 mol % H_2 . It is to be understood that any method known in the art may be used to desorb the N_2O . In one embodiment, the second desorption pressure preferably includes a pressure of from 0.5 to 10, more preferably of from 0.5 to 6, and even more preferably of from 0.5 to 3, and still more preferably of from 0.5 to 1.5, psia. Most preferably, in this embodiment, the second desorption pressure is from 1 to 1.5 psia. In another embodiment, second desorption pressure preferably includes a pressure of from 0.1 to 15, more preferably of from 0.5 to 10, and even more preferably of from 1 to 3, psia. Most preferably, in this embodiment, the second desorption pressure is 1.5 psia.

[0049] Additionally, after the N_2O is desorbed from the nitrous oxide adsorbent material (67), the N_2 (12) may again be flowed through the nitrous oxide adsorbent material (67). Also, after the N_2O is desorbed from the nitrous oxide adsorbent material (67), the multi-component gas from the light product tank (71) may be flowed through the nitrous oxide adsorbent material (67) via a fourth gas valve (G) and/or a fifth gas valve (E). It is contemplated that after a suitable purge is completed, the fifth gas valve (E) may be closed and the multi-component gas from the light product tank (71) may be allowed to flow into first adsorbent bed (30) via the fourth gas valve (G) to re-pressurize the first adsorbent bed (30). Most preferably, the

multi-component gas stream is flowed through the nitrous oxide adsorbent material (67) after the N₂ is flowed through the nitrous oxide adsorbent material (67). It is also contemplated that the multi-component gas may be flowed through the nitrous oxide adsorbent material (67) and through the void spaces. Preferably, the multi-component gas is flowed counter-currently to the third vent gas (3). If the multi-component gas is flowed through the nitrous oxide adsorbent material (67), the N₂ (12) is preferably displaced from the void spaces in a third effluent such that additional amounts of the third vent gas (3) can be utilized. Preferably, the third effluent flows via the fifth gas valve (E) to a recycle tank (73) via appropriate fluid movers (76), valves and instrumentation that are well known in the art. Preferably, flow of the third effluent is stopped once the N₂ (12) is displaced from the void spaces. It is also contemplated that a variety of intermediate con-current and/or counter-current depressurization steps and purges may be utilized to improve recovery or purity of the N₂O present in the second effluent.

[0050] It is also contemplated that a plurality of additional PSA systems (50, 52) may be used in the present invention to increase a purity of the N₂O present in the second effluent, as seen in Figure 3. The additional PSA systems (50, 52) may be the same as the second PSA systems (65) or may be different. However, it is preferred that each of the additional PSA systems (50, 52) operate in a similar manner as the second PSA system (65). Specifically, each of the additional PSA systems (50, 52) preferably includes a second nitrous oxide adsorbent material. Preferably, the second nitrous oxide adsorbent material is the same as the nitrous oxide adsorbent material (67). However, the second nitrous oxide adsorbent material may be different from the nitrous oxide adsorbent material (67).

[0051] In a second embodiment of the method, the method includes the step of synthesizing hydroxylamine thereby producing the third vent gas (3). It is to be understood that the hydroxylamine may be synthesized by any method known in the art. The second embodiment also includes the steps of the first embodiment including the step of flowing the third vent gas (3) through the nitrous oxide adsorbent material (67) such that the N₂O is adsorbed onto the nitrous oxide adsorbent material (67). The second embodiment also includes the step of flowing the N₂ through the nitrous oxide adsorbent material (67) to displace the other gas. The second embodiment further includes the step of desorbing the N₂O from the nitrous oxide adsorbent

material (67). Preferably, the N₂O is desorbed by lowering a pressure of the second adsorbent bed (66) through counter-current depressurization. It is to be understood that the second embodiment may also include any and/or all of the steps of the first embodiment.

[0052] In a third embodiment of the method, the method includes the step of adsorbing the N₂O from the third vent gas (3) onto the nitrous oxide adsorbent material (67). It is contemplated that the N₂O may be adsorbed onto the nitrous oxide adsorbent material (67) by any method known in the art including, but not limited to, passing the N₂O over, through, or around the nitrous oxide adsorbent material (67).

[0053] The third embodiment further includes the step of flowing N₂ into the nitrous oxide adsorbent material (67). The third embodiment still further includes the step of desorbing the N₂O from the nitrous oxide adsorbent material (67), as in the first and second embodiments. It is to be understood that the third embodiment may also include any and/or all of the steps of the first and second embodiments. It is contemplated that in each of the PSA systems, the adsorbent beds may be re-pressurized at any point in the method, as determined by one skilled in the art depending on the desired results of the method.

EXAMPLES

[0054] Hydroxylamine is formed in a reactor (26) by combining nitric oxide gas (NO), nitrogen gas (N₂), and hydrogen gas (H₂) in an aqueous medium to partially hydrogenate the NO. As a result, nitrous oxide gas (N₂O) is formed as a gaseous by-product. The N₂O and H₂O, in addition to unused N₂, H₂, and NO, are preferably removed from the reactor in a first vent gas (1).

[0055] The first vent gas (1) typically includes approximately 67% H₂, 14% NO, 9% N₂, 4% H₂O, 8% N₂O, and a trace amount of other gases such as methane, CO₂ and CO. However, the following examples utilize synthetic mixtures of the aforementioned gases representative of a typical first vent gas (1). The synthetic mixtures of the gases are hereafter referred to as "mixtures."

[0056] A first pressure swing adsorption (PSA) system (29) is employed and utilizes a first mixture of N₂, N₂O, and H₂O. A second PSA system (65) is also employed and utilizes a second mixture. The second mixture includes H₂, N₂, NO, and also includes N₂O. A removal unit (27) is not used with the first and second mixtures.

[0057] The first PSA system (29) includes a water adsorbent material (31) that has a high selectivity for the H₂O, thereby allowing for removal of the H₂O from the first mixture. Specific parameters to be used in the first PSA system (29) along with the results of the removal of the H₂O from the first mixture are set forth in Table 1. A first pressurization time, a first adsorption time, and a first blowdown time, included in Table 1, are as first introduced and described above.

[0058] More specifically, the first PSA system (29) includes two first adsorbent beds (30), bed A and bed B, respectively. The first adsorbent beds (30) are constructed from two inch Sch 80 PVC pipe and include Davison 03 commercially available from Grace Davison of Columbia, MD, as the water adsorbent material (31). In all examples in Table 1, the length of the first adsorbent beds (30) is 5.47 inches, the mass of the water adsorbent material (31) in bed A is 180.41 grams, the mass of the water adsorbent material (31) in bed B is 180.43 grams, and the amount of H₂O in the first mixture used in Table 1 is 50% relative humidity at 25°C and 31 psia.

TABLE 1

Examples	First Pressurization Time (s)	First Adsorption Time (s)	First Blowdown Time (s)	H₂O Initially Present (ppm)	H₂O Finally Present (ppm)
Example 1	10	620	10	7463	1535
Example 2	10	600	10	7413	252.6
Example 3	10	600	10	7423	21.4
Example 4	10	600	10	7274	25.7

[0059] H₂O initially present includes an amount of H₂O initially present in the first mixture. Similarly, H₂O finally present includes an amount of H₂O finally present in the first mixture.

[0060] The second PSA system (65) includes a nitrous oxide adsorbent material (67) that has a high selectivity for the N₂O. Specific parameters to be used in the second PSA system (65) along with the results of the removal of the N₂O from the second and third mixtures, are set forth in Table 2. A second pressurization time, a second adsorption time, an N₂ pressurization time, a second blowdown time, a second

desorption time, and a desorption rate, included in Table 2, are as first introduced and described above.

[0061] More specifically, the second PSA system (65) includes one second adsorbent bed (66), bed C, operated in batch mode. The second adsorbent bed (66) is constructed from one inch 304 SS pipe and include HISIV-3000 commercially available from UOP LLC of Des Plaines, Il, as the nitrous oxide adsorbent material (67). In all examples in Table 2, the length of the second adsorbent beds (66) is 36 inches, the mass of the nitrous oxide adsorbent material (67) in bed C is 0.7174 grams. Examples 1 to 3 utilize the second mixture.

TABLE 2

Examples	Second Pressurization Time (min)	Second Adsorption Time (min)	N₂ Pressurization Time (min)	Second Blow-Down Time (min)
Example 1	1.10	8	0.98	0.28
Example 2	1.17	0.5	1.25	0.25
Example 3	1.17	4.25	1.25	0.25

TABLE 2 (cont)

Examples	Second Desorption Time (min)	Low Pressure N₂O lean Purge Time (min)	Maximum Pressure of 2nd PSA System (psia)	Minimum Pressure of 2nd PSA System (psia)
Example 1	19.02	2.97	28	2
Example 2	3.5	2	28	2
Example 3	11	2	28	2

[0062] The invention has been described in an illustrative manner, and it is to be understood that the terminology which has been used is intended to be in the nature of words of description rather than of limitation. Obviously, many modifications and

variations of the present invention are possible in light of the above teachings, and the invention may be practiced otherwise than as specifically described.

CLAIMS

What is claimed is:

1. A method of removing nitrous oxide from a vent gas comprising the nitrous oxide and at least one other gas, said method comprising the steps of:
 - flowing the vent gas through a nitrous oxide adsorbent material such that the nitrous oxide is adsorbed onto the nitrous oxide adsorbent material;
 - flowing nitrogen through the nitrous oxide adsorbent material to displace the other gas; and
 - desorbing the nitrous oxide from the nitrous oxide adsorbent material.
2. A method as set forth in claim 1 further comprising the step of isolating the nitrous oxide adsorbent material from the flow of the vent gas prior to flowing the nitrogen through the nitrous oxide adsorbent material.
3. A method as set forth in claim 2 wherein the step of flowing the nitrogen comprises the step of flowing the nitrogen counter-currently to the flow of the vent gas after the nitrous oxide adsorbent material is isolated from the flow of the vent gas.
4. A method as set forth in claim 1 wherein the step of flowing the nitrogen comprises the step of flowing the nitrogen counter-currently to the flow of the vent gas.
5. A method as set forth in claim 1 further comprising the step of flowing a multi-component gas through the nitrous oxide adsorbent material.
6. A method as set forth in claim 5 wherein the step of flowing the multi-component gas comprises flowing the multi-component gas counter-currently to the vent gas.

7. A method as set forth in claim 1 wherein the nitrous oxide adsorbent material is disposed in an adsorbent bed and the step of desorbing the nitrous oxide comprises the step of lowering a pressure of the adsorbent bed. A method of removing nitrous oxide from a vent gas comprising the nitrous oxide and at least one other gas, said method comprising the steps of:

synthesizing hydroxylamine thereby producing the vent gas;

flowing the vent gas through a nitrous oxide adsorbent material such that the nitrous oxide is adsorbed onto the nitrous oxide adsorbent material;

flowing nitrogen through the nitrous oxide adsorbent material to displace the other gas; and

desorbing the nitrous oxide from the nitrous oxide adsorbent material.

8. A method as set forth in claim 8 further comprising the step of isolating the nitrous oxide adsorbent material from the flow of the vent gas prior to flowing the nitrogen through the nitrous oxide adsorbent material.

9. A method as set forth in claim 9 wherein the step of flowing the nitrogen comprises the step of flowing the nitrogen counter-currently to the flow of the vent gas after the nitrous oxide adsorbent material is isolated from the flow of the vent gas.

10. A method as set forth in claim 8 wherein the step of flowing the nitrogen comprises flowing the nitrogen counter-currently to the vent gas.

11. A method as set forth in claim 8 further comprising the step of flowing a multi-component gas stream through the nitrous oxide adsorbent material.

12. A method as set forth in claim 12 wherein the step of flowing the multi-component gas comprises flowing the multi-component gas counter-currently to the vent gas.

13. A method as set forth in claim 8 wherein the nitrous oxide adsorbent material is disposed in an adsorbent bed and the step of desorbing the nitrous oxide comprises the step of lowering a pressure of the adsorbent bed.

14. A method of removing nitrous oxide from a vent gas comprising the nitrous oxide and at least one other gas, said method comprising the steps of:

adsorbing the nitrous oxide from a vent gas onto a nitrous oxide adsorbent material;

flowing nitrogen into the nitrous oxide adsorbent material; and

desorbing the nitrous oxide from the nitrous oxide adsorbent material.

AMENDED CLAIMS

Received by the International Bureau on 14 March 2007 (14.03.07)

+Statement**WHAT IS CLAIMED IS:**

1. A method of removing nitrous oxide from a vent gas comprising the nitrous oxide and at least one other gas, said method comprising the steps of:

flowing the vent gas through a nitrous oxide adsorbent material such that the nitrous oxide is adsorbed onto the nitrous oxide adsorbent material;

flowing nitrogen through the nitrous oxide adsorbent material to displace the other gas;

desorbing the nitrous oxide from the nitrous oxide adsorbent material; and

flowing a multi-component gas through the nitrous oxide adsorbent material.

2. A method as set forth in claim 1 further comprising the step of isolating the nitrous oxide adsorbent material from the flow of the vent gas prior to flowing the nitrogen through the nitrous oxide adsorbent material.

3. A method as set forth in claim 2 wherein the step of flowing the nitrogen comprises the step of flowing the nitrogen counter-currently to the flow of the vent gas after the nitrous oxide adsorbent material is isolated from the flow of the vent gas.

4. A method as set forth in claim 1 wherein the step of flowing the nitrogen comprises the step of flowing the nitrogen counter-currently to the flow of the vent gas.

5. (Cancelled)

6. A method as set forth in claim 1 wherein the step of flowing the multi-component gas comprises flowing the multi-component gas counter-currently to the vent gas.

7. A method as set forth in claim 1 wherein the nitrous oxide adsorbent material is disposed in an adsorbent bed and the step of desorbing the nitrous oxide comprises the step of lowering a pressure of the adsorbent bed.

8. A method as set forth in claim 15 further comprising the step of isolating the nitrous oxide adsorbent material from the flow of the vent gas prior to flowing the nitrogen through the nitrous oxide adsorbent material.

9. A method as set forth in claim 8 wherein the step of flowing the nitrogen comprises the step of flowing the nitrogen counter-currently to the flow of the vent gas after the nitrous oxide adsorbent material is isolated from the flow of the vent gas.

10. A method as set forth in claim 15 wherein the step of flowing the nitrogen comprises flowing the nitrogen counter-currently to the vent gas.

11. (Cancelled)

12. A method as set forth in claim 15 wherein the step of flowing the multi-component gas comprises flowing the multi-component gas counter-currently to the vent gas.

13. A method as set forth in claim 15 wherein the nitrous oxide adsorbent material is disposed in an adsorbent bed and the step of desorbing the nitrous oxide comprises the step of lowering a pressure of the adsorbent bed.

14. A method of removing nitrous oxide from a vent gas comprising the nitrous oxide and at least one other gas, said method comprising the steps of:

adsorbing the nitrous oxide from a vent gas onto a nitrous oxide adsorbent material;

flowing nitrogen into the nitrous oxide adsorbent material;

desorbing the nitrous oxide from the nitrous oxide adsorbent material; and
flowing a multi-component gas stream through the nitrous oxide adsorbent material.

15. A method of removing nitrous oxide from a vent gas comprising nitrous oxide and at least one other gas, said method comprising the steps of:

synthesizing hydroxylamine thereby producing the vent gas;
flowing the vent gas through a nitrous oxide adsorbent material such that the nitrous oxide is adsorbed onto the nitrous oxide adsorbent material;
flowing nitrogen through the nitrous oxide adsorbent material to displace the other gas;
desorbing the nitrous oxide from the nitrous oxide adsorbent material; and
flowing a multi-component gas stream through the nitrous oxide adsorbent material.

STATEMENT UNDER ARTICLE 19(1)

Claims 1 and 14 have been amended to include the step of flowing a multi-component gas stream through the nitrous oxide adsorbent material. Claim 15, which has been added to remedy the typographical error in the originally submitted claim 7, sets forth a method of removing nitrous oxide from a vent gas including the step of flowing a multi-component gas stream through the nitrous oxide adsorbent material. The multi-component gas may be used to purge an absorbent bed and/or displace N₂ from void spaces of the nitrous oxide adsorbent material such that additional amounts of the vent gas can be utilized.

With regard to the novelty and the inventive step of the claimed invention in view of:

(D1) U.S. 4,507,271

(D3) EP 1 275 616 A

(D2) DE 198 08 939 A1

(D4) U.S. 6,080,226

which were each cited in the ISR, none of these documents disclose, teach, or suggest the all of the features of the amended claims 1 or 14 or the newly added claim 15. With respect to DE 198 08 939 A1, the Applicant bases the aforementioned statement on an analysis of the English language abstract of the document presented in WO 99/44938 and on an English translation of the document obtained through the European Patent Office (esp@cenet).

With respect to amended claims 1 and 14 and claim 15, each claim requires the step of flowing a multi-component gas through the nitrous oxide adsorbent material. As set forth in the instant specification, the multi-component gas may include H₂, NO, N₂, other gasses and trace components. In one embodiment, the multi-component gas is used to purge an

absorbent bed. In another embodiment, the multi-component gas displaces N_2 from void spaces of the nitrous oxide adsorbent material such that additional amounts of the vent gas can be utilized.

D1 generally focuses on removal of nitrous oxide from gases which are treated with molecular sieves. More specifically, D1 focuses on flushing adsorbent beds with N_2 gas to remove H_2 and NO and follows this step with the step of desorbing the N_2O . However, D1 does not disclose the step of flowing a multi-component gas stream through the nitrous oxide adsorbent material. As such, D1 does not disclose, teach, or suggest the novelty and the inventive step of the amended claims.

D2 is presented in German and has no English family member or equivalent. As a result, Applicant relies on an English language abstract of the document presented in WO 99/44938 and on an English translation of the document obtained through the European Patent Office (esp@cenet). According to the English abstract and the English translation, D2 focuses on a method for producing hydroxylammonium salts by means of catalytic reduction of nitrogen monoxide (NO) with hydrogen and purifying the nitrogen monoxide by at least one treatment with porous oxides based on silicon and/or aluminum or with activated carbon. There is no mention of pressure swing adsorption in the document or any mention of specific steps of such a process. Importantly, nitrogen monoxide (NO) is not the same as nitrous oxide (N_2O). As such, D2 does not disclose, teach, or suggest the novelty and the inventive step of the amended claims.

D3 focuses on a process for the preparation of hydroxylammonium and includes separating hydrogen and gaseous non-hydrogen compounds. However, this process does not include the step of flowing nitrogen through the nitrous oxide adsorbent material to displace the other gas or the step of flowing a multi-component gas through the nitrous oxide adsorbent material. Instead, D3 only references pressure swing adsorption in passing and only generally discloses the well known steps of pressure swing adsorption including separating mixtures of gasses to obtain two or more separated and at least partially purified gasses. Thus, D3 does not disclose or teach the step of flowing a multi-component gas through the nitrous oxide adsorbent material and therefore does not disclose, teach, or suggest the novelty and the inventive step of the amended claims.

D4 focuses on purifying nitrous oxide using pressure swing adsorption. More specifically, D4 focuses on counter-currently purging adsorbent beds with oxygen lean gas streams, i.e., substantially pure nitrogen streams. Yet, D4 does not disclose the step of flowing a multi-component gas stream through the nitrous oxide adsorbent material. Thus, D4 does not disclose, teach, or suggest the novelty and the inventive step of the amended claims.

In sum, none of these documents, either independently or in combination, disclose, teach, or suggest the novelty and the inventive step as set forth in amended claims 1 and 14 or claim 15. Claims 2-7 depend from claim 1 and claims 8-13 depend from claim 15. Therefore, these claims are also novel and involve an inventive step.

Item V of Written Opinion:

In reference to Item V of the Written Opinion regarding a lack of conciseness between claims 1 and 15 [sic] (claim 14), the Applicant points out that the scope of these claims are different and do not differ simply with regard to the definition of the subject-matter for which protection is sought. Specifically, claim 1 requires the steps of:

flowing the vent gas through a nitrous oxide adsorbent material such that the nitrous oxide is adsorbed onto the nitrous oxide adsorbent material; and

flowing nitrogen through the nitrous oxide adsorbent material to displace the other gas.

Claim 14, on the other hand, requires the steps of:

adsorbing the nitrous oxide from a vent gas onto a nitrous oxide adsorbent material; and

flowing nitrogen into the nitrous oxide adsorbent material

As such, it is clear that the step of claim 1 of flowing the vent gas through the nitrous oxide adsorbent such that the nitrous oxide is adsorbed is related but not the same as the step of claim 14 of adsorbing the nitrous oxide from the vent gas. The step of adsorbing the nitrous oxide from the vent gas is broader in scope because it does not require flowing the vent gas through the nitrous oxide adsorbent material. It is also clear that the step of claim 1 of flowing nitrogen through the nitrous oxide adsorbent material to displace the other gas is not the same as the step of claim 14 of flowing nitrogen into the nitrous oxide adsorbent material. Flowing nitrogen through a material to displace a gas is different than simply flowing nitrogen into the material. Therefore, these steps are also of different scope. For this reason, Applicant respectfully submits that claims 1 and 14 are not appropriately rejected due

to a lack of conciseness.

The Applicant respectfully submits that the claimed invention, as amended, is novel and involves an inventive step over the documents cited in the ISR. Further and favorable reconsideration of the subject application is hereby requested. The Applicant also respectfully submits that claims 1 and 14 should not be rejected due to a lack of conciseness.

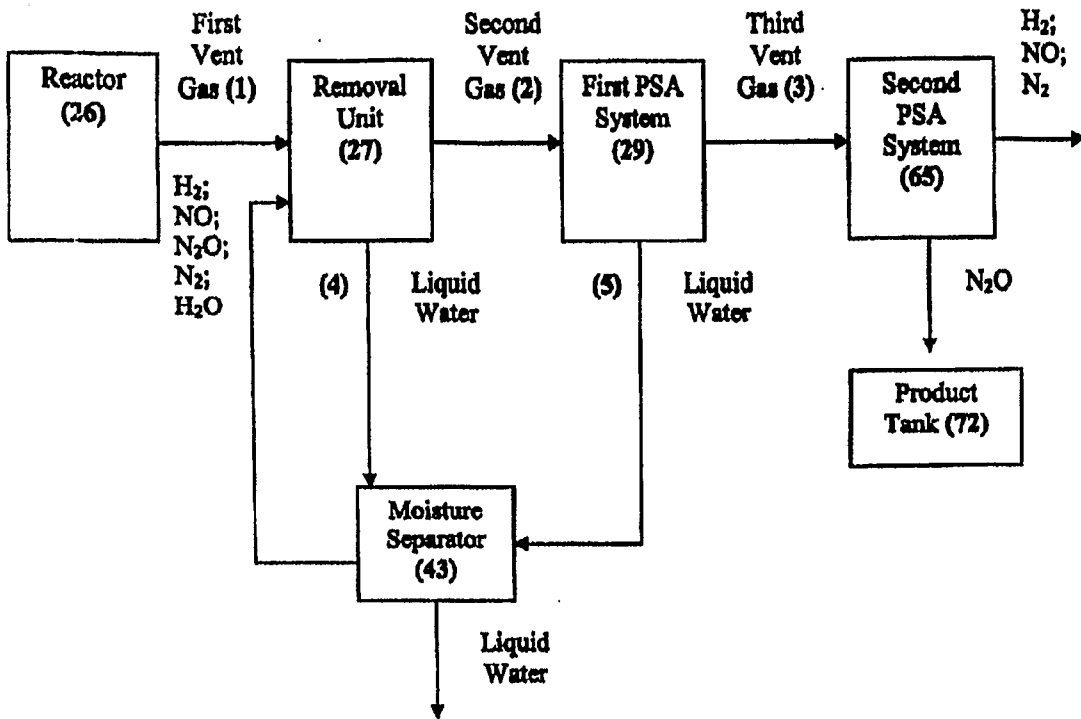
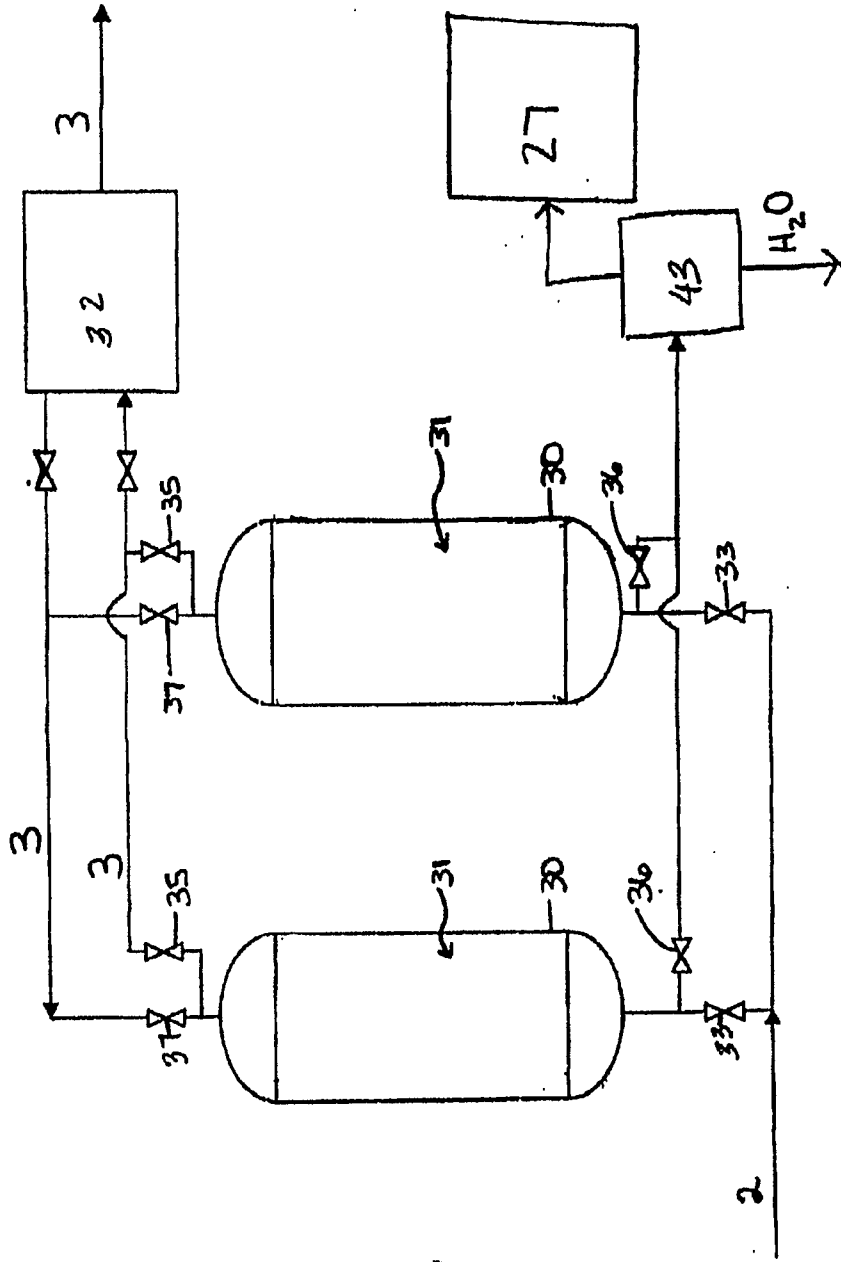


Figure 1



29 ~>

Figure 2

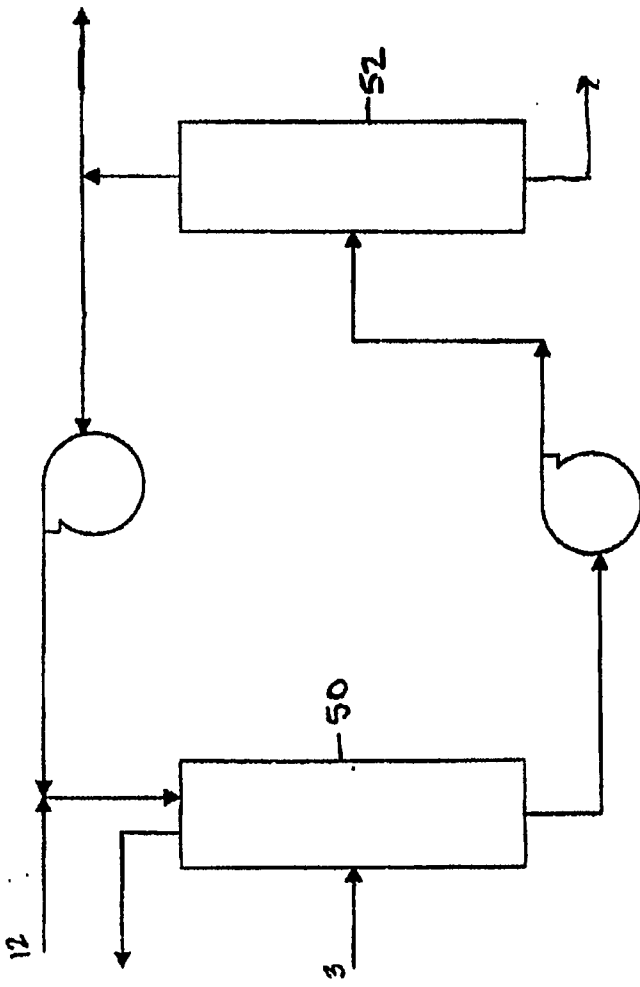


Figure 3 -

65 ~>

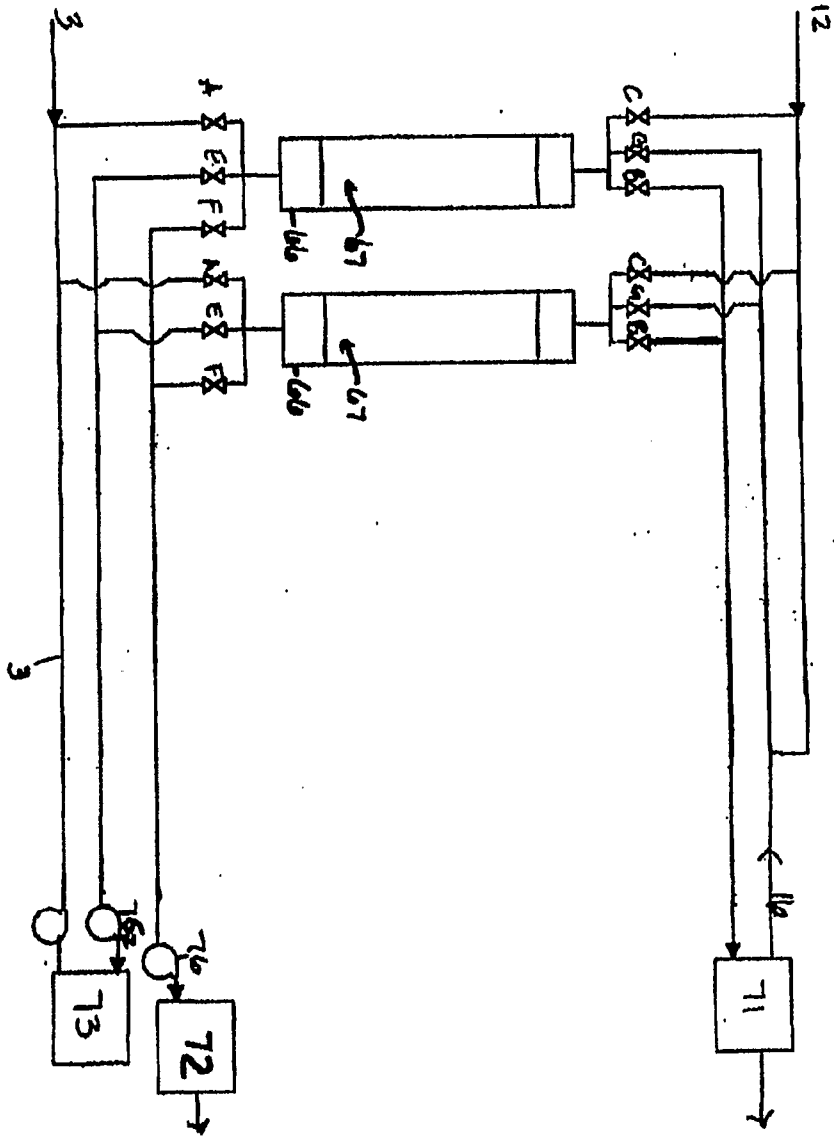


Figure 4:

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2006/037650

A. CLASSIFICATION OF SUBJECT MATTER INV. B01D53/04 C01B21/14		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols) B01D C01B		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practical, search terms used) EPO-Internal, WPI Data		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 507 271 A (VAN DEYCK FRANS [BE] ET AL) 26 March 1985 (1985-03-26) examples 1-4	1-4, 7-10,13, 14
X	DE 198 08 939 A1 (BASF AG [DE]) 9 September 1999 (1999-09-09) example 1	1,7,14
X	EP 1 275 616 A (DSM NV [NL]) 15 January 2003 (2003-01-15) claim 1	1,7,14
X	US 6 080 226 A (DOLAN WILLIAM B [US] ET AL) 27 June 2000 (2000-06-27) claim 1	1,7,14
<input type="checkbox"/> Further documents are listed in the continuation of Box C.		
<input checked="" type="checkbox"/> See patent family annex.		
* Special categories of cited documents :		
A document defining the general state of the art which is not considered to be of particular relevance *E* earlier document but published on or after the international filing date *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) *O* document referring to an oral disclosure, use, exhibition or other means *P* document published prior to the international filing date but later than the priority date claimed	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. *&* document member of the same patent family	
Date of the actual completion of the international search <p style="text-align: center;">9 January 2007</p>	Date of mailing of the international search report <p style="text-align: center;">16/01/2007</p>	
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer <p style="text-align: center;">Faria, Cristina</p>	

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/US2006/037650

Patent document cited in search report	.	Publication date	Patent family member(s)	Publication date
US 4507271	A	26-03-1985	CS 249131 B2	12-03-1987
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			PL 244895 A2	30-07-1984
DE 19808939	A1	09-09-1999	BG 104723 A	31-05-2001
			BR 9908398 A	31-10-2000
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