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(54) NITROGEN PRODUCTION METHOD AND APPARATUS

VERFAHREN UND VORRICHTUNG ZUR STICKSTOFFERZEUGUNG

PROCÉDÉ ET APPAREIL DE PRODUCTION D'AZOTE

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Description

Field of the Invention

[0001] The present invention relates to a process and apparatus for separating a gaseous mixture within a rectification column by cryogenic rectification to produce a nitrogen product in which an oxygen-enriched liquid composed of a column bottoms is vaporized against condensing a nitrogen containing column overhead to produce reflux for the rectification column. More particularly, the present invention relates to such a process and apparatus in which the oxygen-enriched liquid is successively vaporized and a nitrogen-rich vapor phase produced by the successive vaporization of the oxygen-enriched liquid is condensed and then reintroduced into the column to enhance nitrogen recovery.

Document JP-A-2000 18813, which can be considered as the closest prior art, discloses a method of separating a gaseous mixture comprising nitrogen and oxygen to produce a nitrogen product, said method comprising: introducing a purified, pressurized and cooled gaseous stream into a rectification column to produce an overhead nitrogen-rich vapor and an oxygen-enriched liquid bottoms; depressurizing a first oxygen-enriched liquid stream composed at least in part of the oxygen-enriched liquid bottoms, partially vaporizing the first oxygen-enriched liquid stream within a first heat exchanger, disengaging a vapor phase from a liquid phase formed by the partial vaporization of the first oxygen-enriched liquid stream, depressurizing a second oxygen-enriched liquid stream composed at least in part of the liquid phase; condensing a first part of a column overhead nitrogen-rich stream composed of the overhead nitrogen-rich vapor in the first heat exchanger, and returning at least part of the condensed column overhead nitrogen-rich stream to the rectification as reflux; introducing at least part of the nitrogen-rich liquid stream into the rectification column, above the purified, pressurized and cooled gaseous stream; and producing a product nitrogen stream from part of the overhead nitrogen-rich vapor.

Background of the Invention

[0002] Nitrogen can be separated from gaseous mixtures that comprise nitrogen and oxygen, for example, air, by cryogenic rectification. Typically, a compressed and purified air stream is cooled to a temperature suitable for its rectification and then rectified within a rectification column to produce a nitrogen-rich vapor as column overhead and an oxygen-enriched column bottoms. Reflux for the column is produced by condensing some of the nitrogen-rich vapor. This condensation is effectuated through indirect heat exchange of a stream of the nitrogen-rich vapor and a stream of the oxygen-enriched column bottoms. Part of the liquid can be taken as a product.

[0003] The resultant vaporization of the oxygen-enriched column bottoms produces a stream that is referred

to as a waste stream. The waste stream and a nitrogen product stream are passed through a main heat exchanger in order to cool the incoming compressed and purified air. Refrigeration can be supplied by partially heating the waste stream within the main heat exchanger, passing the waste stream through a turboexpander and then subsequently warming the waste stream within the main heat exchanger.

[0004] In general, the performance of the condenser used to exchange heat between oxygen-enriched liquid and the nitrogen-rich vapor is inefficient. Excess temperature difference exists between the oxygen-enriched liquid stream that is to be vaporized and the overhead nitrogen that is to be condensed. As a consequence, additional compression power is consumed by the process due in large part to the heat transfer within the condenser. Additionally, excess compositional gradients exist near the bottom of the rectification column. In particular, rapid compositional changes occur over few stages. Such steep compositional changes correspond to substantial thermodynamic irreversibility that translates into lost work. In the prior art, substantial efforts have focused primarily on condenser operation and column recovery.

[0005] U.S. 4,867,773 and U.S. 4,872,893 detail similar processes that involve a single column nitrogen rectification process in which at least a portion of the evaporated oxygen-rich bottoms is warmed, compressed and recycled to the column. The recycled stream is fed to a point lower than the feed air. The effect of the modification allows the overhead condenser to operate more efficiently to increase nitrogen recovery.

[0006] U.S. 4,883,519 illustrates another single column nitrogen rectification process wherein the overhead nitrogen is condensed by way of two heat exchangers. In this process the oxygen-rich column bottoms stream is depressurized to a first pressure and partially vaporized. The resulting vapor is recycled to the main air compressor. The remaining oxygen-rich liquid is further depressurized and directed to a second lower pressure heat exchanger where it is substantially vaporized. At least a portion of the resulting vapor is directed to a turbine expander for refrigeration production.

[0007] U.S. 4,927,441 discloses another single column nitrogen process that is similar to that disclosed in U.S. 4,883,519. In this arrangement, the oxygen-rich column bottoms is depressurized to a first pressure and then introduced into a separation vessel which incorporates a small mass-transfer section containing mass-transfer elements such as a packing. The mass-transfer column section serves to further enrich the oxygen content of the stream prior to its depressurization and introduction into a second heat exchanger. The advantage of this arrangement is that the overhead produced in the mass-transfer section has very nearly the composition of air and thus, can be recycled by way of the main air compressor, without associated compositional mixing losses.

[0008] U.S. 5,711,167 discloses a single column nitrogen process in which two overhead condensers are used

to generate reflux to the rectification column by successive vaporizations of oxygen-enriched liquid. In a first partial vaporization of the oxygen-enriched liquid conducted in one of the two overhead condensers, the resulting vapor is compressed in a cold compressor and redirected back to the base of the rectification column. The oxygen-rich waste produced from a second partial vaporization of the oxygen-enriched liquid, conducted in the second condenser, is warmed and expanded prior to venting. At least a portion of the shaft work of expansion is directed to the cold compression.

[0009] U.S. 5,899,093 details a process in which the oxygen-enriched column bottoms is first partially depressurized and introduced into a dephlegmator-type condenser. The oxygen-rich bottoms, produced in the condenser, is separated into a nitrogen-rich gas which is recycled by way of gas compression within an air compressor. The further oxygen-rich remaining fluid is further depressurized and used to condense an additional portion of the overhead nitrogen from the column. The further oxygen enriched remaining fluid is further depressurized to condense an additional portion of the overhead nitrogen from the column. The nitrogen fraction resulting from the first partial vaporization is recycled to a gas compression and then to the column.

[0010] U.S. 5,934,106 and U.S. 5,868,006 disclose a single column nitrogen generator in which overhead nitrogen is condensed against two streams derived from the column system. A first nitrogen enriched air-like liquid stream is evaporated and recompressed back to the column. A second oxygen-rich column bottoms stream is extracted from the column and also evaporated. The second evaporated fraction is warmed and then expanded. The work of expansion provides the shaft work required for the cold compression of the first recycled stream.

[0011] As will be discussed, the present invention involves a process for recovering nitrogen-rich vapor by the cryogenic rectification of air or other oxygen and nitrogen containing gas within a rectification column in which a nitrogen-rich liquid condensate produced in the course of sequential vaporizations of an oxygen-enriched liquid stream, made-up at least in part from liquid column bottoms, is introduced into the rectification column to increase production of the nitrogen-rich vapor in an energy efficient and cost effective manner.

Summary of the Invention

[0012] The present invention provides a method of separating a gaseous mixture comprising nitrogen and oxygen to produce a nitrogen product according to claim 1. In accordance with the method, a purified, pressurized and cooled gaseous stream composed of the gaseous mixture is introduced into a rectification column to produce an overhead nitrogen-rich vapor and an oxygen-enriched liquid bottoms. A first oxygen-enriched liquid stream that is at least in part composed of the oxygen-enriched liquid bottoms is depressurized and then par-

tially vaporized within a first heat exchanger. A vapor phase is disengaged from a liquid phase that is formed by the partial vaporization of the first oxygen-enriched liquid stream. A second oxygen-enriched liquid stream composed at least in part of the liquid phase is depressurized and is then partially vaporized through indirect heat exchange with at least a portion of a vapor phase stream composed of the vapor phase within a second heat exchanger. This substantially condenses the vapor phase stream to form a nitrogen-rich liquid stream.

[0013] A first part of a column overhead nitrogen-rich stream that is composed of the overhead nitrogen-rich vapor is condensed in the first heat exchanger and a second part of the column overhead nitrogen-rich stream is condensed in a third heat exchanger. The condensation of the second part of the column overhead nitrogen-rich stream is conducted through indirect heat exchange with the second oxygen-enriched stream after the partial vaporization thereof, thereby further vaporizing the second oxygen-enriched stream. At least part of the column overhead nitrogen-rich stream after having been condensed is returned to the rectification column as reflux.

[0014] At least part of the nitrogen-rich liquid stream is then introduced into the rectification column, above the purified, pressurized and cooled stream. A product nitrogen stream is produced from part of the nitrogen-rich vapor.

[0015] The first oxygen-enriched liquid stream can be subcooled through indirect heat exchange with the nitrogen product stream and a waste stream composed of a vapor fraction of the second oxygen-enriched liquid stream after having been further vaporized. A compressed and purified stream composed of the gaseous mixture can be cooled by indirect heat exchange with the nitrogen product stream and the waste stream after having subcooled the oxygen-enriched stream and if present, the second part of the nitrogen-rich vapor stream prior to its compression. The cooling of the compressed and purified stream thereby forms at least a portion of the purified, pressurized and cooled stream.

[0016] In a specific embodiment of the present invention, a first part of the vapor phase stream can be substantially condensed within the second heat exchanger to form the nitrogen-rich liquid stream and a second part of the vapor phase stream can be warmed, compressed and cooled and recycled back to the rectification column. The cooling and the recycling can be accomplished by combining the second part of the vapor phase stream, after having been warmed and compressed, with the compressed and purified stream to form a combined compressed and purified stream. In such embodiment, the combined compressed and purified stream is cooled by the nitrogen product stream, the waste stream and the second part of the nitrogen-rich vapor stream. As such, the second part of the vapor phase stream is cooled and recycled back to the rectification column by being combined with the compressed and purified stream.

[0017] Preferably, the waste stream and the nitrogen

product stream and if present, the second part of the nitrogen-rich vapor stream prior to its compression, all indirectly exchange heat with the compressed and purified stream within a main heat exchanger. The waste stream can be partially warmed within the main heat exchanger and is then expanded with the performance of work to generate an exhaust stream. The exhaust stream is reintroduced into the main heat exchanger and fully warmed to refrigerate the cryogenic rectification process. It is to be noted that the term "partially warmed" as used herein and in the claims means that the waste stream is warmed to a temperature intermediate the temperatures of the warm and cold ends of the main heat exchanger. The term, "fully warmed" as used herein and in the claims means fully warmed to the warm end temperature of the main heat exchanger.

[0018] The pressure of the nitrogen-rich liquid stream can be adjusted after having been substantially condensed and prior to its being introduced into the rectification column. This adjustment can be effectuated by mechanically pumping the nitrogen-rich liquid stream after having been substantially condensed or by expanding the nitrogen-rich liquid stream.

[0019] In another aspect, the present invention provides an apparatus for separating a gaseous mixture comprising nitrogen and oxygen to produce a nitrogen product according to claim 8. In accordance with this aspect of the present invention a rectification column is connected to the main heat exchanger for rectifying a purified, pressurized and cooled stream composed of the gaseous mixture to produce an overhead nitrogen-rich vapor and an oxygen-enriched liquid bottoms.

[0020] A first valve is provided to depressurize a first oxygen-enriched stream composed at least in part of the oxygen-enriched liquid bottoms. A first heat exchanger is connected to the first valve for partially vaporizing the first oxygen-enriched liquid stream and a phase separator is connected to the first heat exchanger for disengaging a vapor phase from a liquid phase formed by the partial vaporization of the first oxygen-enriched stream. A second valve is connected to the phase separator for depressurizing a second oxygen-enriched liquid stream composed at least in part of the liquid phase and a second heat exchanger is connected to the second valve and to the phase separator for partially vaporizing the second oxygen-enriched liquid stream through indirect heat exchange with at least a portion of a vapor phase stream composed of the vapor phase formed by the partial vaporization of the first oxygen-enriched stream. This substantially condenses the vapor phase stream to produce a nitrogen-rich liquid stream. A third heat exchanger is operated in series with the second heat exchanger for further vaporizing the second oxygen-enriched liquid stream.

[0021] The rectification column is connected to the first heat exchanger and the third heat exchanger for purposes of condensing at least a portion of a column overhead nitrogen-rich stream composed of the overhead nitrogen-

rich vapor and returning at least a part of the column overhead nitrogen-rich stream after having been condensed to the rectification column as reflux.

[0022] The second heat exchanger is connected to the rectification column for introducing at least part of the nitrogen-rich liquid stream into the rectification column, above the purified, pressurized and cooled stream. A means is provided for extracting a product nitrogen stream formed from part of the overhead nitrogen-rich vapor.

[0023] In a specific embodiment of the present invention, a main heat exchanger can be provided to cool a compressed and purified stream composed of the gaseous mixture and thereby to form the purified, pressurized and cooled stream. In an alternative embodiment, the second heat exchanger can be connected to the phase separator so that a first part of the vapor phase stream is substantially condensed within the second heat exchanger to form the nitrogen-rich liquid stream. A compressor can be connected in flow communication with the phase separator and to the main heat exchanger and the main heat exchanger can be configured so that a second part of the vapor phase stream is warmed within the main heat exchanger and compressed within the compressor. The main heat exchanger in such alternative is also simultaneously in flow communication with the compressed and purified stream and the compressor such that the second part of the vapor phase stream combines with the compressed and purified stream to form a combined compressed and purified stream. In such embodiment the compressed and purified stream is cooled within the main heat exchanger to form the purified, pressurized and cooled stream.

[0024] A subcooler can be connected to the rectification column so that the first oxygen-enriched liquid stream is subcooled through indirect heat exchange with the nitrogen product stream and a waste stream composed of a vapor fraction of the second oxygen-enriched liquid stream after having been further vaporized. The main heat exchanger is also connected to the subcooler and configured so that the compressed and purified air stream is cooled by indirect heat exchange with a nitrogen product stream and the waste stream after having subcooled the first oxygen-enriched stream. If present, the second part of the vapor phase stream also serves to cool the combined compressed and purified air stream.

[0025] Preferably, the main heat exchanger can also be configured such that the waste stream partially warms within the main heat exchanger and an exhaust stream fully warms within the main heat exchanger to refrigerate the apparatus. An expander is connected to the main heat exchanger so that the waste stream after having been partially warmed is expanded within the expander with the performance of work to generate the exhaust stream.

[0026] A pump can be interposed between the second heat exchanger and the rectification column to pressurize the nitrogen-rich liquid stream after having been substan-

tially condensed and prior to its introduction into the rectification column. Alternatively, a third valve can be interposed between the second heat exchanger and the rectification column in order to reduce the pressure of the nitrogen-rich liquid stream after having been substantially condensed and prior to its introduction into the rectification column. The choice of pump or valve will depend upon the gravitation head generated by the elevation difference between the second condenser and the feed location for the nitrogen-rich liquid.

[0027] As is apparent from the description of the present invention in both aspects of its method and apparatus, the return of the nitrogen-rich liquid to the column has the advantage of increasing the production of the nitrogen-rich vapor. It also decreases compositional variations within the bottom of the column to bring the operating line closer to the vapor liquid equilibrium curve thereby generating greater efficiency. This decreases the lost work which translates into decreased compression requirements.

The indirect heat exchange between the oxygen-enriched liquid stream and the nitrogen-rich vapor that is used in refluxing the column can be conducted more efficiently than in the prior art due to the three-stage vaporization process that reduces the log mean temperature differences of the streams subjected to indirect heat exchange. This results in a process that is more efficient than those conducted in the prior art.

Brief Description of the Drawings

[0028] While the specification concludes with claims distinctly pointing out the subject matter that Applicant regards as his invention, it is believed that the invention will be better understood when taken in connection with the accompanying drawings in which:

[0029] Fig. 1 is a schematic, process flow diagram of an apparatus that can be used for carrying out a method in accordance with the present invention; and

[0030] Fig. 2 is an alternative embodiment of Fig. 1.

Detailed Description

[0031] With reference to Fig. 1, an apparatus 1 for carrying out a method in accordance with the present invention is illustrated.

[0032] A compressed and purified stream 10 that is composed of nitrogen and oxygen, for instance, air, is cooled in a main heat exchanger 12 to form a purified, pressurized and cooled stream 13 that is then introduced into a rectification column 14 for separation of the oxygen and nitrogen. Within rectification column 14, the purified, pressurized and cooled purified stream 13 is rectified and separated into an oxygen-enriched liquid bottoms 16 and an overhead nitrogen-rich vapor 18. A product stream 20 composed of the nitrogen-rich vapor 18 can be fully warmed within the main heat exchanger 12 against cooling compressed and purified stream 10.

[0033] Rectification column 14 contains mass-transfer contacting elements such as structured packing or trays that are generally disposed within a bottom region 22 of rectification column 14 and the remaining region 24 situated above bottom region 22. Typically, rectification column 14 will operate in a pressure range of between about 5 bar absolute and about 12 bar absolute.

[0034] Compressed and purified stream 10 can be formed as a result of unit operations being conducted in another process or as known in the art, can also be formed with the use of a compressor and a prepurification unit utilizing an adsorbent to adsorb water, carbon dioxide and potentially dangerous hydrocarbons that could otherwise freeze-out or accumulate within the cryogenic process. As can be appreciated, a compressor and purification unit could be used in connection with the present invention.

[0035] Compressed and purified stream 10 is introduced into rectification column 14 at about its saturation temperature and as such its introduction initiates the formation of an ascending vapor phase that becomes evermore rich in nitrogen to form the overhead nitrogen-rich vapor 18. A column overhead nitrogen-rich stream 26 that is composed of the overhead nitrogen-rich vapor is withdrawn from rectification column 14 and is divided into a subsidiary column overhead nitrogen-rich stream 28 and nitrogen product stream 20. It is understood, however, that nitrogen product stream 20 could be separately withdrawn from rectification column 14. As will be discussed, subsidiary column overhead nitrogen-rich stream 28 is condensed to produce a reflux stream 29 that is introduced into rectification column 14 to initiate the formation of a descending liquid phase which contacts the ascending vapor phase and becomes evermore rich in oxygen as it descends to form oxygen-enriched liquid 16 as the column bottoms.

[0036] A first oxygen-enriched liquid stream 30 composed of the oxygen-enriched liquid bottoms is optionally subcooled within a subcooling heat exchanger 32. The resulting subcooled liquid has its pressure reduced by a first valve 34. First oxygen-enriched liquid stream 30 after passage through first valve 34 is then partially vaporized within a first heat exchanger 36 to form a two-phase stream 38. The liquid and vapor phases within two-phase stream 38 are separated within a phase separator 40 into a liquid phase 42 and a vapor phase 44. It is to be noted that oxygen-enriched liquid stream 30 after passage through first heat exchanger 36 can typically have a vapor fraction of between about 10% and about 40% and more preferably, about 30%.

[0037] A second oxygen-enriched liquid stream 46 composed of liquid phase 42 is depressurized with the use of a second valve 48 and then partially vaporized within a second heat exchanger 50. The pressure drop across second valve 48 will typically be in a range of between about 1.0 and 1.5 bar gauge. Also introduced into second heat exchanger 50 is a vapor phase stream 52 composed of vapor phase 44 that is substantially con-

densified to form a nitrogen-rich liquid stream 54.

The term "substantially condensed" as used herein and in the claims means a stream having a liquid fraction that will typically exceed about 95% by volume. Moreover, it is to be noted that a typical composition for such nitrogen-rich liquid stream is in the range of 75 to 90% nitrogen.

[0038] The second oxygen-enriched liquid stream 46 after having been partially vaporized within second heat exchanger 50 is then introduced into a third heat exchanger 56 which is shown to operate as a natural thermo-siphon to further vaporize the second oxygen-enriched liquid stream 46 and thereby produce a vapor fraction thereof designated by reference number 58 that is discharged as a waste stream 60. The second oxygen-enriched liquid stream 46 after such partial vaporization within exchanger 50 will have a vapor fraction in a range of between about 40% and about 60%, more preferably, about 45% to about 50%.

[0039] First portion 62 of column overhead nitrogen-rich stream 28 is condensed within first heat exchanger 36 and a second portion 64 is condensed within third heat exchanger 56 to produce a liquid stream 66. At least part of combined liquid condensate stream 66 is returned as reflux stream 29 to rectification column 14. An optional liquid product stream 68 can also be taken and directed to suitable storage (not shown).

[0040] It is understood that first heat exchanger 36, second heat exchanger 50 and third heat exchanger 56 are typically brazed aluminum heat exchangers. Other exchanger types could be employed, for instance shell and tube type. Given the use of brazed aluminum heat exchangers, first heat exchanger 36 and third heat exchanger 56 could be integrated into a single block. Moreover any number of heat transfer flow configurations could be employed. The heat exchangers may be once-through vaporizers as illustrated or they may be configured for recirculated vaporization. An example, all the exchangers may be configured as thermo-siphons of both natural and pump circulation type. It is understood that exchanger 56 contained within vessel 70 attached to rectification column 14 is a natural thermo-siphon in which boiling flow is induced by gravitational liquid head.

[0041] It is also noted that the distribution of two-phase streams into aluminum heat exchangers often require separate liquid and vapor inlet distribution passages. In order to facilitate such distribution, first oxygen-enriched liquid stream 30 entering first heat exchanger 36 and two-phase stream 46 entering second heat exchanger 50 may be subject to phase separation. Additionally, it is possible that compressed and purified stream 10 would be introduced into rectification column 14 as liquid and vapor phase streams that were produced by partial condensation of compressed and purified stream 10. In yet another variation, first heat exchanger 36 and second heat exchanger 50 could be used for purposes of sub-cooling other streams, for example stream 68 prior to its being sent to storage.

[0042] Phase separator 40 can be a simple vapor liquid

disengagement vessel. Oxygen enrichment of second oxygen-enriched liquid stream 46 may be increased by inclusion of mass-transfer media such as structured packing or trays. A number of streams may be used to impart additional heat to the base of phase separator 40 to facilitate additional oxygen enrichment if warranted.

[0043] Nitrogen-rich liquid stream 54 is introduced into rectification column 14 above the location of compressed and purified stream 10. This increases the production of nitrogen-rich vapor and also decreases the compositional gradients within the bottom regions of rectification column 14. As can be appreciated, the nitrogen content of condensed stream 54 will be higher than that of oxygen-enriched liquid 16 and thus, it is introduced at a higher level of rectification column 14. It is to be noted that not all of the nitrogen-rich liquid stream 54 need be introduced into rectification column 14. A portion of stream 54 may be: combined with stream 10, vaporized and warmed and/or recycled, or directed to vessel 70 for purposes of heat exchanger control.

[0044] In order to introduce nitrogen-rich liquid stream 54 into rectification column 14, in most cases, there will have to be a pressure adjustment to condensed stream 54 by way of a device 72. If for instance, the pressure of liquid stream 54 is too low for entry into rectification column 14, device 72 can be a pump. If a pump is used, it may be advantageous to employ a vessel to provide additional liquid residence time. Such a vessel would preferably employ a small vapor vent line (which may be connected to the waste stream as necessary). Alternatively, if the static head developed in condensed stream 54 is sufficient due to the placement of components within a cold box, device 72 might simply be a valve.

[0045] As stated above, first oxygen-enriched liquid stream 30 is subcooled within a subcooling unit 32 which can be a brazed aluminum heat exchanger that in fact can be part of heat exchanger 12. First oxygen-enriched liquid stream 30 is subcooled by partly warming waste stream 60 and nitrogen product stream 20. After having been partly warmed, waste stream 60 and nitrogen product stream 20 are introduced into main heat exchanger 12 to cool the incoming compressed and purified steam 10.

[0046] Waste stream 60 upon its discharge from shell 70 typically can have a pressure of between about 2 and about 7 bar absolute. In the illustrated embodiment, the apparatus 1 is refrigerated by partly warming waste stream 60 within a main heat exchanger 12 to form a partly warmed stream 74 that is then expanded within a turboexpander 76 to produce an exhaust stream 78 that is fully warmed within main heat exchanger 12 to a temperature and pressure near ambient, thereby to refrigerate apparatus 1. The shaft work of expansion may be imparted to a generator or used to compress air, nitrogen or waste stream 60 prior to expansion or dissipated by an oil brake as heat. It is to be noted that a portion of stream 74 could bypass turbine 76 and directed into exhaust stream 78 by use of a valve. Other types of refrigeration

eration are possible with the present invention, including, an external refrigeration source or even air expansion as illustrated in the prior art.

[0047] As illustrated in Fig. 2, nitrogen-rich liquid stream 54 is formed by a first part 52a of vapor phase stream 52. A second part 52b of vapor phase stream 52 is passed within main heat exchanger 12' and then compressed within a compressor 80. After having been fully warmed within main heat exchanger 12', second part 52b of vapor phase stream 52 can then be combined with compressed and purified stream 10 to form a combined compressed and purified stream 11. After cooling within main heat exchanger 12', a purified, pressurized and cooled stream 13' is produced for introduction into rectification column 14. It is to be noted that a similar configuration might involve splitting of partially warmed stream 74 to subject a portion of the flow to compression and return another portion to the base of rectification column 14. Another alternative would be to recycle second part 52b of vapor phase stream 52 back into the distillation column 14 by provision of separate passage provided in the main heat exchanger 12' for such purpose. Moreover, waste stream 60 could be directed to another rectification section or alternately, could be warmed, compressed and then fed to a similar process.

[0048] The present invention is applicable to any number of combinations of rectification columns forming similar functions to that as described. For example, rectification column 14 might employ an auxiliary reboiler for further increase and recovery. In such an arrangement, an additional stream of air and nitrogen would be compressed to a higher pressure and condensed within the reboiler and thereby to provide additional vapor flow.

[0049] Furthermore, rectification column 14 may employ a combination of packing, dumped and structured. Rectification column 14 could be split into multiple sections. As known in the art, a "reflux pump" may be employed to motivate column liquids to and from the column sections. In this regard, device 72 might be a mechanical pump that serves as both a reflux pump and a pressure manipulation device.

[0050] In a further possible embodiment of the present invention, other oxygen-enriched fluid might be extracted from rectification column 14 and added for the make-up of oxygen-enriched liquid stream 30 for purposes of temperature control or to reduce the size of first heat exchanger 36, second heat exchanger 50 and third heat exchanger 56.

[0051] It is also possible to utilize certain aspects of the present invention without the direct use of heat exchanger 12 or 12' (or preceding compression and pre-purification equipment). For example, a conventional single column nitrogen generator could be utilized to generate the purified, pressurized and cooled stream. Such a stream could be obtained from the vaporized oxygen-enriched bottoms extracted from the condenser in association with the single column nitrogen generator. In this regard, the single column nitrogen generator would pref-

erably operate within a pressure range of between 10 and about 20 bar. Rectification column 14 would operate as described above. Nitrogen product and waste streams would pass through the main heat exchanger in a manner similar to that described with respect to Figure 1.

[0052] While the present invention has been described with reference to a preferred embodiment, as will occur to those skilled in the art, numerous changes and additions can be made without departing from the scope of the present invention as set forth in the presently pending claims.

Claims

1. A method of separating a gaseous mixture (10) comprising nitrogen and oxygen to produce a nitrogen product, said method comprising:

introducing a purified, pressurized and cooled gaseous stream (10) into a rectification column (14) to produce an overhead nitrogen-rich vapor (26) and an oxygen-enriched liquid bottoms (16, 30);

depressurizing a first oxygen-enriched liquid stream composed at least in part of the oxygen-enriched liquid bottoms (16), partially vaporizing the first oxygen-enriched liquid stream within a first heat exchanger (32), disengaging a vapor phase from a liquid phase (42) formed by the partial vaporization of the first oxygen-enriched liquid stream, depressurizing a second oxygen-enriched liquid stream (46) composed at least in part of the liquid phase and partially vaporizing the second oxygen-enriched liquid stream through indirect heat exchange with at least a portion of a vapor phase stream composed of the vapor phase within a second heat exchanger (50), thereby substantially condensing at least a portion of the vapor phase stream (52) to form a nitrogen-rich liquid stream (54) ;

condensing a first part of a column overhead nitrogen-rich stream (20) composed of the overhead nitrogen-rich vapor in the first heat exchanger (32), condensing a second part (28) of the overhead nitrogen-rich stream in a third heat exchanger (36) through indirect heat exchange with the second oxygen-enriched liquid stream after having been partially vaporized, thereby further vaporizing the second oxygen-enriched liquid stream, and returning at least part of the condensed column overhead nitrogen-rich stream to the rectification as reflux (29) ;

introducing at least part of the nitrogen-rich liquid stream (54) into the rectification column (14), above the purified, pressurized and cooled gaseous stream; and

producing a product nitrogen stream from part

of the overhead nitrogen-rich vapor.

2. The method of claim 1, further comprising:

subcooling the first oxygen-enriched liquid stream (30) through indirect heat exchange with the nitrogen product stream and a waste stream (60) composed of a vaporized fraction of the second oxygen-enriched liquid stream after having been further vaporized; and
cooling the compressed and purified stream, composed of the gaseous mixture, by indirect heat exchange with the nitrogen product stream and the waste stream after having subcooled the oxygen-enriched stream, thereby to form at least a portion of the purified, pressurized and cooled stream from the compressed and purified stream.

3. The method of claim 2, wherein:

the waste stream (60) and the nitrogen product stream indirectly exchange heat with the compressed and purified stream within a main heat exchanger (12);
the waste stream (60) is partially warmed within the main heat exchanger (12) and is then expanded with the performance of work to generate an exhaust stream; and
the exhaust stream is reintroduced into the main heat exchanger and fully warmed to refrigerate the cryogenic rectification process.

4. The method of claim 1, wherein pressure of the nitrogen-rich liquid stream is adjusted prior to its being introduced into the rectification column (14), preferably by mechanically pumping the nitrogen-rich liquid stream or by valve expanding the nitrogen-rich liquid stream.

5. The method of claim 1, wherein:

a first part of the vapor phase stream is substantially condensed within the second heat exchanger (50) to form the nitrogen-rich liquid stream; and
a second part of the vapor phase stream is warmed, compressed and cooled and recycled back to the rectification column (14).

6. The method of claim 5, wherein:

the first oxygen-enriched liquid stream is subcooled through indirect heat exchange with the nitrogen product stream and the waste stream (60); and
the compressed and purified stream is combined with the second part of the vapor phase

stream after having been compressed to form a combined, compressed and purified stream and the combined compressed and purified stream is cooled by indirect heat exchange with the nitrogen product stream and the waste stream (60) after having subcooled the first oxygen-enriched liquid stream and the second part of the nitrogen-rich vapor stream prior to its compression, thereby to form the purified, pressurized and cooled stream from the combined compressed and purified stream;
whereby the second part of the vapor phase stream is cooled and recycled back to the rectification column (14) by being combined with the compressed and purified stream.

7. The method of claim 6, wherein:

the waste stream (60), the nitrogen product stream and second part of the vapor phase stream indirectly exchange heat with the compressed and purified stream within a main heat exchanger (12);
the waste stream is partially warmed within the main heat exchanger (12) and is then expanded with the performance of work to generate an exhaust stream; and
the exhaust stream is reintroduced into the main heat exchanger (12) and fully warmed to refrigerate the cryogenic rectification process.

8. An apparatus for separating a gaseous mixture (10) comprising nitrogen and oxygen to produce a nitrogen product, said apparatus comprising:

a rectification column (14) connected to a main heat exchanger (12) for rectifying a purified, pressurized and cooled stream composed of the gaseous mixture to produce an overhead nitrogen-rich vapor and an oxygen-enriched liquid bottoms;
a first valve (34) for depressurizing a first oxygen-enriched liquid stream composed at least in part of the oxygen-enriched liquid bottoms;
a first heat exchanger (36) connected to the first valve for partially vaporizing the first oxygen-enriched liquid stream;
a phase separator (40) connected to the first heat exchanger for disengaging a vapor phase from a liquid phase formed by the partial vaporization of the first oxygen-enriched liquid stream;
a second valve (48) connected to the phase separator for depressurizing a second oxygen-enriched liquid stream composed at least in part of the liquid phase; (50)
a second heat exchanger (50) connected to the second valve and to the phase separator for partially vaporizing the second oxygen-enriched liq-

uid stream through indirect heat exchange with at least a portion of a vapor phase stream composed of the vapor phase, thereby to substantially condense at least a portion of the vapor phase stream and to form a nitrogen-rich liquid stream;

a third heat exchanger (58) connected to the second heat exchanger for further vaporizing the second oxygen-enriched liquid stream; the rectification column (14) connected to the first heat exchanger (36) and the third heat exchanger for condensing at least a portion of a column overhead nitrogen-rich stream composed of the overhead nitrogen-rich vapor and returning at least part of the column overhead nitrogen-rich stream after having been condensed to the rectification column as reflux; the second heat exchanger (50) connected to the rectification column (14) for introducing at least part of the nitrogen-rich liquid stream into the rectification column, above the purified, pressurized and cooled stream; and means for extracting a product nitrogen stream composed of part of the overhead nitrogen-rich vapor.

9. The apparatus of claim 8, further comprising:

a main heat exchanger (12) to cool a compressed and purified stream composed of the gaseous mixture (10) and thereby to form at least a portion of the purified, pressurized and cooled stream;

a subcooler (32) connected to the rectification column so that the first oxygen-enriched liquid stream (30) is subcooled through indirect heat exchange with the nitrogen product stream and a waste stream (60) composed of a vapor fraction of the second oxygen-enriched liquid stream after having been further vaporized; and the main heat exchanger (12) also connected to the subcooler and configured so that the compressed and purified stream is cooled by indirect heat exchange with the nitrogen product stream and the waste stream after having subcooled the first oxygen-enriched stream.

10. The apparatus of claim 9, wherein:

the main heat exchanger (12) is configured such that the waste stream (60) and the nitrogen product stream indirectly exchange heat with the compressed and purified stream, the waste stream partially warms within the main heat exchanger and an exhaust stream (78) fully warms within the main heat exchanger to refrigerate the apparatus; and an expander (76) is connected to the main heat

exchanger so that the waste stream after having partially warmed is expanded within the expander with the performance of work to generate the exhaust stream.

11. The apparatus of claim 8, wherein a pump (72) is interposed between the second heat exchanger and the rectification column to pressurize the nitrogen-rich liquid stream (54) after having been substantially condensed prior to its introduction into the rectification column.

12. The apparatus of claim 8, wherein a third valve (72) is interposed between the second heat exchanger (50) and the rectification column (14) to reduce the pressure of the nitrogen-rich liquid stream prior to its introduction into the rectification column.

13. The apparatus of claim 9, wherein:

the second heat exchanger (50) is connected to the phase separator (40) so that a first part of the vapor phase stream is substantially condensed within the second heat exchanger to form the nitrogen-rich liquid stream;

a compressor is connected in flow communication with the phase separator and to the main heat exchanger and the main heat exchanger also configured so that a second part of the vapor phase stream is warmed within the main heat exchanger and compressed within the compressor; and

the main heat exchanger is simultaneously in flow communication with the compressed and purified stream and the compressor such that the second part of the vapor phase stream combines with the compressed and purified stream to form a combined compressed and purified stream and the combined compressed and purified stream is cooled within the main heat exchanger to form the purified, pressurized and cooled stream.

14. The apparatus of claim 13, wherein the main heat exchanger (12) is also configured so that the combined compressed and purified stream is cooled by indirect heat exchange with the nitrogen product stream, the waste stream after having subcooled the first oxygen-enriched stream and the second part of the vapor phase stream.

15. The apparatus of claim 14, wherein:

the main heat exchanger (12) is configured such that the waste stream (60) and the nitrogen product stream indirectly exchange heat with the compressed and purified stream, the waste stream partially warms within the main heat ex-

changer and an exhaust stream fully warms within the main heat exchanger to refrigerate the apparatus; and
 an expander (76) is connected to the main heat exchanger so that the waste stream after having partially warmed is expanded within the expander with the performance of work to generate the exhaust stream.

Patentansprüche

1. Verfahren zum Trennen eines gasförmigen Gemisches (10), welches Stickstoff und Sauerstoff enthält, um ein Stickstoffprodukt zu erzeugen, wobei im Zuge des Verfahrens:

ein gereinigter, aufgedrückter und gekühlter Gasstrom (10) in eine Rektifikationskolonne (14) eingeleitet wird, um einen stickstoffreichen Überkopfdampf (26) und mit Sauerstoff angereicherte flüssige Sumpfprodukte (16, 30) zu erzeugen;

ein erster mit Sauerstoff angereicherter, flüssiger Strom, der mindestens zum Teil aus den mit Sauerstoff angereicherten flüssigen Sumpfprodukten (16) besteht, entspannt wird, der erste, mit Sauerstoff angereicherte, flüssige Strom innerhalb eines ersten Wärmetauschers (32) teilweise verdampft wird, eine Dampfphase von einer flüssigen Phase (42), die durch das teilweise Verdampfen des ersten, mit Sauerstoff angereicherten, flüssigen Stromes gebildet wurde, getrennt wird, ein zweiter, mit Sauerstoff angereicherter, flüssiger Strom (46), der mindestens zum Teil aus der flüssigen Phase besteht, entspannt wird, und der zweite, mit Sauerstoff angereicherte, flüssige Strom durch indirekten Wärmeaustausch mit mindestens einem Teil eines Dampfphasenstromes, der aus der Dampfphase besteht, innerhalb eines zweiten Wärmetauschers (50) teilweise verdampft wird, wodurch mindestens ein Teil des Dampfphasenstromes (52) im Wesentlichen kondensiert wird, um einen mit Stickstoff angereicherten flüssigen Strom (54) zu bilden;

ein erster Teil eines stickstoffreichen Kolonnenüberkopfstromes (20) der aus den stickstoffreichen Überkopfdampf besteht, in dem ersten Wärmetauscher (32) kondensiert wird, ein zweiter Teil (28) des stickstoffreichen Überkopfstromes in einem dritten Wärmetauscher (36) mittels indirektem Wärmeaustausch mit dem zweiten, mit Sauerstoff angereicherten, flüssigen Strom kondensiert wird, nachdem dieser teilweise verdampft wurde, wodurch der zweite mit Sauerstoff angereicherte flüssige Strom weiter verdampft wird, und mindestens ein Teil des

kondensierten, stickstoffreichen, Kolonnenüberkopfstromes als Rücklauf (29) zu der Rektifikation zurückgeführt wird;
 mindestens ein Teil des stickstoffreichen, flüssigen Stromes (54) in die Rektifikationskolonne (14) oberhalb des gereinigten, aufgedrückten und gekühlten gasförmigen Stromes eingeleitet wird; und
 ein Produktstickstoffstrom aus einem Teil des stickstoffreichen Überkopfdampfes erzeugt wird.

2. Verfahren gemäß Anspruch 1, bei dem ferner:

der erste mit Sauerstoff angereicherte flüssige Strom (30) durch indirekten Wärmeaustausch mit dem Stickstoffproduktstrom und einem Abstrom (60), der aus einem verdampften Teil des zweiten mit Sauerstoff angereicherten flüssigen Stroms nach dessen weiterer Verdampfung besteht, unterkühlt wird; und
 der verdichtete und gereinigte Strom, der aus dem gasförmigen Gemisch besteht, mittels indirektem Wärmeaustausch mit dem Stickstoffproduktstrom und dem Abstrom, nachdem dieser den mit Sauerstoff angereicherten Strom unterkühlt hat, gekühlt wird, um mindestens einen Teil des gereinigten, aufgedruckten und gekühlten Stromes aus dem verdichteten und gereinigten Strom zu bilden.

3. Verfahren nach Anspruch 2, bei welchem:

der Abstrom (60) und der Stickstoffproduktstrom mit dem verdichteten und gereinigten Strom innerhalb eines Hauptwärmetauschers (12) indirekt Wärme tauschen;
 der Abstrom (60) innerhalb des Hauptwärmetauschers (12) teilweise erwärmt und dann unter Verrichtung von Arbeit expandiert wird, um einen Abgasstrom zu bilden; und
 der Abgasstrom erneut in den Hauptwärmetauscher eingeführt und vollständig erwärmt wird, um dem Tieftemperaturrektifikationsprozess Kälte zuzuführen.

4. Verfahren gemäß Anspruch 1, bei welchem der Druck des stickstoffreichen flüssigen Stromes eingestellt wird, bevor dieser in die Rektifikationskolonne (14) eingebracht wird, vorzugsweise durch mechanisches Pumpen des stickstoffreichen flüssigen Stromes oder durch Expandieren des stickstoffreichen flüssigen Stromes durch ein Ventil.

5. Verfahren gemäß Anspruch 1, bei welchem:

ein erster Teil des Dampfphasenstromes innerhalb des zweiten Wärmetauschers (50) im We-

sentlichen kondensiert wird, um den stickstoffreichen flüssigen Strom zu bilden; und ein zweiter Teil des Dampfphasenstromes erwärmt, verdichtet und gekühlt und zu der Rektifikationskolonne (14) zurückgeführt wird.

6. Verfahren gemäß Anspruch 5, bei welchem:

der erste mit Sauerstoff angereicherte flüssige Strom durch indirekten Wärmeaustausch mit dem Stickstoffproduktstrom und dem Abstrom (60) unterkühlt wird; und
der verdichtete und gereinigte Strom mit dem zweiten Teil des Dampfphasenstromes, nachdem dieser verdichtet wurde, kombiniert wird, um einen kombinierten, verdichteten und gereinigten Strom zu bilden, und der kombinierte, verdichtete und gereinigte Strom mittels indirektem Wärmeaustausch mit dem Stickstoffproduktstrom und dem Abstrom (60), nachdem dieser den ersten mit Sauerstoff angereicherten flüssigen Strom und den zweiten Teil des stickstoffreichen Dampfstromes vor dessen Verdichtung unterkühlt hat, gekühlt wird, umso den gereinigten, aufgedrückten und gekühlten Strom aus dem vereinigten, verdichteten und gereinigten Strom zu bilden;
wobei der zweite Teil des Dampfphasenstromes gekühlt und zu der Rektifikationskolonne (14) zurückgeführt wird, indem dieser mit dem verdichteten und gereinigten Strom vereinigt wird.

7. Verfahren gemäß Anspruch 6, bei welchem:

der Abstrom (60), der Stickstoffproduktstrom und der zweite Teil des Dampfphasenstromes innerhalb eines Hauptwärmetauschers (12) mit dem verdichteten und gereinigten Strom indirekt Wärme tauschen;
der Abstrom innerhalb des Hauptwärmetauschers (12) teilweise erwärmt wird und dann unter Verrichtung von Arbeit expandiert wird, um einen Abgasstrom zu bilden; und
der Abgasstrom erneut in den Hauptwärmetauscher (12) eingeleitet und vollständig erwärmt wird, um dem Tieftemperaturrektifikationsverfahren Kälte zuzuführen.

8. Vorrichtung zum Trennen eines gasförmigen Gemisches (10), welches Stickstoff und Sauerstoff enthält, um ein Stickstoffprodukt zu erzeugen, wobei die Vorrichtung versehen ist mit;
einer Rektifikationskolonne (14), die mit einem Hauptwärmetauscher (12) verbunden ist, zum Rektifizieren eines gereinigten, unter Druck stehenden und gekühlten Stromes, der aus dem gasförmigen Gemisch besteht, um einen stickstoffreichen Überkopfdampf und mit Sauerstoff angereicherte flüssige

Sumpfprodukte zu erzeugen;
einem ersten Ventil (34) zum Entspannen eines ersten mit Sauerstoff angereicherten flüssigen Stromes, der sich zumindest zum Teil aus den mit Sauerstoff angereicherten flüssigen Sumpfprodukten zusammensetzt;
einem ersten Wärmetauscher (36), der mit dem ersten Ventil verbunden ist, zum teilweisen Verdampfen des ersten mit Sauerstoff angereicherten flüssigen Stroms;
einem Phasenabscheider (40), der mit dem ersten Wärmetauscher verbunden ist, um eine Dampfphase von einer flüssigen Phase abzutrennen, die durch das teilweise Verdampfen des ersten mit Sauerstoff angereicherten flüssigen Stromes gebildet wurde;
einem zweiten Ventil (48), welches mit den Phasenabscheider verbunden ist, um einen zweiten mit Sauerstoff angereicherten flüssigen Strom, der mindestens zum Teil aus der flüssigen Phase besteht, zu entspannen;
einem zweiten Wärmetauscher (50), der mit dem zweiten Ventil und dem Phasenabscheider verbunden ist, zum teilweisen Verdampfen des zweiten mit Sauerstoff angereicherten flüssigen Stroms mittels indirektem Wärmeaustausch mit mindestens einem Teil eines Dampfphasenstromes, der aus der Dampfphase besteht, um **dadurch** mindestens einen Teil des Dampfphasenstromes im Wesentlichen zu kondensieren und einen stickstoffreichen flüssigen Strom zu bilden;
einem dritten Wärmetauscher (58), der mit dem zweiten Wärmetauscher verbunden ist, um den zweiten mit Sauerstoff angereicherten flüssigen Strom weiter zu verdampfen; wobei die Rektifikationskolonne (14) mit dem ersten Wärmetauscher (36) und dem dritten Wärmetauscher verbunden ist, um mindestens einen Teil eines stickstoffreichen Kolonnenüberkopfstromes zu kondensieren, der aus dem stickstoffreichen Überkopf dampf besteht, und mindestens einen Teil des stickstoffreichen Kolonnenüberkopf stromes nach dessen Kondensation als Rückfluss zu der Rektifikationskolonne zurückzuführen;
wobei der zweite Wärmetauscher (50) mit der Rektifikationskolonne (14) verbunden ist, um mindestens einen Teil des stickstoffreichen flüssigen Stromes in die Rektifikationskolonne oberhalb des gereinigten, aufgedrückten und gekühlten Stromes einzubringen; und
Mitteln zum Ableiten eines Produktstickstoffstromes, der aus einem Teil des stickstoffreichen Überkopfdampfes besteht.

9. Vorrichtung gemäß Anspruch 8, ferner versehen mit:

einem Hauptwärmetauscher (12) zum Kühlen eines verdichteten und gereinigten Stromes, der aus dem gasförmigen Gemisch (10) besteht, um

dadurch mindestens einen Teil des gereinigten, aufgedrückten und gekühlten Stromes zu bilden;

einem Unterkühler (32), der mit der Rektifikationskolonne so verbunden ist, dass der erste mit Sauerstoff angereicherte flüssige Strom (30) durch indirekten Wärmeaustausch mit dem Stickstoffproduktstrom und einem Abstrom (60), unterkühlt wird, der aus einem Dampfanteil des zweiten mit Sauerstoff angereicherten flüssigen Stroms nach dessen weiteren Verdampfen besteht; und

wobei der Hauptwärmetauscher (12) ferner mit dem Unterkühler verbunden ist und so konfiguriert ist, dass der verdichtete und gereinigte Strom durch indirekten Wärmeaustausch mit dem Stickstoffproduktstrom und dem Abstrom gekühlt wird, nachdem dieser den ersten mit Sauerstoff angereicherten Strom unterkühlt hat.

10. Vorrichtung gemäß Anspruch 9, bei welcher:

der Hauptwärmetauscher (12) so konfiguriert ist, dass der Abstrom (60) und der Stickstoffproduktstrom mit dem verdichteten und gereinigten Strom indirekt Wärme tauschen, wobei der Abstrom sich innerhalb des Hauptwärmetauschers teilweise erwärmt und sich ein Abgasstrom (78) innerhalb des Hauptwärmetauschers vollständig erwärmt, um der Vorrichtung Kälte zuzuführen; und

wobei ein Expander (76) mit dem Hauptwärmetauscher so verbunden ist, dass der Abstrom nach dessen teilweiser Erwärmung innerhalb des Expanders unter Leistung von Arbeit expandiert wird, um den Abgasstrom zu erzeugen.

11. Vorrichtung gemäß Anspruch 8, wobei zwischen dem zweiten Wärmetauscher und der Rektifikationskolonne eine Pumpe (72) vorgesehen ist, um den stickstoffreichen flüssigen Strom (54) aufzudrücken, nachdem dieser im Wesentlichen kondensiert wurde bevor er in die Rektifikationskolonne eingeleitet wird.

12. Vorrichtung gemäß Anspruch 8, bei welcher ein drittes Ventil (72) zwischen dem zweiten Wärmetauscher (50) und der Rektifikationskolonne (14) vorgesehen ist, um den Druck des stickstoffreichen flüssigen Stromes vor dessen Einleitung in die Rektifikationskolonne zu vermindern.

13. Vorrichtung gemäß Anspruch 9, bei welcher:

der zweite Wärmetauscher (50) so mit dem Phasenabscheider (40) verbunden ist, dass ein erster Teil des Dampfphasenstromes innerhalb des zweiten Wärmetauschers im Wesentlichen kondensiert wird, um den stickstoffreichen flüs-

sigen Strom zu bilden;

ein Kompressor in Strömungsverbindung mit dem Phasenabscheider und dem Hauptwärmetauscher angeschlossen ist, wobei der Hauptwärmetauscher ferner so konfiguriert ist, dass ein zweiter Teil des Dampfphasenstromes innerhalb des Hauptwärmetauschers erwärmt wird und innerhalb des Kompressors verdichtet wird; und

der Hauptwärmetauscher gleichzeitig in Strömungsverbindung mit dem verdichteten und gereinigten Strom und dem Kompressor steht, so dass der zweite Teil des Dampfphasenstromes sich mit dem verdichteten und gereinigten Strom verbindet, um einen kombinierten verdichteten und gereinigten Strom zu bilden, und der kombinierte, verdichtete, und gereinigte Strom innerhalb des Hauptwärmetauschers gekühlt wird, um den gereinigten, aufgedrückten und gekühlten Strom zu bilden.

14. Vorrichtung gemäß Anspruch 13, bei welcher der Hauptwärmetauscher (12) ferner so konfiguriert ist, dass der kombinierte, verdichtete und gereinigte Strom durch indirekten Wärmeaustausch mit dem Stickstoffproduktstrom, dem Abstrom nachdem dieser den ersten mit Sauerstoff angereicherten Strom unterkühlt hat, und dem zweiten Teil des Dampfphasenstromes gekühlt wird.

15. Vorrichtung gemäß Anspruch 14, bei welcher:

der Hauptwärmetauscher (12) so konfiguriert ist, dass der Abstrom (60) und der Stickstoffproduktstrom mit dem verdichteten und gereinigten Strom indirekt Wärme tauschen, wobei der Abstrom sich teilweise innerhalb des Hauptwärmetauschers erwärmt und sich ein Abgasstrom innerhalb des Hauptwärmetauschers vollständig erwärmt, um der Vorrichtung Kälte zuzuführen; und

ein Expander (76) mit dem Hauptwärmetauscher so verbunden ist, dass der Abstrom, nachdem dieser sich teilweise erwärmt hat, innerhalb des Expanders unter Leistung von Arbeit expandiert wird, um den Abgasstrom zu bilden.

Revendications

1. Procédé pour séparer un mélange gazeux (10) comprenant de l'azote et de l'oxygène afin de former un produit consistant en azote, ledit procédé comprenant :

l'introduction d'un courant gazeux purifié, mis et sous pression et refroidi (10) dans une colonne de rectification (14) pour produire une vapeur

riche en azote de tête (26) et un résidu liquide enrichi en oxygène (16, 30) ;
la réduction de pression d'un premier courant liquide enrichi en oxygène constitué au moins en partie du résidu liquide enrichi en oxygène (16), la vaporisation partielle du premier courant liquide enrichi en oxygène dans un premier échangeur de chaleur (32), le désengagement d'une phase vapeur d'une phase liquide (42), formée par la vaporisation partielle du premier courant liquide enrichi en oxygène, la réduction de pression d'un second courant liquide enrichi en oxygène (46) constitué au moins en partie de la phase liquide et la vaporisation partielle du second courant liquide enrichi en oxygène par échange indirect de chaleur avec au moins une partie d'un courant en phase vapeur constitué de la phase vapeur dans un deuxième échangeur de chaleur (50), en condensant ainsi substantiellement au moins une partie du courant en phase vapeur (52) pour former un courant liquide riche en azote (54) ;
la condensation d'une première partie d'un courant riche en azote de tête de colonne (20) constitué de la vapeur riche en azote de tête dans le premier échangeur de chaleur (32), la condensation d'une seconde partie (28) du courant riche en azote de tête dans un troisième échangeur de chaleur (36) par échange indirect de chaleur avec le second courant liquide enrichi en oxygène après sa vaporisation partielle, ce qui provoque une vaporisation supplémentaire du second courant liquide enrichi en oxygène, et le retour d'au moins une partie du courant enrichi en azote de tête de colonne condensé à la rectification, en tant que reflux (29) ;
l'introduction d'au moins une partie du courant liquide riche en azote (54) dans la colonne de rectification (14), au-dessus du courant gazeux purifié, mis sous pression et refroidi ; et
la formation comme produit d'un courant d'azote à partir d'une partie de la vapeur riche en azote de tête.

2. Procédé suivant la revendication 1, comprenant en outre :

le sous-refroidissement du premier courant liquide enrichi en oxygène (30) par échange indirect de chaleur avec le courant de produit consistant en azote et un courant résiduel (60) constitué d'une fraction vaporisée du second courant liquide enrichi en oxygène après sa vaporisation supplémentaire ; et
le refroidissement du courant comprimé et purifié, constitué du mélange gazeux, par échange indirect de chaleur avec le courant de produit consistant en azote et le courant résiduel après

sous-refroidissement du courant enrichi en oxygène, pour former ainsi au moins une partie du courant purifié mis sous pression et refroidi à partir du courant comprimé et purifié.

3. Procédé suivant la revendication 2, dans lequel :

le courant résiduel (60) et le courant de produit consistant en azote échangent indirectement de la chaleur avec le courant comprimé et purifié dans un échangeur de chaleur principal (12) ; le courant résiduel (60) est chauffé partiellement dans l'échangeur de chaleur principal (12) et est ensuite soumis à une expansion avec pour but d'engendrer un courant d'échappement ; et le courant d'échappement est réintroduit dans l'échangeur de chaleur principal et chauffé totalement pour réfrigérer le procédé de rectification cryogénique.

4. Procédé suivant la revendication 1, dans lequel la pression du courant liquide riche en azote est ajustée avant l'introduction de ce courant dans la colonne de rectification (14), de préférence par pompage mécanique du courant liquide riche en azote ou par expansion par valve du courant liquide riche en azote.

5. Procédé suivant la revendication 1, dans lequel :

une première partie du courant en phase vapeur est condensée substantiellement dans le second échangeur de chaleur (50) pour former le courant liquide riche en azote ; et
une seconde partie du courant en phase vapeur est chauffée, comprimée et refroidie, puis recyclée à nouveau à la colonne de rectification (14).

6. Procédé suivant la revendication 5, dans lequel :

le premier courant liquide enrichi en oxygène est sous-refroidi par échange indirect de chaleur avec le courant de produit consistant en azote et le courant résiduel (60) ; et
le courant comprimé et purifié est combiné avec la seconde partie du courant en phase vapeur après sa compression pour former un courant combiné, comprimé et purifié et le courant combiné, comprimé et purifié est refroidi par échange indirect de chaleur avec le courant de produit consistant en azote et le courant résiduel (60) après sous-refroidissement du premier courant liquide enrichi en oxygène et de la seconde partie du courant de vapeur riche en azote avant sa compression, pour former ainsi le courant purifié, mis sous pression et refroidi à partir du courant combiné, comprimé et purifié ;
la seconde partie du courant en phase vapeur étant ainsi refroidie et recyclée à nouveau à la

colonne de rectification (14) en étant combinée avec le courant comprimé et purifié.

7. Procédé suivant la revendication 6, dans lequel :

le courant résiduel (60), le courant de produit consistant en azote et la seconde partie du courant en phase vapeur échangent indirectement de la chaleur avec le courant comprimé et purifié dans un échangeur de chaleur principal (12) ; le courant résiduel est partiellement chauffé dans l'échangeur de chaleur principal (12) et est ensuite soumis à une expansion avec pour but d'engendrer un courant d'échappement ; et le courant d'échappement est réintroduit dans l'échangeur de chaleur principal (12) et est chauffé totalement pour réfrigérer le procédé de rectification cryogénique.

8. Appareil pour séparer un mélange gazeux (10) comprenant de l'azote et de l'oxygène afin de former un produit consistant en azote, ledit appareil comprenant :

une colonne de rectification (14) connectée à un échangeur de chaleur principal (12) pour la rectification d'un courant purifié, mis sous pression et refroidi constitué du mélange gazeux afin de produire une vapeur riche en azote de tête et un résidu liquide enrichi en oxygène ; une première valve (34) pour la réduction de pression d'un premier courant liquide enrichi en oxygène constitué au moins en partie du résidu liquide enrichi en oxygène ; un premier échangeur de chaleur (36) connecté à la première valve pour la vaporisation partielle du premier courant liquide enrichi en oxygène ; un séparateur de phases (40) connecté au premier échangeur de chaleur pour le désengagement d'une phase vapeur à partir d'une phase liquide formée par la vaporisation partielle du premier courant liquide enrichi en oxygène ; une seconde valve (48) connectée au séparateur de phases pour la réduction de pression d'un second courant liquide enrichi en oxygène constitué au moins en partie de la phase liquide ; un deuxième échangeur de chaleur (50) connecté à la seconde valve et séparateur de phases pour la vaporisation partielle du second courant liquide enrichi en oxygène par échange indirect de chaleur avec au moins une partie d'un courant en phase vapeur constitué de la phase vapeur, pour condenser ainsi substantiellement au moins une partie du courant en phase vapeur et former un courant de liquide riche en azote ; un troisième échangeur de chaleur (58) connecté au deuxième échangeur de chaleur pour une vaporisation supplémentaire du second courant

liquide enrichi en oxygène ; la colonne de rectification (14) connectée au premier échangeur de chaleur (36) et au troisième échangeur de chaleur pour la condensation d'au moins une partie d'un courant riche en azote de tête de colonne constitué de la vapeur riche en azote de tête et le retour d'au moins une partie du courant riche en azote de tête de colonne après sa condensation à la colonne de rectification, en tant que reflux ; le deuxième échangeur de chaleur (50) connecté à la colonne de rectification (14) pour l'introduction d'au moins une partie du courant liquide riche en azote dans la colonne de rectification, au-dessus du courant purifié, mis sous pression et refroidi ; et des moyens pour extraire un courant de produit consistant en azote, formé d'une partie de la vapeur riche en azote de tête.

9. Appareil suivant la revendication 8, comprenant en outre :

un échangeur de chaleur principal (12) pour refroidir un courant comprimé et purifié constitué du mélange gazeux (10) et pour former ainsi au moins une partie du courant purifié, mis sous pression et refroidi ; un sous-refroidisseur (32) connecté à la colonne de rectification de telle sorte que le premier courant liquide enrichi en oxygène (30) subisse un sous-refroidissement par échange indirect de chaleur avec le courant de produit consistant en azote et un courant résiduel (60) constitué d'une fraction de vapeur du second courant liquide enrichi en oxygène après sa vaporisation supplémentaire ; et l'échangeur de chaleur principal (12) connecté également au sous-refroidisseur est configuré de telle sorte que le courant comprimé et purifié soit refroidi par échange indirect de chaleur avec le courant de produit consistant en azote et le courant résiduel après le sous-refroidissement du premier courant enrichi en oxygène.

10. Appareil suivant la revendication 9, dans lequel :

l'échangeur de chaleur principal (12) est configuré de telle sorte que le courant résiduel (60) et le courant de produit consistant en azote échangent indirectement de la chaleur avec le courant comprimé et purifié, le courant résiduel se réchauffe partiellement dans l'échangeur de chaleur principal et un courant d'échappement (78) se réchauffe totalement dans l'échangeur de chaleur principal pour la réfrigération de l'appareil ; et un dispositif d'expansion (76) est connecté à

l'échangeur de chaleur principal de telle sorte que le courant résiduel après son réchauffement partiel subisse une expansion dans le dispositif d'expansion avec pour but d'engendrer le courant d'échappement.

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11. Appareil suivant la revendication 8, dans lequel une pompe (72) est interposée entre le deuxième échangeur de chaleur et la colonne de rectification pour la mise sous pression du courant liquide enrichi en azote (54) après sa condensation substantielle avant son introduction dans la colonne de rectification.

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12. Appareil suivant la revendication 8, dans lequel une troisième valve (72) est interposée entre le deuxième échangeur de chaleur (50) et la colonne de rectification (14) pour réduire la pression du courant liquide riche en azote avant son introduction dans la colonne de rectification.

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13. Appareil suivant la revendication 9, dans lequel :

le deuxième échangeur de chaleur (50) est connecté au séparateur de phases (40) de telle sorte qu'une première partie du courant en phase vapeur soit condensée substantiellement dans le deuxième échangeur de chaleur pour former le courant liquide riche en azote ;

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un compresseur est connecté en communication par écoulement au séparateur de phases et à l'échangeur de chaleur principal et l'échangeur principal est configuré également de telle sorte qu'une seconde partie du courant en phase vapeur soit réchauffée dans l'échangeur de chaleur principal et comprimée dans le compresseur ; et

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l'échangeur de chaleur principal est simultanément en communication par écoulement avec le courant comprimé et purifié et le compresseur de telle sorte que la seconde partie du courant en phase vapeur se combine avec le courant comprimé et purifié pour former un courant combiné, comprimé et purifié et le courant combiné, comprimé et purifié est refroidi dans l'échangeur de chaleur principal pour former le courant purifié, mis sous pression et refroidi.

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14. Appareil suivant la revendication 13, dans lequel l'échangeur de chaleur principal (12) est configuré également de telle sorte que le courant combiné, comprimé et purifié soit refroidi par échange indirect de chaleur avec le courant de produit consistant en azote, le courant résiduel après sous-refroidissement du premier courant enrichi en oxygène et de la seconde partie du courant en phase vapeur.

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15. Appareil suivant la revendication 14, dans lequel :

l'échangeur de chaleur principal (12) est configuré de telle sorte que le courant résiduel (60) et le courant de produit consistant en azote échangent indirectement de la chaleur avec le courant comprimé et purifié, le courant résiduel se réchauffe partiellement dans l'échangeur de chaleur principal et un courant d'échappement se réchauffe totalement dans l'échangeur de chaleur principal pour la réfrigération de l'appareil ; et un dispositif d'expansion (76) est connecté à l'échangeur de chaleur principal de telle sorte que le courant résiduel après son réchauffement partiel subisse une expansion dans le dispositif d'expansion avec pour but d'engendrer le courant d'échappement.

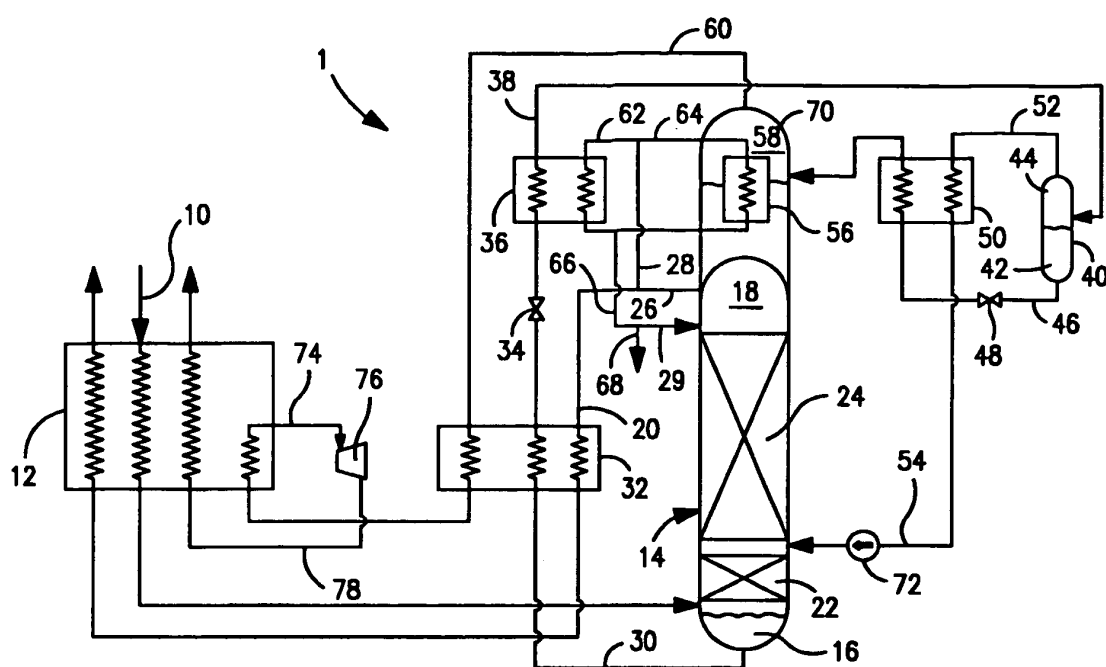


FIG. 1

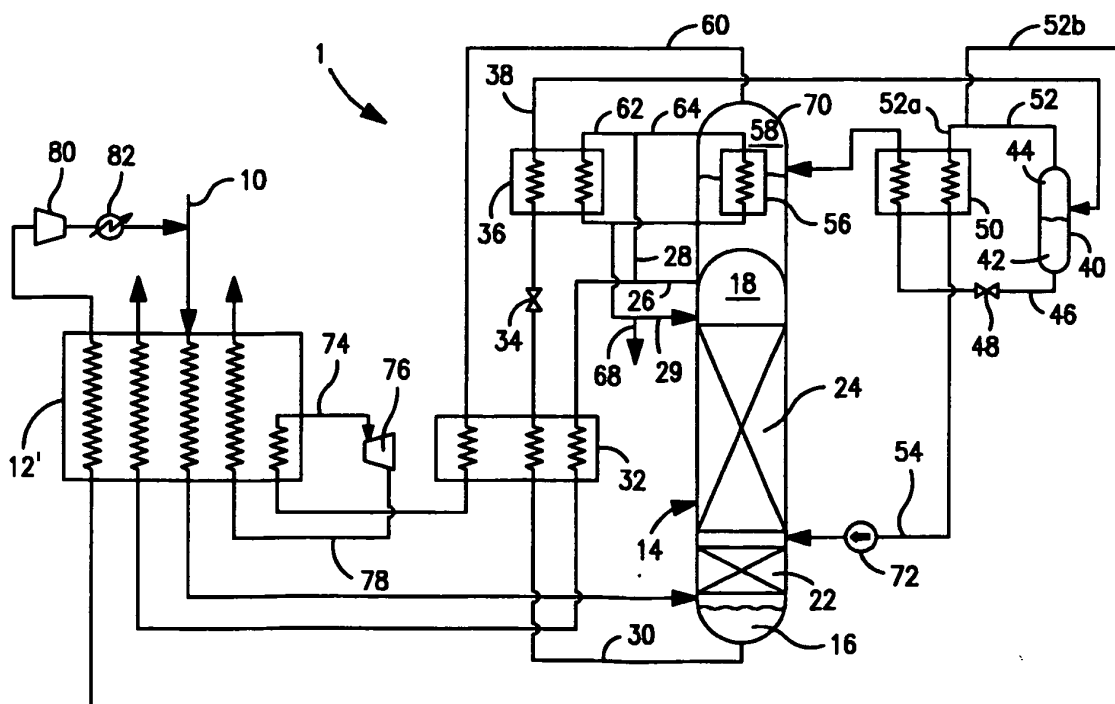


FIG. 2

REFERENCES CITED IN THE DESCRIPTION

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