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(54) **Process and apparatus for the production of low pressure gaseous oxygen**

Vorrichtung und Verfahren zur Herstellung von gasförmigem Sauerstoff unter niedrigem Druck

Procédé et dispositif de production d'oxygène gazeux à basse pression

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US-A- 5 505 052 **US-A- 5 941 098**

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Description

[0001] The present invention relates generally to the production of oxygen gas ("GOX") and, in particular, to the production of low pressure GOX by the cryogenic distillation of air.

[0002] There is a considerable market, particularly in the glass and metallurgical industries, for low purity, e. g. from 90 to 98 wt %, low pressure, e. g. from 1.5 to 3.0 bar absolute (0.15 to 0.3 MPa), GOX. The GOX is used in processes requiring oxygen-enriched combustion in which the required pressure of the oxygen at the point of use is near atmospheric.

[0003] An O₂ vacuum swing absorption ("VSA") process is commonly used for applications requiring 90 to 93 % O₂. However, up to 98 % O₂ GOX is often required and, thus, cryogenic plant processes are also used. There are many prior public disclosures of processes using cryogenic distillation of air to produce a GOX product. A number of the disclosed processes use a liquid cryogen from an external source as a refrigerant. For example, in US-A-4853015 (Yoshino) and US-A-4732595 (Yoshino), liquid oxygen ("LOX") is injected into the low pressure column of a double column distillation system to provide refrigeration. In US-A-4732595, an expander is used to provide some of the refrigeration requirement of the process. The use of such an expander increases the overall capital and running costs of the process and, as such, is undesirable.

[0004] In US-A-5408831 (Guillard *et al*), air is distilled cryogenically in a double distillation column system without the use of an expander to provide a portion of the refrigeration duty. GOX at from 2 to 5 bar absolute (0.2 to 0.5 MPa) is taken from the low pressure ("LP") column of the distillation column system as product. It is an essential feature of the Guillard process that some refrigeration is provided by expansion of at least one gaseous product from an LP column of the distillation column system. Part of the refrigeration duty required to condense the feed air fed to the column system can be provided by LOX refrigerant from an external source. The refrigerant may be introduced into the LP column or into the GOX product at an intermediate location of the main heat exchanger. The actual temperature at which the LOX is introduced is chosen to minimise the risk of explosion of any hydrocarbon impurities.

[0005] In order to achieve the required pressure of the GOX product, the column system in US-A-5408831 is back-pressurised. As a result of the back-pressurisation of the column system, the air pressure is necessarily higher, at a pressure from 8 to 16 bar absolute (0.8 to 1.6 MPa), than that in processes without column back-pressurisation giving a significant power penalty of about 12% for a given air flow. Such a penalty represents an undesirable increase in operating cost especially when it is considered that power is the main operating cost of an air separation plant.

[0006] Processes producing low pressure GOX in

which air compressor power is minimised without adversely effecting both the overall capital and running costs are desirable. In this connection, it is known to provide at least part of the refrigeration duty required to cool and at least partially condense by heat exchange feed air prior to cryogenic distillation through the use of a LOX refrigerant from an external source.

[0007] US-A-5505052 (Ekins *et al*) discloses a process for the cryogenic distillation of air using a double column system having a high pressure ("HP") column and a LP column to produce GOX at a pressure of about 25 bar (2.5 MPa) for use in installations comprising, for example, electric arc furnaces adapted to produce stainless steel. Oxygen is withdrawn in liquid form from the base of the LP column, brought to the utilisation pressure by a pump and vaporised and reheated to about ambient temperature in the heat exchange line against the feed air. The gaseous oxygen is then fed to the installation.

[0008] A portion of the LOX withdrawn from the base of the column may be sent to storage, for example, during periods of low demand for GOX in the installation where it is kept until such time as the demand for GOX at the installation becomes high whereupon it is pumped to the utilisation pressure and vaporised and reheated to about ambient temperature in the heat exchange line against the feed air. The LOX from storage may travel through the same vaporisation passages through the heat exchange line as the LOX from the column system or it may travel through separate vaporisation passages. The gaseous oxygen is then fed to the installation.

[0009] Additional LOX may be added to the LOX in storage from tank trucks, for example, during prolonged periods of high demand for GOX. Alternatively, the storage facility may not be connected to the double column system and may be supplied only by tank trucks. In the exemplified embodiments of the process disclosed in Ekins *et al*, LOX, whether from the double column system or from storage, enters the heat exchange line at the cold end, i.e. the end at which cooled feed air exits the line.

[0010] In Ekins *et al*, both the LOX product from the distillation column and the additional LOX from storage are pumped to a pressure (about 25 bar (2.5 MPa)) that is substantially higher than the pressure (about 5 to 6 bar (0.5 to 0.6 MPa)) of the LP column. In addition, a portion of the total refrigeration duty requirement of the process is provided by an expander and a further portion is provided by the warming and evaporation of a stream of liquid argon.

[0011] There is a need for a process and apparatus for the production of low pressure GOX with lower capital and operating costs compared with existing processes. There is also a need for a process for the production of lower pressure GOX in which the risk of explosion resulting from deposition in the heat exchange line of impurities, for example hydrocarbons, CO₂ and N₂O, from LOX is reduced. This risk is discussed below in

more detail in the general description of the invention.

[0012] According to a first aspect of the present invention, there is provided a process for the production of GOX, said process comprising:

cooling and at least partially condensing feed air by heat exchange using heat exchange means having a warm end and a cold end to produce cooled and at least partially condensed feed air;
distilling said cooled and at least partially condensed feed air in a distillation column system to produce LOX product;
removing a stream of said LOX product from the distillation column system and vaporising said LOX product stream by heat exchange against the feed air to produce GOX; and

separately from the LOX product, vaporising LOX refrigerant from an external source by heat exchange against the feed air to produce vaporised refrigerant thereby providing a portion of the refrigeration duty required to cool and at least partially condense the feed air; said process comprising the LOX refrigerant being injected into the heat exchange means either:

- (a) at a pressure greater than that of the LOX product entering the heat exchange means; or
- (b) at a pressure substantially equal to that of the LOX product entering the heat exchange means and at an intermediate point between the warm and cold ends where the temperature of the heat exchange means is above the boiling temperature of the LOX refrigerant.

[0013] The heat exchange means comprises a "warm end" (or "hot end") and a "cold end". The warm end (or hot end) is the end at which the feed air enters the heat exchange means and the cold end is the end at which the cooled and at least partially condensed feed air leaves the heat exchange means. The terms "warm end" (or "hot end") and "cold end" are commonly used in the art to distinguish the two ends of heat exchange means by their relative temperatures.

[0014] GOX may be produced at a slightly elevated pressure by a known technique in which LOX product is withdrawn from the LP column of a double column system. LOX is withdrawn from the distillation column system and is vaporised and warmed by heat exchange against the feed air. A fraction of the feed air is condensed by heat exchange against the withdrawn LOX and, thus, there is less air vapour entering the distillation column system than there would otherwise be if the feed air were to be subjected to indirect heat exchange with GOX withdrawn from the column system. This has the effect of reducing the efficiency of the distillation when compared to a process in which GOX is withdrawn from the distillation column system. However, as only low purity GOX is required, there is no performance penalty as

about 99.7 % of the O₂ becomes product. Therefore, the process is surprisingly efficient.

[0015] One reason for using LOX as the refrigerant is that the vaporised LOX refrigerant may be combined with the GOX produced by the vaporisation of the LOX product to produce GOX product. In this way, there is no wastage of vaporised refrigerant.

[0016] Preferably, the distillation column system comprises a multiple column system having a higher pressure ("HP") column and a lower pressure ("LP") column thermally integrated by the condensation of nitrogen overhead from the HP column against liquid bottoms in the LP column. A portion of the condensed HP column nitrogen overhead may be subcooled by heat exchange to produce a subcooled nitrogen stream which can be fed to the LP column.

[0017] In preferred processes, substantially all of the refrigeration duty required to keep the plant in energy balance is provided by the LOX refrigerant. Preferably, no refrigeration duty is provided by expansion of a process stream. Any heat leak into the process via the insulation and the fact that the product streams leave the heat exchanger at a temperature that is slightly lower than the entry temperature of the feed air is taken into account in calculating the amount of LOX refrigerant required.

[0018] Surprisingly, vaporising and warming LOX refrigerant separately from the product LOX has little effect on the temperature profiles of the main heat exchanger and causes only a very small increase in the amount of the refrigerant. This is particularly apparent for a process of the invention in which the LOX refrigerant is vaporised and warmed at substantially the same pressure as the LOX product but is introduced to the heat exchanger at an intermediate point between the warm and cold ends of the heat exchanger.

[0019] The LOX refrigerant is vaporised separately from the LOX product to reduce the risk of any problems resulting from the build up of hydrocarbon impurities such as ethylene due to deposition of CO₂ and N₂O on the interior wall surfaces of the boiling passages through the heat exchanger.

[0020] Commercial sources of LOX refrigerant, e.g. produced by an air separation plant, will contain hydrocarbons, CO₂ and N₂O impurities. The concentration of these impurities in the LOX refrigerant will vary depending on the plant producing it, the mode of operation of the plant and the ratio of LOX produced to feed air entering the plant. Concentrations of about 1500 ppb (vol.) CO₂ and about 3000 ppb (vol.) N₂O are typical.

[0021] As the LOX product boils at about 2.2 bar absolute (0.22 MPa), the vapour phase solubility of CO₂ and N₂O impurities is about 50 ppb (vol.) and about 500 ppb (vol.) respectively. If the LOX refrigerant were to be introduced directly into the LOX product stream, the impurity concentration of the combined LOX stream would be sufficiently increased to warrant concern about the unwanted and dangerous build up of impurity deposits

in the heat exchanger. Even a very small amount of "slip-page" of CO₂ and N₂O from the air purification will cause the concentration of CO₂ and/or N₂O to exceed the vapour phase solubility limit and result in at least partial blockage of the heat exchanger by deposited CO₂ and N₂O.

[0022] In one embodiment, vaporising LOX refrigerant without causing blockage of the heat exchanger by CO₂ and N₂O deposits is achieved by vaporising the refrigerant at a greater pressure than the LOX product such that, at the boiling temperature of the LOX refrigerant, the CO₂ and N₂O impurity concentrations are below the vapour phase solubility limits.

[0023] In an alternative embodiment, blockage of the heat exchanger by unwanted impurity deposits is avoided by injecting the LOX refrigerant into the heat exchange means at a pressure that is substantially equal to the pressure of the LOX product as it enters the heat exchange means, provided that the point of injection is between the warm and cold ends of the heat exchange means. Preferably, the temperature of the heat exchange means at the intermediate point of injection is from -165°C to -80°C, i.e. substantially above the O₂ boiling temperature.

[0024] In this alternative embodiment, as the preferred temperature of the point of injection in the heat exchanger is relatively warm, CO₂ and N₂O solubilities are relatively high and deposition on the surfaces of the heat exchanger will not occur. For the preferred range of O₂ vaporisation pressure, i.e. about 1.5 bar absolute (0.15 MPa) to 3.0 bar absolute (0.3 MPa), vaporisation actually occurs at about -179°C to about -171°C respectively and any solid CO₂ and N₂O that form initially does not deposit on the metal but is carried onwards towards a warmer part of the heat exchanger and after a short distance the whole stream has reached or exceeded about -165°C to -80°C by which time CO₂ and N₂O will have sublimed into vapour and cannot precipitate.

[0025] In preferred embodiments of the process, the LOX product stream is pressurised before vaporising said stream by heat exchange to provide GOX. The LOX product stream may be pumped. Preferably, however, the LOX product stream is not pumped and, instead, pressurisation may be achieved hydrostatically by, for example piping the stream from the distillation column system to a lower elevation.

[0026] The pressure of the LOX product leaving the distillation column system is usually about 1.4 bar absolute (0.14 MPa). The pressure of the LOX refrigerant is preferably from 4 bar absolute (0.4 MPa) to 10 bar absolute (1.0 MPa). The pressure of the GOX product may be from 1.5 bar absolute (0.15 MPa) to 3.0 bar absolute (0.3 MPa), preferably from 1.8 bar absolute (0.18 MPa) to 2.5 bar absolute (0.25 MPa).

[0027] The process may further comprise combining LOX refrigerant with the cooled and at least partially condensed feed air to further cool the feed air, preferably during plant cooldown. The process may also comprise

introducing LOX refrigerant to the distillation column system under level control.

[0028] Preferably, the LOX refrigerant is provided by an air separation plant.

[0029] The process may further comprise withdrawing at least one nitrogen gas product stream from the distillation column system.

[0030] The feed air is preferably purified before heat exchange to reduce the CO₂ and N₂O impurity concentrations to a level which ensures that these impurity concentrations in the LOX product are below their vapour phase solubilities at the vaporising pressure and temperature heat exchange conditions. The feed air may be purified using, for example, either a temperature swing absorber system using alumina and CaX or a pressure swing adsorber system using alumina and 13X.

[0031] In a second aspect of the present invention, there is provided apparatus for carrying out the process of the first aspect of the present invention for producing gaseous oxygen, said apparatus comprising:

heat exchange means for cooling and at least partially condensing feed air to produce cooled and at least partially condensed feed air, said heat exchange means having a warm end and a cold end; a distillation column system for distilling cooled and at least partially condensed feed air to produce LOX product;

conduit means to carry cooled feed air from the heat exchange means to the distillation column system; and

conduit means to carry LOX product from the distillation column system to the heat exchange means;

said apparatus further comprising either:

(a) conduit means to carry LOX refrigerant at a greater pressure than the pressure of the LOX product entering the heat exchange means from an external supply to the heat exchange means but does not comprise an expander for expanding a process stream to provide refrigeration duty; or

(b) conduit means to carry LOX refrigerant at a pressure that is substantially equal to the pressure of the LOX product entering the heat exchange means from an external supply to an intermediate point between the warm and cold ends of the heat exchange means where the temperature of the heat exchange means is above the boiling temperature of the LOX refrigerant and the heat exchange means has separate circuits for LOX refrigerant and LOX product.

[0032] The apparatus is preferably adapted or constructed to carry out any combination of the preferred features of the process discussed above.

[0033] It is preferable to minimise the amount of LOX refrigerant consumed by the process as it is expensive. The amount required is highly dependent on the number

of transfer units (NTU) of the heat exchange means. NTU is defined as follows:

$$NTU = (T_{\text{airhot}} - T_{\text{aircold}}) / \text{mean DT}$$

Where

T_{airhot} = air temperature of hot end of heat exchanger;

T_{aircold} = air temperature of cold end of heat exchanger

mean DT = effective mean differential temperature between hot and cold stream in heat exchanger between air entry and exit.

[0034] The heat exchange means of the apparatus may have at least 55 NTU, preferably from 70 to 90 NTU and more preferably about 80 NTU.

[0035] The amount of LOX refrigerant consumed in the process is also very dependent on the heat gain through the insulation. Preferably, the cryogenic portion of the apparatus, i.e. the distillation column system and the heat exchange means, is vacuum insulated to reduce heat loss.

[0036] In particularly preferred embodiments, the LOX refrigerant passes through the heat exchange means via a separate circuit to the LOX product. The LOX refrigerant preferably passes through the heat exchange means via a single passage. Further, the LOX refrigerant is preferably introduced into the heat exchange means at an intermediate point between the cold and warm ends of the heat exchange means where the temperature of the metal of the heat exchange means is above the boiling temperature of the refrigerant.

[0037] The following is a description by way of example only and with reference to the accompanying drawing, which is a flowsheet of a presently preferred embodiment of the invention.

[0038] A purified and compressed feed air stream 1, having a concentration of CO_2 and N_2O low enough to prevent deposition in the main LOX circuit of the main heat exchanger E1, enters the main heat exchanger E1, preferably a plate-fin type, wherein it is cooled to a cryogenic temperature and at least partially condensed. An at least partially condensed feed air stream 2 is removed from the main heat exchanger E1 and fed to the high pressure column C1 in an double distillation column system C1, C2 having a reboiler condenser E2.

[0039] The feed air stream 2 is distilled in the high pressure column C1 and a nitrogen-rich vapour stream 3 is condensed in the condenser E2 to produce a condensed nitrogen-rich stream 13. A portion 5 of the condensed nitrogen-rich stream 13 is returned to the high pressure column C1 as reflux to purify gas rising and the remaining portion 4 is sent to the top of the low pressure column C2 via the main heat exchanger E1 where

it is subcooled. An oxygen-rich stream 6 is removed from the high pressure column C1 and fed to the low pressure column C2 at an intermediate location optionally via a heat exchanger to subcool the stream.

[0040] The two liquid streams 4, 6 entering the low pressure column C2 are distilled due to vapour rising from the reboiler E2. A low pressure waste nitrogen vapour stream 7 is withdrawn from the top of the low pressure column and warmed to ambient temperature in the main heat exchanger E1. A LOX product stream 8 is withdrawn from the bottom of the low pressure column C2 and either piped to a lower elevation to gain static pressure or pumped to gain pressure before being vaporised and then warmed to ambient temperature in the main heat exchanger E1 to form GOX stream 9. In this way, GOX at a pressure typically from 1.8 to 2.5 bar absolute (0.18 to 0.25MPa) may be obtained directly from the plant.

[0041] Even though the cryogenic part of the plant is vacuum insulated to minimise heat loss, some refrigeration must be supplied to maintain a refrigeration balance. A LOX refrigerant stream 10 is introduced to a separate circuit of the main heat exchanger E1 at an intermediate point between the warm and cold ends and at a pressure equal to or higher than that for the LOX product stream 8 in a manner to avoid the deposition of CO_2 and N_2O . The LOX refrigerant stream 10 is vaporised and warming to ambient temperature to produce a stream 11 of vaporised LOX refrigerant which is combined with the GOX stream 9 to form a GOX product stream 12.

[0042] In a specific example, an airflow of $10000 \text{ Nm}^3/\text{h}$ ($167 \text{ Nm}^3/\text{s}$) is compressed to about 6 bar absolute (0.6 MPa), purified, cooled in the main heat exchanger E1 and fed to the HP column C1 at 5.5 bar absolute (0.55 MPa). LOX refrigerant from an external source at a purity of about 99.8 % O_2 is injected into the main heat exchanger E1 at a flow rate of about $50 \text{ Nm}^3/\text{h}$ ($0.8 \text{ Nm}^3/\text{s}$) where it is vaporised and warmed to ambient temperature. LOX product at about 95 % O_2 purity and at a contained O_2 flow of $2090 \text{ Nm}^3/\text{h}$ ($35 \text{ Nm}^3/\text{s}$) leaves the low pressure column C2 at about 1.4 bar absolute (0.14 MPa). The pressure of the LOX product stream is increased by about 0.8 bar absolute (0.08 MPa) due to static head and after vaporisation and warming leaves the main heat exchanger at 2.0 bar absolute (0.2 MPa). The two warmed GOX streams are combined giving a contained O_2 flow of $2140 \text{ Nm}^3/\text{h}$ ($36 \text{ Nm}^3/\text{s}$) of GOX.

[0043] The economics of the present invention compare favourably with those of O_2 VSA plants at product flows above $870 \text{ Nm}^3/\text{h}$ ($15 \text{ Nm}^3/\text{s}$). The present invention has the same or lower gas cost without the much higher capital cost or reliability issues of the O_2 VSA plants. In addition, the economics of the present invention also compare favourably with those of a cryogenic plant with an expander at a capacity of about $3480 \text{ Nm}^3/\text{h}$ ($58 \text{ Nm}^3/\text{s}$). Again, the present invention is economic having the same gas cost and lower capital cost.

[0044] It is not obvious to introduce the LOX refrigerant into the heat exchanger at an intermediate point between the warm and cold ends to provide refrigeration for several reasons. First, it is less efficient thermodynamically to provide refrigeration by indirect heat exchange using a stream of LOX refrigerant injected into a heat exchanger separately from the LOX product rather than to provide equivalent refrigeration by injection of the LOX refrigerant directly into the LOX product stream before vaporisation in the heat exchanger. In addition, the design of a heat exchanger that is suitable for carrying out the invention is more complicated and therefore more expensive than a conventional heat exchanger. Further, it is simply inefficient thermodynamically to inject a LOX refrigerant stream into a warm part of a heat exchanger. One reason for injecting the LOX refrigerant in this way is to reduce the likelihood of an explosive energy release following the build up of hydrocarbons as a result of the deposition of dissolved impurities in the LOX.

[0045] Some of the advantages of the exemplified embodiment of the present invention are as follows:

- the distillation column system is not back-pressurised and hence the air pressure is minimised;
- GOX is produced with minimal air flow as substantially all of the O₂ in the feed air and all of the LOX refrigerant becomes GOX product;
- as a result of the lack of back-pressurisation and minimal air flow, the air compressor power is minimised;
- GOX is produced at about 2.0 bar absolute (0.2 MPa) directly from the cryogenic section; and
- as a result of using vacuum insulation and a main heat exchanger with at least 55 NTU, the amount of LOX refrigerant is minimised.

[0046] It will be appreciated that the invention is not restricted to the details described above with reference to the preferred embodiments but that numerous modifications and variations can be made without departing from the scope of the invention as defined in the following claims.

Claims

1. A process for the production of gaseous oxygen ("GOX"), said process comprising:

cooling and at least partially condensing feed air (1) by heat exchange using heat exchange means (E1) having a warm end and a cold end to produce cooled and at least partially condensed feed air (2);
distilling said cooled and at least partially condensed feed air (2) in a distillation column system (C1, C2) to produce liquid oxygen ("LOX")

product;
removing a stream (8) of said LOX product from the distillation column system (C1, C2) and vaporising said LOX product stream (8) by heat exchange (E1) against the feed air (1) to produce GOX; and
separately from the LOX product, vaporising LOX refrigerant (10) from an external source by heat exchange (E1) against the feed air (1) to produce vaporised refrigerant (11) thereby providing a portion of the refrigeration duty required to cool and at least partially condense the feed air;

said process comprising the LOX refrigerant being injected into the heat exchange means (E1) either:

- (a) at a pressure greater than that of the LOX product entering the heat exchange means; or
- (b) at a pressure substantially equal to that of the LOX product entering the heat exchange means and at an intermediate point between the warm and cold ends where the temperature of the heat exchange means (E1) is above the boiling temperature of the LOX refrigerant.

2. A process as claimed in Claim 1, wherein the temperature of the heat exchange means (E1) at the intermediate point at which the LOX refrigerant is injected is from -165°C to -80°C.
3. A process as claimed in Claim 1 or Claim 2 further comprising combining the vaporised LOX refrigerant (10) with the GOX (9) produced by the vaporisation of the LOX product to produce GOX product (12).
4. A process as claimed in any one of Claims 1 to 3 wherein the distillation column system comprises a multiple column system having a higher pressure ("HP") column (C1) and a lower pressure ("LP") column (C2) thermally integrated by the condensation of nitrogen overhead from the HP column against liquid bottoms in the LP column.
5. A process as claimed in any one of Claims 1 to 4, wherein no refrigeration duty is provided by expansion of a process stream.
6. A process as claimed in any one of Claims 1 to 5, wherein the LOX refrigerant (10) provides all of the external refrigeration duty required to keep the process in energy balance.
7. A process as claimed in any one of Claims 1 to 6 wherein the pressure of the LOX refrigerant (10) is from 0.4 MPa (4 bar absolute) to 1.0 MPa (10 bar absolute).

8. A process as claimed any one of Claims 1 to 7 further comprising pressurising the LOX product stream (8) before vaporising said stream by heat exchange (E1) to provide GOX (9).

9. A process as claimed in Claim 8, wherein the LOX product stream is not pressurised by pumping the stream.

10. A process as claimed in Claim 8 or Claim 9, wherein the LOX product stream is pressurised hydrostatically.

11. A process as claimed in Claim 8 wherein the LOX product stream is pressurised by pumping the stream.

12. A process as claimed in any one of Claims 1 to 11 wherein the pressure of the GOX product is from 0.15 MPa (1.5 bar absolute) to 0.3 MPa (3.0 bar absolute).

13. A process as claimed in any one of Claims 1 to 12 wherein the pressure of the GOX product (12) is from 0.18 MPa (1.8 bar absolute) to 0.25 MPa (2.5 bar absolute).

14. A process as claimed in any one of Claims 1 to 13 further comprising combining LOX refrigerant with the cooled and at least partially condensed feed air to further cool the feed air.

15. A process as claimed in any one of Claims 1 to 14 further comprising introducing LOX refrigerant to the distillation column system under level control.

16. A process as claimed in any one of Claims 1 to 15 wherein the LOX refrigerant is provided by an air separation plant.

17. Apparatus for carrying out the process of Claim 1 to produce gaseous oxygen, said apparatus comprising:

heat exchange means (E1) for cooling and at least partially condensing feed air (1) to produce cooled and at least partially condensed feed air (2), said heat exchange means (E1) having a warm end and a cold end;
a distillation column system (C1, C2) for distilling cooled and at least partially condensed feed air (2) to produce LOX product (8);
conduit means to carry the cooled and at least partially condensed feed air (2) from the heat exchange means (E1) to the distillation column system (C1, C2); and
conduit means to carry LOX product (8) from the distillation column system (C1, C2) to the

heat exchange means (E1);

said apparatus further comprising either:

(a) conduit means to carry LOX refrigerant (10) at a higher pressure than the pressure of the LOX product entering the heat exchange means from an external supply to the heat exchange means (E1) but does not comprise an expander for expanding a process stream to provide refrigeration duty; or
(b) conduit means to carry LOX refrigerant (10) at a pressure that is substantially equal to the pressure of the LOX product entering the heat exchange means from an external supply to an intermediate point between the warm and cold ends of the heat exchange means where the temperature of the heat exchange means is above the boiling temperature of the LOX refrigerant and the heat exchange means has separate circuits for LOX refrigerant and LOX product.

18. Apparatus as claimed in Claim 17 adapted or constructed to carry out the process as defined in any one of Claims 2 to 16.

19. Apparatus as claimed in Claim 17 or Claim 18 wherein the cryogenic portion of the apparatus is vacuum insulated to reduce heat loss.

20. Apparatus as claimed in any one of Claims 17 to 19 wherein the heat exchange means (E1) has at least 55 NTU.

21. Apparatus as claimed in any one of Claims 17 to 20 wherein the heat exchange means (E1) has from 70 to 90 NTU.

22. Apparatus as claimed in any one of Claims 17 to 21 wherein the heat exchange means (E1) has about 80 NTU.

23. Apparatus as claimed in any of Claims 17 to 22 wherein, in option (a), the heat exchange means has separate circuits for LOX refrigerant and LOX product.

24. Apparatus as claimed in any of Claims 17 to 23 wherein, in option (b), the apparatus does not comprise an expander for expanding a process stream to provide refrigeration duty.

25. Apparatus as claimed in any one of Claims 17 to 24 wherein the LOX refrigerant (10) passes through the heat exchange means (E1) via a single passage.

Patentansprüche

1. Verfahren für die Herstellung von gasförmigem Sauerstoff ("GOX" für "gaseous oxygen") mit:

Kühlen und wenigstens teilweise Kondensieren von Speiseluft (1) durch Wärmeaustausch unter Verwendung einer Wärmeaustauscheranordnung (E1) mit einem warmen Ende und einem kalten Ende, um gekühlte und wenigstens teilweise kondensierte Speiseluft (2) zu erzeugen;
 Destillieren der gekühlten und wenigstens teilweise kondensierten Speiseluft (2) in einem Destillationssäulensystem (C1, C2), um ein Flüssigsauerstoff- ("LOX" für "liquid oxygen") Produkt zu erzeugen;
 Entnehmen eines Stroms (8) des LOX-Produktes aus dem Destillationssäulensystem (C1, C2) und Verdampfen des LOX-Produkts (8) durch Wärmeaustausch (E1) gegen die Speiseluft (1), um GOX zu erzeugen; und
 Verdampfen - getrennt von dem LOX-Produkt - von LOX-Kühl- bzw. Kältemittel (10) aus einer externen Quelle durch Wärmeaustausch (E1) gegen die Speiseluft (1), um verdampftes Kühlmittel (11) zu erzeugen, wodurch ein Teil der Kühlleistung zur Verfügung gestellt wird, der zum Kühlen und zum wenigstens teilweise Kondensieren der Speiseluft benötigt wird;

wobei das Verfahren den Schritt aufweist, dass das LOX-Kühlmittel in die Wärmetauscheranordnung (E1) injiziert wird entweder:

- (a) bei einem Druck, der größer als der des LOX-Produktes ist, das in die Wärmetauscheranordnung eintritt; oder
 (b) bei einem Druck, der im wesentlichen gleich dem des LOX-Produktes ist, das in die Wärmetauscheranordnung eintritt, und zwar an einem Zwischenpunkt zwischen den warmen und kalten Enden, an dem die Temperatur der Wärmetauscheranordnung (E1) sich über der Siedetemperatur des LOX-Kühlmittels befindet.

2. Verfahren nach Anspruch 1, wobei die Temperatur der Wärmetauscheranordnung (E1) an dem Zwischenpunkt, an dem das LOX-Kühlmittel injiziert wird, von -165°C bis -80°C beträgt.
 3. Verfahren nach Anspruch 1 oder Anspruch 2, wobei das verdampfte LOX-Kühlmittel (10) mit dem GOX (9) kombiniert wird, das durch die Verdampfung des LOX-Produktes hergestellt wird, um das GOX-Produkt (12) herzustellen.

4. Verfahren nach einem der Ansprüche 1 bis 3, wobei das Destillationssäulensystem ein Mehrfachsäulensystem mit einer Säule (C1) mit höherem Druck ("HP" für "higher pressure") und einer Säule (C2) mit niedrigerem Druck ("LP" für "lower pressure") aufweist, die durch die Kondensation des Stickstoff-Kopfproduktes von der HP-Säule gegen das flüssige Bodenprodukt in der LP-Säule thermisch integriert sind.

5. Verfahren nach einem der Ansprüche 1 bis 4, wobei keine Kühlleistung durch die Expansion eines Verfahrensstroms zur Verfügung gestellt wird.

6. Verfahren nach einem der Ansprüche 1 bis 5, wobei das LOX-Kühlmittel (10) die gesamte externe Kühlleistung zur Verfügung stellt, die benötigt wird, um das Verfahren im Energie-Gleichgewicht zu halten.

7. Verfahren nach einem der Ansprüche 1 bis 6, wobei der Druck des LOX-Kühlmittels (10) von 0,4 MPa (4 bar absolut) bis 1,0 MPa (10 bar absolut) beträgt.

8. Verfahren nach einem der Ansprüche 1 bis 7, wobei weiterhin der LOX-Produktstrom (8) unter Druck gesetzt wird, bevor dieser Strom durch Wärmeaustausch (E1) verdampft wird, um GOX (9) zur Verfügung zu stellen.

9. Verfahren nach Anspruch 8, wobei der LOX-Produktstrom nicht durch Pumpen des Stroms unter Druck gesetzt wird.

10. Verfahren nach Anspruch 8 oder Anspruch 9, wobei der LOX-Produktstrom hydrostatisch unter Druck gesetzt wird.

11. Verfahren nach Anspruch 8, wobei der LOX-Produktstrom durch Pumpen des Stroms unter Druck gesetzt wird.

12. Verfahren nach einem der Ansprüche 1 bis 11, wobei der Druck des GOX-Produktes von 0,15 MPa (1,5 bar absolut) bis 0,3 MPa (3,0 bar absolut) beträgt.

13. Verfahren nach einem der Ansprüche 1 bis 12, wobei der Druck des GOX-Produktes (12) von 0,18 MPa (1,8 bar absolut) bis 0,25 MPa (2,5 bar absolut) beträgt.

14. Verfahren nach einem der Ansprüche 1 bis 13, weiterhin mit Kombinieren des LOX-Kühlmittels mit der gekühlten und wenigstens teilweise kondensierten Speiseluft, um die Speiseluft weiter zu kühlen.

15. Verfahren nach einem der Ansprüche 1 bis 14, weiterhin mit Einführen des LOX-Kühlmittels in das De-

stillationssäulensystem unter Niveau-Steuerung bzw. -Regelung.

16. Verfahren nach einem der Ansprüche 1 bis 15, wobei das LOX-Kühlmittel durch eine Luftzerlegungsanlage zur Verfügung gestellt wird.

17. Vorrichtung zur Durchführung des Verfahrens nach Anspruch 1 zur Erzeugung von gasförmigem Sauerstoff mit:

einer Wärmetauscheranordnung (E1) zum Kühlen und wenigstens teilweise Kondensieren von Speiseluft (1), um gekühlte und wenigstens teilweise kondensierte Speiseluft (2) zu erzeugen, wobei die Wärmetauscheranordnung (E1) ein warmes Ende und ein kaltes Ende hat;
einem Destillationssäulensystem (C1, C2) zum Destillieren der gekühlten und wenigstens teilweise kondensierten Speiseluft (2), um ein LOX-Produkt (8) zu erzeugen;
einer Leitungsanordnung zur Führung der gekühlten und wenigstens teilweise kondensierten Speiseluft (2) von der Wärmetauscheranordnung (E1) zu dem Destillationssäulensystem (C1, C2); und
einer Leitungsanordnung zur Führung des LOX-Produktes (8) von dem Destillationssäulensystem (C1, C2) zu der Wärmetauscheranordnung (E1);

wobei die Vorrichtung weiterhin aufweist entweder

(a) eine Leitungsanordnung zur Führung des LOX-Kühlmittels (10) bei einem höheren Druck als der Druck des LOX-Produktes, das in die Wärmetauscheranordnung eintritt, von einem externen Vorrat zu der Wärmetauscheranordnung (E1), jedoch keinen Expander zum Expandieren eines Prozessstroms aufweist, um Kühlleistung zur Verfügung zu stellen; oder
(b) eine Leitungsanordnung zur Führung des LOX-Kühlmittels (10) bei einem Druck, der im Wesentlichen gleich dem Druck des LOX-Produktes ist, das in die Wärmetauscheranordnung eintritt, von einem externen Vorrat zu einem Zwischenpunkt zwischen dem warmen und dem kalten Ende der Wärmetauscheranordnung, wo die Temperatur der Wärmetauscheranordnung sich über der Siedetemperatur des LOX-Kühlmittels befindet, wobei die Wärmetauscheranordnung getrennte Kreisläufe für das LOX-Kühlmittel und das LOX-Produkt hat.

18. Vorrichtung nach Anspruch 17, angepasst oder aufgebaut, um das Verfahren nach einem der Ansprü-

che 2 bis 16 durchzuführen.

19. Vorrichtung nach Anspruch 17 oder Anspruch 18, wobei der Kryogen- bzw. Tieftemperatur-Teil der Vorrichtung vakuumisoliert ist, um Wärmeverluste zu reduzieren.

20. Vorrichtung nach einem der Ansprüche 17 bis 19, wobei die Wärmetauscheranordnung (E1) wenigstens 55 NTU hat.

21. Vorrichtung nach einem der Ansprüche 17 bis 20, wobei die Wärmetauscheranordnung (E1) von 70 bis 90 NTU hat.

22. Vorrichtung nach einem der Ansprüche 17 bis 21, wobei die Wärmetauscheranordnung (E1) ungefähr 80 NTU hat.

23. Vorrichtung nach einem der Ansprüche 17 bis 22, wobei bei der Option (a) die Wärmetauscheranordnung getrennte Kreisläufe für das LOX-Kühlmittel und das LOX-Produkt hat.

24. Vorrichtung nach einem der Ansprüche 17 bis 23, wobei bei der Option (b) die Vorrichtung keinen Expander zum Expandieren eines Prozessstroms aufweist, um Kühlleistung zur Verfügung zu stellen.

25. Vorrichtung nach einem der Ansprüche 17 bis 24, wobei das LOX-Kühlmittel (10) über einen einzigen Durchgang durch die Wärmetauscheranordnung (E1) verläuft

Revendications

1. Procédé pour la production d'oxygène gazeux (« GOX »), ledit procédé comprenant :

le refroidissement et la condensation au moins partielle d'air d'alimentation (1) par échange de chaleur en utilisant un moyen d'échange de chaleur (E1) ayant une extrémité chaude et une extrémité froide pour produire un air d'alimentation (2) refroidit et au moins partiellement condensé ;

la distillation dudit air d'alimentation (2) refroidi et au moins partiellement condensé dans un système à colonne de distillation (C1, C2) pour produire le produit oxygène liquide (« LOX ») ;
le retrait d'un courant (8) dudit produit LOX du système de colonne de distillation (C1, C2) et la vaporisation dudit courant (8) de produit LOX par échange de chaleur (E1) contre l'air d'alimentation (1) pour produire le GOX ; et
séparément à partir du produit LOX, la vaporisation le réfrigérant LOX (10) à partir d'une

source externe par échange de chaleur (E1) contre l'air d'alimentation (1) pour produire le réfrigérant vaporisé (11) fournissant de ce fait une partie de la charge calorifique nécessaire pour refroidir et condenser au moins partiellement l'air d'alimentation ;

ledit procédé comprenant le réfrigérant LOX que l'on injecte dans le moyen d'échange de chaleur (E1) soit :

- (a) à une pression supérieure que celle du produit LOX qui entre dans le moyen d'échange de chaleur ; ou
- (b) à une pression sensiblement égale à celle du produit LOX qui entre dans le moyen d'échange de chaleur et à un point intermédiaire entre les extrémités chaudes et froides où la température du moyen d'échange de chaleur (E1) est au-dessus de la température d'ébullition du réfrigérant LOX.

2. Procédé selon la revendication 1, dans lequel la température du moyen d'échange de chaleur (E1) au point intermédiaire auquel on injecte le réfrigérant LOX est de -165°C à -80°C.

3. Procédé selon la revendication 1 ou la revendication 2, comprenant en outre la combinaison du réfrigérant LOX vaporisé (10) avec le GOX (9) produit au moyen de la vaporisation du produit LOX pour produire le produit GOX (12).

4. Procédé selon l'une quelconque des revendications 1 à 3, dans lequel le système de colonne de distillation comprend un système de colonne multiple ayant une pression (« HP ») de colonne (C1) plus élevée et une pression (« LP ») de colonne (C2) plus basse intégrée de façon thermique au moyen de la condensation d'azote de t^{te} depuis la colonne HP contre les résidus liquides dans la colonne LP.

5. Procédé selon l'une quelconque des revendications 1 à 4, dans lequel on ne fournit aucune charge calorifique par extension du cycle de fabrication.

6. Procédé selon l'une quelconque des revendications 1 à 5, dans lequel le réfrigérant LOX (10) fournit toutes les charges calorifiques externes nécessaires pour maintenir le processus équilibré en énergie.

7. Procédé selon l'une quelconque des revendications 1 à 6, dans lequel la pression du réfrigérant LOX (10) est de 0,4 MPa (4 bars absolus) à 1,0 MPa (10 bars absolus).

8. Procédé selon l'une quelconque des revendications

1 à 7, comprenant en outre la mise sous pression du courant du produit LOX (8) avant la vaporisation dudit courant par échange de chaleur (E1) pour fournir le GOX (9).

9. Procédé selon la revendication 8, dans lequel on ne met pas sous pression le courant de produit LOX en pompant le courant.

10. Procédé selon la revendication 8 ou la revendication 9, dans lequel on met sous pression le courant de produit LOX de façon hydrostatique.

11. Procédé selon la revendication 8, dans lequel on met sous pression le courant de produit LOX en pompant le courant.

12. Procédé selon l'une quelconque des revendications 1 à 11, dans lequel la pression du produit GOX est de 0,15 MPa (1,5 bar absolu) à 0,3 MPa (3,0 bar absolu).

13. Procédé selon l'une quelconque des revendications 1 à 12, dans lequel la pression du produit GOX (12) est de 0,18 MPa (1,8 bar absolu) à 0,25 MPa (2,5 bars absolus).

14. Procédé selon l'une quelconque des revendications 1 à 13, comprenant la combinaison du réfrigérant LOX avec l'air d'alimentation refroidi et au moins partiellement condensé pour ensuite refroidir l'air d'alimentation.

15. Procédé selon l'une quelconque des revendications 1 à 14, comprenant en outre l'introduction du réfrigérant LOX dans le système de colonne de distillation sous commande de niveau.

16. Procédé selon l'une quelconque des revendications 1 à 15, dans lequel on fournit le réfrigérant LOX au moyen d'une unité de séparation d'air.

17. Appareil pour la réalisation du procédé de la revendication 1 pour produire de l'oxygène gazeux, ledit appareil comprenant :

- un moyen d'échange de chaleur (E1) pour refroidir et condenser au moins partiellement l'air d'alimentation (1) pour produire un air d'alimentation (2) refroidi et au moins partiellement condensé, ledit moyen d'échange de chaleur (E1) ayant une extrémité chaude et une extrémité froide ;
- un système de colonne de distillation (C1, C2) pour distiller l'air d'alimentation refroidi et au moins partiellement condensé (2) pour produire le produit LOX (8) ;
- un moyen de conduite pour transporter l'air

d'alimentation (2) refroidi et au moins partiellement condensé depuis le moyen d'échange de chaleur (E1) vers le système de colonne de distillation (C1, C2) ; et

un moyen de conduite pour transporter le produit LOX (8) depuis le système de colonne de distillation (C1, C2) vers le moyen d'échange de chaleur (E1) ;

ledit appareil comprenant soit :

(a) un moyen de conduit pour transporter le réfrigérant LOX (10) à une pression supérieure à la pression du produit LOX qui entre dans le moyen d'échange de chaleur depuis une alimentation externe vers le moyen d'échange de chaleur (E₁) mais ne comprend pas un expandeur pour étendre un cycle de fabrication pour fournir une charge calorifique ; soit

(b) un moyen de conduite pour transporter le réfrigérant LOX (10) à une pression qui est sensiblement égale à la pression du produit LOX qui entre dans le moyen d'échange de chaleur depuis une alimentation externe vers un point intermédiaire entre les extrémités chaudes et froides du moyen d'échange de chaleur où la température du moyen d'échange de chaleur est supérieure de la température d'ébullition du réfrigérant LOX et le moyen d'échange de chaleur a des circuits séparés pour le réfrigérant LOX et le produit LOX.

18. Appareil selon la revendication 17, adapté ou construit pour réaliser le procédé que l'on définit dans l'une quelconque des revendications 2 à 16.

19. Appareil selon la revendication 17 ou 18, dans lequel la partie cryogénique de l'appareil est isolée sous vide pour réduire la perte de chaleur.

20. Appareil selon l'une quelconque des revendications 17 à 19, dans lequel le moyen d'échange de chaleur (E1) a au moins 55 NTU.

21. Appareil selon l'une quelconque des revendications 17 à 20, dans lequel le moyen d'échange de chaleur (E1) a de 70 à 90 NTU.

22. Appareil selon l'une quelconque des revendications 17 à 21, dans lequel le moyen d'échange de chaleur (E1) a de 70 à 90 NTU.

23. Appareil selon l'une quelconque des revendications 17 à 22, dans lequel, de façon facultative (a), le moyen d'échange de chaleur a des circuits séparés pour le réfrigérant LOX et le produit LOX.

24. Appareil selon l'une quelconque des revendications

17 à 23, dans lequel, de façon facultative (b), l'appareil ne comprend pas un expandeur pour étendre un cycle de fabrication pour fournir la charge calorifique.

25. Appareil selon l'une quelconque des revendications 17 à 24, dans lequel le réfrigérant LOX (10) passe à travers le moyen d'échange de chaleur (E1) via un passage seul.

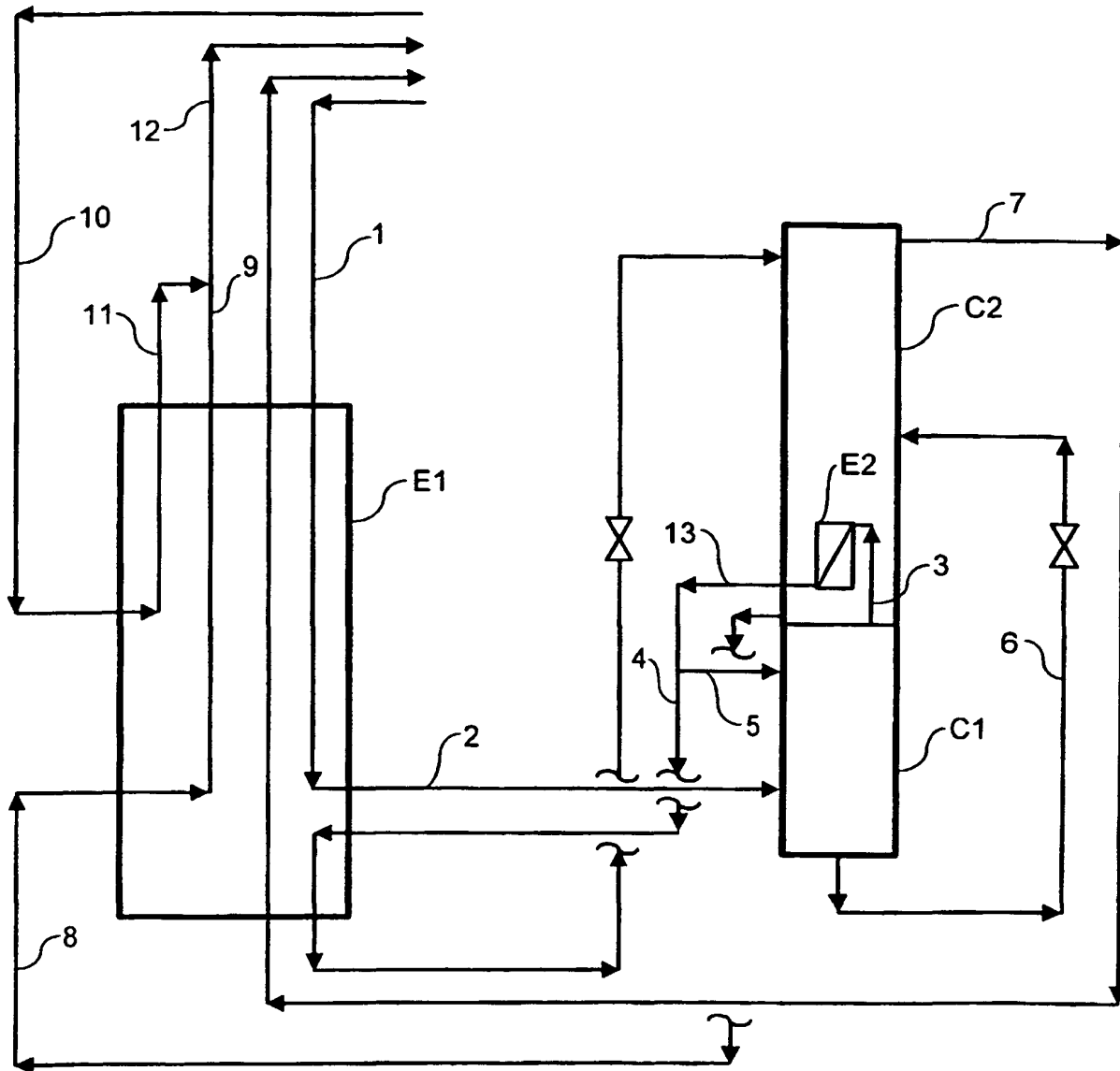


FIG. 1