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(54) **PROCESS AND APPARATUS FOR PRODUCING ULTRAPURE OXYGEN**

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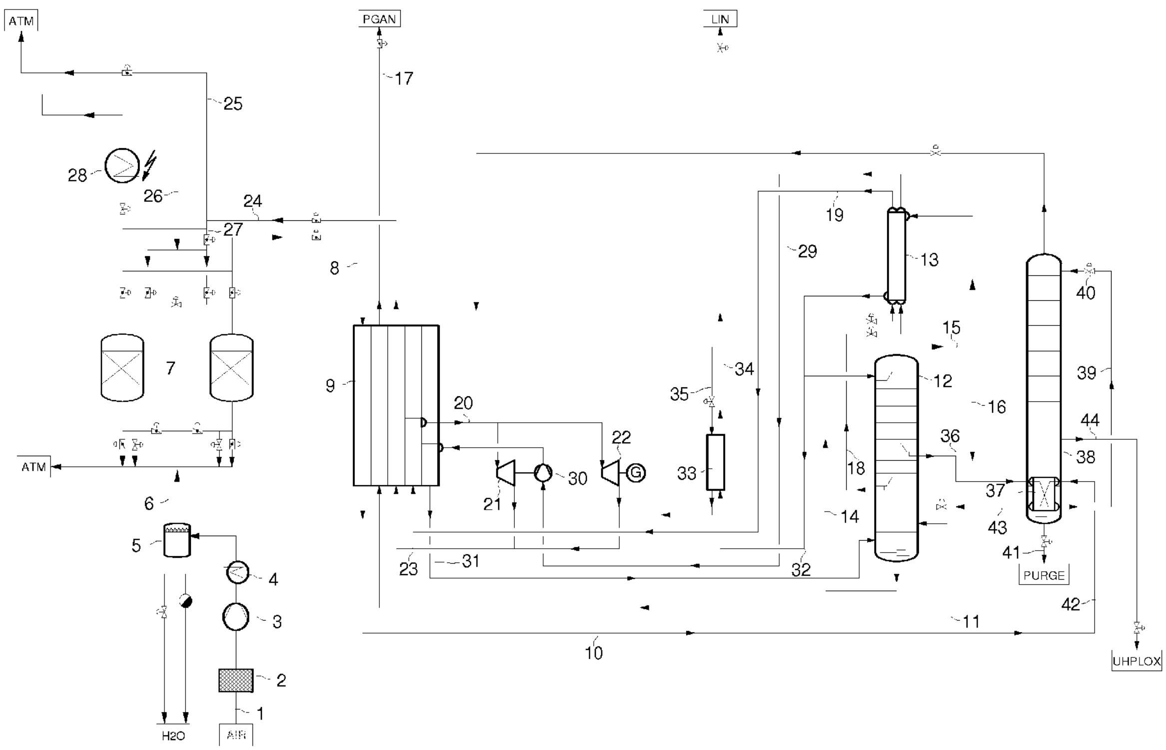
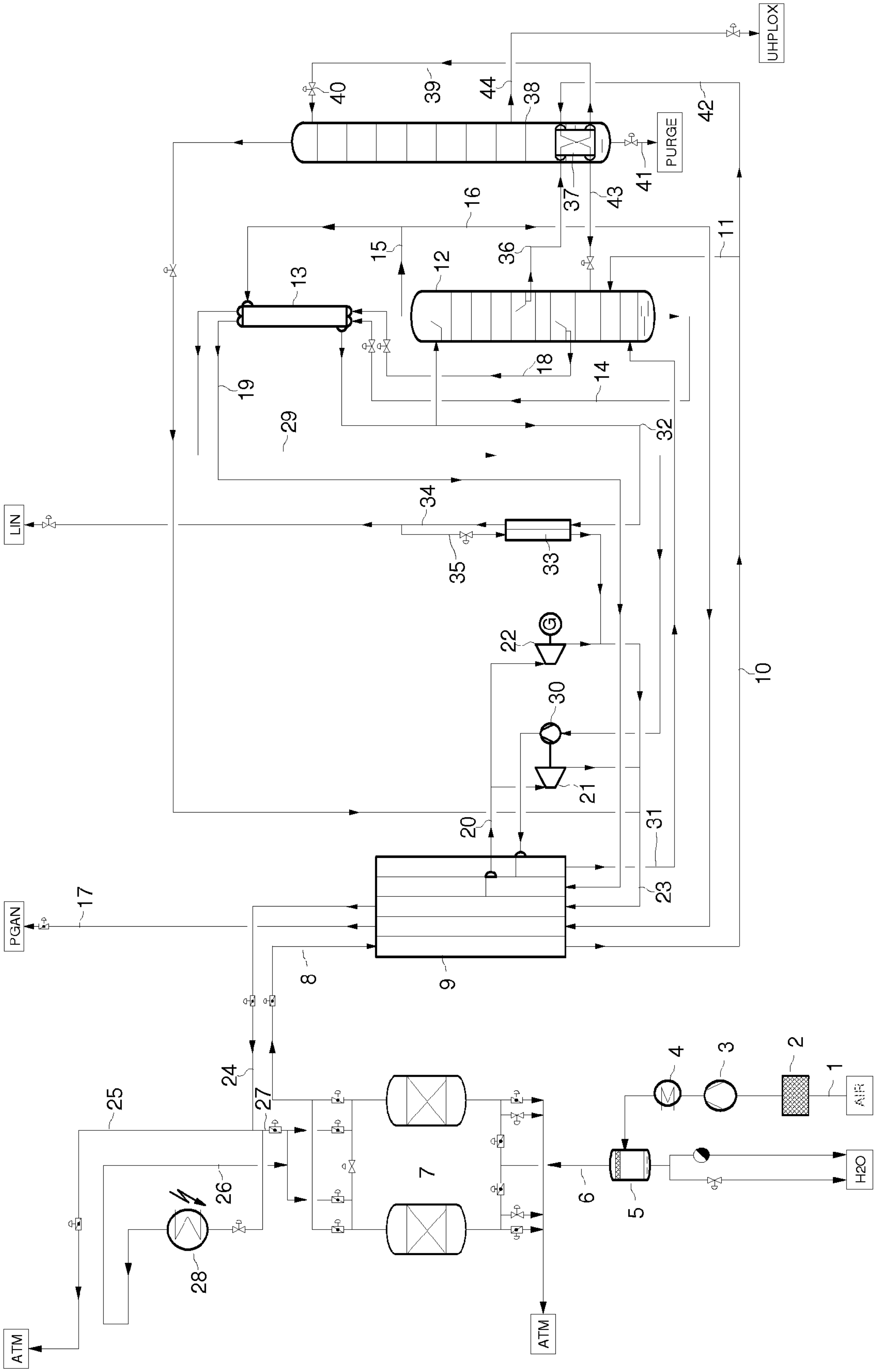
(57) **ABSTRACT**

For the production of ultrapure oxygen, conduct the followig steps: introduce a feed air stream (**10, 11, 42, 43**) into a single distillation column (**12**) for producing nitrogen; withdraw an oxygen-containing stream (**36, 39**) from an intermediate stage of the single distillation column (**12**), introduce the oxygen-containing stream (**36, 39**) into an upper section of a high-purity oxygen column (**38**), and withdraw an ultrapure oxygen product stream (**44**) from the high-purity oxygen column (**38**), with the caveat that rising vapour for the high-purity oxygen column (**38**) is produced by indirect heat exchange in a reboiler (**37**) between a liquid fraction from a lower section of the high-purity oxygen column and a gaseous heating stream (**42**), so that the heating stream (**42**) is at least partially condensed during such indirect heat exchange.

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PROCESS AND APPARATUS FOR PRODUCING ULTRAPURE OXYGEN

[0001] This application claims the benefit of the filing date of U.S. Provisional Application Ser. No. 60/774,662 filed Feb. 21, 2006.

[0002] The invention relates to a process and an apparatus for producing ultrapure oxygen incorporating a single distillation column for nitrogen production and a high-purity oxygen column. Preferably, the process and the apparatus do not exhibit any further separation column.

BACKGROUND OF THE INVENTION

[0003] Such process is known from U.S. Pat. No. 5,689,973 where rising vapour for the high-purity oxygen column is produced by condensing and subcooling a portion of the oxygen-containing feed mixture for the high-purity oxygen column in a bottom reboiler.

SUMMARY OF THE INVENTION

[0004] It is an object of the invention to provide a process and apparatus which is economically advantageous.

[0005] It is a further object of the invention to provide a process and an apparatus with optimized reboiling of the high-purity oxygen column.

[0006] Upon further study of the specification and appended claims, other aspects of the invention will become apparent.

[0007] In brief, the invention comprises a process and an apparatus for producing ultrapure oxygen, the process comprising the following steps:

[0008] introducing a feed air stream into a single distillation column for producing nitrogen,

[0009] withdrawing an oxygen-containing stream from an intermediate stage of the single distillation column,

[0010] introducing the oxygen-containing stream into an upper section of a high-purity oxygen column, and

[0011] withdrawing an ultrapure oxygen product stream from the high-purity oxygen column,

whereby

[0012] rising vapour for the high-purity oxygen column is produced by indirect heat exchange in a reboiler between a liquid fraction from a lower section of the high-purity oxygen column and a gaseous heating stream,

[0013] whereby the heating stream is at least partially condensed during such indirect heat exchange.

[0014] By providing reboil for the high-purity oxygen column by a gaseous heating stream, which is at least partially condensed in a reboiler of the high-purity oxygen column, the following advantages are achieved:

[0015] Increased yield of either ultrapure oxygen, gaseous or liquid nitrogen product, or both.

[0016] Reduced feed air requirement for fixed ultrapure oxygen or nitrogen production.

[0017] Reduced length of high-purity oxygen column.

[0018] "Ultrapure" oxygen means an oxygen fraction with a total molar content of impurities of 1000 ppb or less, preferably 100 ppb or less, more preferably 10 ppb or less.

[0019] The "oxygen-containing stream" used as feed mixture for the high-purity oxygen has an oxygen content of 5% or more, preferably 10% or more, more preferably 15% or more. (All percentage values are on a molar basis.)

[0020] Additional preferred features of the invention are listed in the dependent claims.

BRIEF DESCRIPTION OF THE DRAWING

[0021] The single drawing shows an embodiment of the invention.

DETAILED DESCRIPTION OF THE DRAWING

[0022] In the embodiment of the drawing, atmospheric air **1** flows through a filter **2** and is compressed by an air compressor **3** to a pressure of 8 to 13 bars, preferably about 11 bars (all pressure values in this document are gauge, i.e. above atmospheric). After an aftercooler **4** and a water separator **5**, the compressed air **6** is introduced into a purification device **7** comprising a pair of vessels filled with adsorptive material. The purified air **8** is cooled in main heat exchanger **9** to about its dewpoint. A first portion **11** of the cold air feed air stream **10** is introduced in gaseous (or slightly wet) form into a single distillation column **12** for producing nitrogen, preferably some theoretical or practical trays above the bottom.

[0023] The single column **12** is operated at a pressure of 7 to 12 bars, preferably about 10 bars. Its head condenser **13** operated by vaporizing bottom liquid **14** and a nitrogen-richer liquid **18** withdrawn from an intermediate stage some theoretical or practical trays above the gaseous air inlet. As its main product, gaseous pressurized nitrogen is withdrawn via lines **15**,**16**, warmed to about ambient temperature in main heat exchanger **9**. It leaves the plant via line **17** as pressurized gaseous nitrogen product (PGAN).

[0024] A first waste gas **19** produced by evaporation of the bottom liquid **14** is warmed to an intermediate temperature in the main heat exchanger **9**, withdrawn via line **20** and work-expanded in one turbine (not shown) or in two parallel turbines **21**, **22** from a pressure of 4 to 8 bars, preferably about 6 bars to a pressure of pressure of 0.25 to 1 bars, preferably about 0.5 bars. The work-expanded stream **23** is warmed to about ambient temperature in main heat exchanger **9**. The warm first waste gas stream **24** is released to the atmosphere (line **25**), and/or used as regeneration gas **26**, **27** in purification device **7**, eventually after heating in heating device **28**.

[0025] A second waste gas **29** produced by evaporation of the intermediate liquid **18** is compressed from a pressure of 4 to 8 bars, preferably about 6 bars to a pressure of 7 to 12 bars, preferably about 10 bars in a cold compressor **30** driven by turbine **21**, cooled in main heat exchanger **9** and reintroduced via line **31** into the single column **12** at its bottom.

[0026] A portion of the nitrogen product of the single column may be withdrawn as a liquid product LIN from the head condenser **13** via line **32**, after being subcooled in subcooler **33**. A portion **35** of the subcooled liquid nitrogen

34 is expanded and used for delivering refrigeration to the subcooler 33 and admixed to the work-expanded waste gas afterwards.

[0027] An oxygen-containing stream 36, being essentially free of lower-boiling impurities, is withdrawn in liquid form from an intermediate section of the single column 12, which is at least 5 to 25 practical or theoretical trays above any feed air inlet. The liquid oxygen-containing stream is eventually subcooled in a bottom reboiler 37 of a high-purity oxygen column 38 and fed via line 39 to the top of the high-purity oxygen column 38 after having been expanded through valve 40 to a pressure of 0.25 to 1 bars, preferably about 0.5 bars.

[0028] The liquid refluxing column 38 is further enriched in oxygen. A portion 44 is withdrawn as liquid ultrapure oxygen product UHPLOX from 1 to 5 trays, preferably about 3 practical or theoretical trays above the bottom. The remainder is reboiled in bottom reboiler 37 for producing rising vapour in the high-purity oxygen column. Some purge liquid 41 is withdrawn from time to time or continuously from the bottom, i.e. from the evaporation space of bottom reboiler 37.

[0029] According to the invention, reboiler 37 is driven at least in part by a second portion 42 of the feed air stream 10. Such air portion is at least partially, e.g. totally condensed and subcooled in bottom reboiler 37. The liquefied air 43 is introduced into the single column 12 at an intermediate section somewhat above the gaseous air introduction.

[0030] There are many variations of the process within the scope of the invention. In a first variation of the embodiment, at least a portion of the process refrigeration is delivered in form of an external cryogenic liquid, e.g. by liquid nitrogen from a tank, which is injected into single column 12 or into the evaporation space of head condenser 13. Such liquid injection may partially or totally replace the work expansion 21 and 22 of process streams as shown in the drawing. As another variant, the nitrogen subcooler 33 may be omitted and/or a subcooler for liquid ultrapure oxygen product 44 may be added.

[0031] The nitrogen products 17, 34 can be either ultra-high-purity (ppb impurity) or normal commercial grade (ppm impurity). The ultrapure oxygen product 44 can also be recovered as commercial grade LOX (95 to 99.9%); in this case, the safety purge 41 may be omitted. The ultrapure oxygen product can also be withdrawn as a gas and warmed up in the main exchanger 9.

[0032] In a further variation, at least a portion compressed recycled second waste gas 31 may be used as heating medium in the botttom reboiler 37 instead of feed air 42. The condensed recycle fluid produced in the botteom reboiler 37 would flow either to the single column 12 or directly to the head condenser 13, eventually after being combined with stream 18 after its valve.

[0033] Without further elaboration, it is believed that one skilled in the art can, using the preceding description, utilize the present invention to its fullest extent. The preceding preferred specific embodiments are, therefore, to be construed as merely illustrative, and not limitative of the remainder of the disclosure in any way whatsoever.

[0034] In the foregoing and in the examples, all temperatures are set forth uncorrected in degrees Celsius and, all parts and percentages are by weight, unless otherwise indicated.

[0035] The entire disclosures of all applications, patents and publications, cited herein and of corresponding U.S. Provisional Application Ser. No. 60/774,552, filed Feb. 21, 2006, are incorporated by reference herein.

[0036] The preceding examples can be repeated with similar success by substituting the generically or specifically described reactants and/or operating conditions of this invention for those used in the preceding examples.

[0037] From the foregoing description, one skilled in the art can easily ascertain the essential characteristics of this invention and, without departing from the spirit and scope thereof, can make various changes and modifications of the invention to adapt it to various usages and conditions.

1. A process for producing ultrapure oxygen, the process comprising the following steps:

introducing a feed air stream (10, 11, 42, 43) into a single distillation column (12) for producing nitrogen,

withdrawing an oxygen-containing stream (36, 39) from an intermediate stage of the single distillation column (12),

introducing the oxygen-containing stream (36, 39) into an upper section of a high-purity oxygen column (38), and

withdrawing an ultrapure oxygen product stream (44) from the high-purity oxygen column (38), wherein

rising vapour for the high-purity oxygen column (38) is produced by indirect heat exchange in a reboiler (37) between a liquid fraction from a lower section of the high-purity oxygen column and a gaseous heating stream (42),

whereby the gaseous heating stream (42) is at least partially condensed during such indirect heat exchange.

2. A according to claim 1, whereby the gaseous heating stream (42) comprises at least a portion of the feed air stream (10).

3. A process according to claim 1, wherein or the gaseous heating stream (42) downstream of the indirect heat exchange (37) is introduced into the single distillation column (12).

4. A process according to claim 1, wherein the oxygen-containing stream (36, 39) is introduced into the top of the high-purity oxygen column (38).

5. A process according to claim 1, wherein at least a portion of the oxygen-containing stream (36) is subcooled in the reboiler (37) before being introduced (39) into the high-purity oxygen column (38).

6. A process according claim 1, wherein the ultrapure oxygen product stream (44) is withdrawn from an intermediate section of the high-purity oxygen column (38).

7. A process according to claim 1, wherein the ultrapure oxygen product stream (44) is withdrawn from the high-purity oxygen column (38) in a liquid state.

8. A process according to claim 7, wherein the ultrapure oxygen product stream is withdrawn as a final liquid product.

9. Apparatus for producing ultrapure oxygen the process comprising

means for introducing a feed air stream (**10**, **11**, **42**, **43**) into a single distillation column (**12**) for producing nitrogen,

means for withdrawing an oxygen-containing stream (**36**, **39**) from an intermediate stage of the single distillation column (**12**),

means for introducing the oxygen-containing stream (**36**, **39**) into an upper section of a high-purity oxygen column (**38**), and

means for withdrawing an ultrapure oxygen product stream (**44**) from the high-purity oxygen column (**38**),

the apparatus further comprising

a reboiler (**37**) for producing rising vapour for the high-purity oxygen column (**38**) by indirect heat exchange in a reboiler (**37**) between a liquid fraction from a lower section of the high-purity oxygen column and a gaseous heating stream (**42**).

10. A process according to claim 2, wherein the heating stream (**42**) downstream of the indirect heat exchange (**37**) is introduced into the single distillation column (**12**).

11. A process according to claim 10, wherein the oxygen-containing stream (**36**, **39**) is introduced into the top of the high-purity oxygen column (**38**).

12. A process according to claim 2, wherein at least a portion of the oxygen-containing stream (**36**) is subcooled in the reboiler (**37**) before being introduced (**39**) into the high-purity oxygen column (**38**).

13. A process according to claim 3, wherein at least a portion of the oxygen-containing stream (**36**) is subcooled in the reboiler (**37**) before being introduced (**39**) into the high-purity oxygen column (**38**).

14. A process according to claim 10, wherein at least a portion of the oxygen-containing stream (**36**) is subcooled in the reboiler (**37**) before being introduced (**39**) into the high-purity oxygen column (**38**).

15. A process according to claim 12, wherein at least a portion of the oxygen-containing stream (**36**) is subcooled in the reboiler (**37**) before being introduced (**39**) into the high-purity oxygen column (**38**).

16. A process according to claim 15, wherein the ultrapure oxygen product stream (**44**) is withdrawn from the high-purity oxygen column (**38**) in a liquid state.

17. A process according to claim 16, wherein the ultrapure product stream is withdrawn as a final liquid product.

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