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(54) Process and apparatus for producing liquid nitrogen

(57) The process and the apparatus serve for producing high-purity liquid nitrogen from an impure nitrogen feed stream. The process comprises the following steps:

- introducing the feed stream (1, 3) into a single distillation column (4),
- withdrawing a high-purity gaseous nitrogen stream (5) from the single distillation column (4),
- compressing at least a part of the high-purity gaseous nitrogen stream in a recycle compressor (8) of a nitrogen recycle system to produce a high-pressure recycle stream (10),
- work expanding (19, 36) at least a first portion (18, 35) of the high-pressure recycle stream (10) to produce a first low-pressure recycle stream (20, 37),
- recycling (7) at least a portion (23, 37) of the first low-pressure recycle stream to the recycle compressor (8),
- liquefying a second portion of the high-pressure recycle stream to produce a liquid recycle stream (28), and
- recovering at least a first portion (29) of the second low-pressure recycle stream (28) as liquid nitrogen product stream (31).

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Description

[0001] The invention relates to a process and an apparatus for producing liquid nitrogen from air or high-purity liquid nitrogen from impure nitrogen incorporating a single distillation column.

BACKGROUND OF THE INVENTION

[0002] Single-column processes for producing nitrogen from air are known from DE 1199293, GB 1258568, DE 4030750, or US 6470707 B2. Nitrogen purification by single-column distillation is done in EP 1336806, US 3370435, or US 6276172 B1.

SUMMARY OF THE INVENTION

[0003] It is an object of the invention to provide a process and apparatus which is economically advantageous.

[0004] It is a further object of the invention to provide a process and an apparatus with optimized integration between a single-column distillation and a liquefaction recycle.

[0005] Upon further study of the specification and appended claims, other aspects of the invention will become apparent.

[0006] In brief, the invention comprises a process and an apparatus for producing high-purity liquid nitrogen from an impure nitrogen feed stream, the process comprising the following steps:

- introducing the feed stream into a single distillation column,
- withdrawing a high-purity gaseous nitrogen stream from the distillation column,
- compressing at least a part of the high-purity gaseous nitrogen stream in a recycle compressor of a nitrogen recycle system to produce a high-pressure recycle stream,
- work expanding at least a first portion of the high-pressure recycle stream to produce a first low-pressure recycle stream,
- recycling at least a portion of the first low-pressure recycle stream to the recycle compressor,
- liquefying a second portion of the high-pressure recycle stream to produce a liquid recycle stream, and
- recovering at least a first portion of the second low-pressure recycle stream as liquid nitrogen product stream.

[0007] In another variant, the invention comprises a process for producing liquid nitrogen comprising the following steps:

- introducing a nitrogen-containing mixture into a single distillation column,
- withdrawing a gaseous nitrogen stream from the single distillation column,
- compressing at least a part of the gaseous nitrogen stream in the recycle compressor of a nitrogen recycle system to produce a high-pressure recycle stream,
- work expanding a first portion of the high-pressure recycle stream to produce a first low-pressure recycle stream,
- splitting the first low-pressure recycle stream into at least a first portion and a second portion,
- introducing the second portion of the second low-pressure recycle stream into a reboiler of a single distillation column,
- at least partially condensing the second portion of the second low-pressure recycle stream in the reboiler,
- introducing at least a portion of the at least partially condensed low-pressure recycle stream into the distillation column,
- recovering the first portion of the second low-pressure recycle stream or another liquefied portion of the high-pressure recycle stream or both as liquid nitrogen product stream.

[0008] Such process may or may not use a feed compressor for compressing the nitrogen-containing mixture.

[0009] "High-purity" nitrogen means a nitrogen fraction with a total molar content of impurities of 1 % or less, preferably 1000 or less, more preferably 100 ppm or less,

[0010] "Impure nitrogen stream" means a fraction having a molar nitrogen concentration higher than atmospheric air, but lower than the high-purity liquid nitrogen product.

[0011] A "nitrogen-containing mixture" may be constituted by any fluid containing nitrogen, e.g. by atmospheric air.

[0012] In the invention, the liquid nitrogen product is at least partially withdrawn neither from the distillation column nor from one of its condensers, but taken from the working fluid of a recycle system comprising at least a recycle compressor and at least one turbine. Nitrogen gas from the column is used as working fluid of the recycle system.

[0013] Preferably, the distillation column has a reboiler which is heated by a portion of the work fluid of the recycle system. Such portion is thereby condensed and led to the column as reflux liquid.

[0014] In one embodiment of the invention, the distillation column does not have a head condenser. In particular, the liquid formed in the reboiler and/or a portion of the liquefied work fluid of the recycle system provide reflux for the distillation column.

[0015] It is preferred that the distillation has no further reboiler than the bottom reboiler driven by work fluid from the recycle.

[0016] The recycle system may comprise one, two or

more turbines.

BRIEF DESCRIPTION OF THE DRAWING

[0017] The single drawing shows an embodiment of the invention for producing high-purity liquid nitrogen from an impure nitrogen stream.

DETAILED DESCRIPTION OF THE DRAWING

[0018] An impure nitrogen feed stream 1 has a pressure of about 5.4 bars and a total impurity content of about 1000 ppm, including an oxygen content of about 190 ppm, the remainder being exclusively nitrogen. The feed stream 1 is fed under about ambient or higher temperature (e. g. 320 K) into a main heat exchanger 2 where it is cooled in indirect heat exchange with cold streams close to its dew point. The cold feed stream 3 is fed into a distillation system, which comprises a single column 4 operating at a pressure of about 5.3 bars (at the bottom).

[0019] In the distillation column 4, the feed stream oxygen content is further reduced by countercurrent heat and mass exchange. As the main product, high-purity gaseous nitrogen 5 is withdrawn from the upper section of the distillation column 4, preferably from its top. Via line 6, it is introduced into the cold end of main heat exchanger 2, where it is warmed to about ambient or higher temperature. The warm high-purity gaseous nitrogen 7 is compressed in a recycle compressor 8 having an aftercooler 9.

[0020] High-pressure recycle stream 10 is divided into a first partial stream 11 and a second partial stream 12. The first partial stream 11 is further compressed in two serially connected boosters 13, 15 with aftercoolers 14, 16. The further compressed first partial stream 17 is introduced into the warm end of the main heat exchanger 2. At a first intermediate temperature, it is partially withdrawn via line 18 from the main heat exchanger 2 and fed to a cold expansion turbine 19, where it is work expanded to a pressure of about 6 bars. The expanded first partial stream 20 leaves turbine 19 and is introduced into a phase separator 21. (Alternatively, turbine output stream 20 may be directly introduced into line 22.) The remainder of the first partial stream continues its cooling up to the cold end of the main heat exchanger 2 or somewhat above and is finally introduced via line 42 and throttling valve 43 into the phase separator 21.

[0021] The gaseous fraction 22 from phase separator 21 is split into a recycling portion 23 and a bottom reboiler portion 24. The recycling portion is admixed to the high-purity gaseous nitrogen 5 re-warmed in the main heat exchanger 2 and recycled to the inlet of recycle compressor 8. The other portion 24 is fed to a bottom reboiler 25 of the distillation column 4, substantially totally or totally condensed and fed via line 26 and throttling valve 27 into the upper section of the distillation column 4 in order to be used as liquid reflux.

[0022] A portion 29, 31 of the liquid fraction 28 from phase separator 21 is subcooled in subcooler 30 and recovered as the high-purity liquid nitrogen product (LIN) having an oxygen content of 10 ppm and a total impurity content of 100 ppm. The remainder 32 of the liquid fraction 28 from phase separator 21 is fed via throttling valve 33 as further reflux liquid to the upper section of distillation column 4. Alternatively, liquid nitrogen reflux stream 32 may be withdrawn from downstream subcooler 30.

[0023] The second partial stream 12 of high-pressure recycle stream 10 is led to the warm end of main heat exchanger 2 without being further compressed. At a second intermediate temperature, which is higher than the first intermediate temperature, the second partial stream is withdrawn via line 35 from the main heat exchanger 2 and fed to a warm expansion turbine 36, where it is work expanded to a pressure of about 6 bars. The second partial stream leaves turbine 36 as a gaseous stream 37 and is added to the other nitrogen return streams at a third intermediate temperature of the main heat exchanger 2 being close to the first intermediate temperature, and finally recycled to the recycle compressor 8.

[0024] From the bottom of the distillation column 4, a liquid nitrogen stream having an elevated concentration of lower boiling components (e.g. about 1200 ppm oxygen), is withdrawn in liquid form via line 38, throttled to about atmospheric pressure in valve 39, warmed in subcooler 30, introduced into the cold end of main heat exchanger 2 via line 40, evaporated and warmed therein, and finally withdrawn as an impure product or released to the atmosphere via line 41.

[0025] The turbines 19, 36 drive the boosters 13, 15 by direct mechanical coupling. Alternatively, one or both turbines can be connected to any other known braking device as e.g. a dissipative brake or an electric generator. The respective booster would be omitted in such case.

[0026] In the embodiment, the main heat exchanger 2 and the subcooler 30 are realized as a single block. Alternatively, they could be manufactured as two or more separate blocks.

[0027] The process and apparatus of the embodiment can be used for the purification of any impure nitrogen feed stream. Even air can be used as feed, so that it becomes a single column air separation unit. In this case, the bottom liquid 38 of the distillation column 4 would constitute the oxygen-enriched product of the air separation.

Claims

1. Process for producing high-purity liquid nitrogen from an impure nitrogen feed stream, the process comprising the following steps:

- introducing the feed stream (1, 3) into a single distillation column (4),
- withdrawing a high-purity gaseous nitrogen stream (5) from the single distillation column (4),
- compressing at least a part of the high-purity gaseous nitrogen stream in a recycle compressor (8) of a nitrogen recycle system to produce a high-pressure recycle stream (10),
- work expanding (19, 36) at least a first portion (18, 35) of the high-pressure recycle stream (10) to produce a first low-pressure recycle stream (20, 37),
- recycling (7) at least a portion (23, 37) of the first low-pressure recycle stream to the recycle compressor (8),
- liquefying a second portion of the high-pressure recycle stream to produce a liquid recycle stream (28), and
- recovering at least a first portion (29) of the second low-pressure recycle stream (28) as liquid nitrogen product stream (31).

2. Process according to claim 1, comprising

- introducing a reboiler stream (24) derived from the high-pressure recycle stream (10) into a reboiler (25) of the single distillation column (4),
- at least partially condensing the reboiler stream (24) in the reboiler (25), and
- introducing at least a portion of the at least partially condensed reboiler stream (26) into the single distillation column (4).

3. Process according to claim 2, whereby the single distillation column (4) does not have a head condenser.

4. Process according to claim 3, whereby the reboiler (25) is a bottom reboiler-condenser and constitutes the single reboiler-condenser of the single distillation column (4).

5. Process for producing high-purity liquid nitrogen comprising the following steps:

- introducing a nitrogen-containing mixture (1, 3) into a single distillation column (4),
- withdrawing a gaseous nitrogen stream (5) from the single distillation column,
- compressing at least a part of the gaseous nitrogen stream (5) in a recycle compressor (8) of a nitrogen recycle system to produce a high-pressure recycle stream (10),
- expanding (19, 43) a first portion (18, 42) of the high-pressure recycle stream (10) to produce a first low-pressure recycle stream (20, downstream 43),
- splitting the first low-pressure recycle stream (20, downstream 43) into at least a first portion (29) and a second portion (24),
- introducing the second portion (24) of the second low-pressure recycle stream into a reboiler (25) of the single distillation column,
- at least partially condensing the second portion of the second low-pressure recycle stream (24) in the reboiler (25),
- introducing at least a portion of the at least partially condensed low-pressure recycle stream (26) into the single distillation column (4),
- recovering the first portion (29) of the second low-pressure recycle stream or another liquefied portion of the high-pressure recycle stream or both as liquid nitrogen product stream (31).

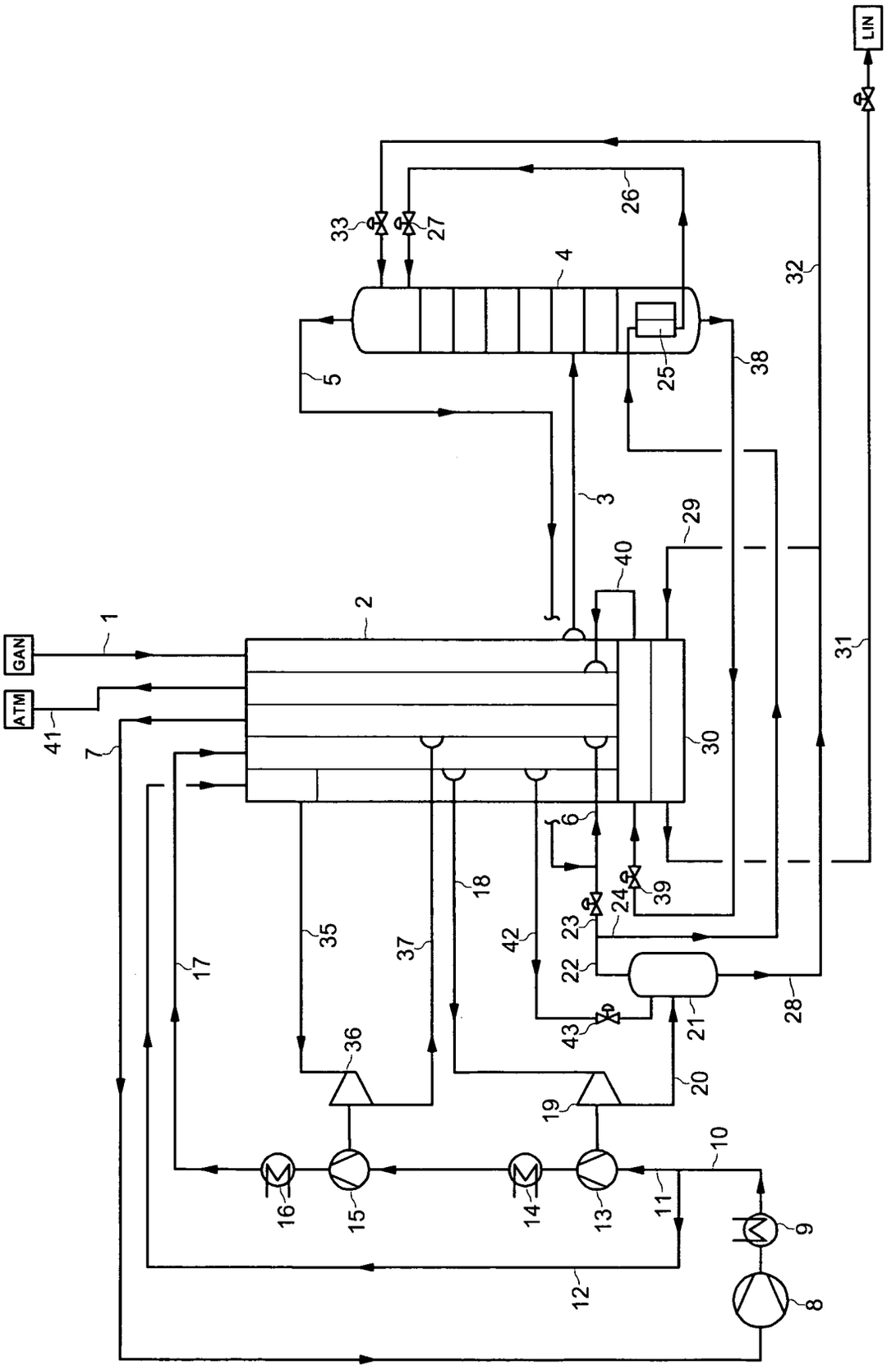
6. Process according to claim 6, whereby the single distillation column does not have a head condenser.

7. Process according to claim 6, whereby the expansion of at least part (18) of the first portion of the high-pressure recycle stream (10) is performed as work-expansion (19).

8. Process according to claim 6, whereby the reboiler (25) is a bottom reboiler-condenser and constitutes the single reboiler-condenser of the single distillation column (4).

9. Process according to any of claims 1 to 8, comprising work expanding (36) a second portion (35) of the high-pressure recycle stream (10) to produce a second low-pressure recycle stream (37).

10. Process according to any of claims 2 to 9, comprising deriving a liquid nitrogen reflux stream (32) from the high-pressure recycle stream (10) and introducing it into the single distillation column (4), the liquid nitrogen reflux stream (32) being separate from the at least partially condensed reboiler stream (26).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EUROPEAN SEARCH REPORT

Application Number
EP 04 02 9742

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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
Munich	16 March 2005	Göritz, D

CATEGORY OF CITED DOCUMENTS

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