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(54) **PROCESS AND APPARATUS FOR THE SEPARATION OF AIR BY CRYOGENIC DISTILLATION**

(57) During shut-down of a double column, the argon column (K10) keeps functioning or is in a ready to function state, using a cycle stream (3) to heat a bottom reboiler (E2) of the argon column.

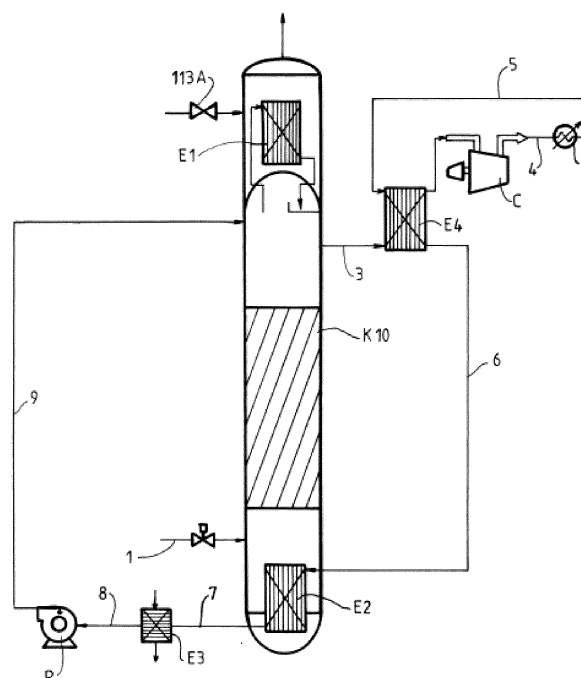


FIG. 2

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Description

[0001] The present invention relates to a process and an apparatus for the separation of air by cryogenic distillation. Argon is mostly produced with an air separation process by distillation at cryogenic temperatures.

[0002] The compressed and purified air is sent to a double column including a first column operating at a first pressure and a second column operating at a second pressure, the second pressure being lower than the first pressure. Oxygen enriched liquid and nitrogen enriched liquid are sent from the first column to the second column. Oxygen rich fluid is removed from the bottom of the second column and nitrogen enriched gas is removed from the top of the second column.

[0003] An argon enriched stream, contains nitrogen and oxygen, is removed from an intermediate point of the second column and separated by cryogenic distillation. The stream typically contains between 5 and 15 mol% argon. Argon volatility being between those of oxygen and nitrogen, for a pure argon product, two argon columns are required to get on specification, typically less than 1 ppm oxygen and nitrogen being required in the argon product, whilst for a less pure argon product one argon column is sufficient.

[0004] The first column (called crude argon column in the remaining description) removes oxygen from argon and nitrogen, this separation is the more difficult one due to the close volatility of oxygen and argon. Such a column is typically 60 meters in height. The second column (called pure argon column) removes nitrogen from argon; this separation, much easier, requires only a short column.

[0005] The top product of the crude argon column, free of oxygen, is then sent to the pure argon column. The reflux of the crude argon column flows down the column and the bottom liquid is pumped back to the second column.

[0006] Starting-up the crude argon column and converging to the required purity specification take a long time, on average 18 hours, because of the difficulty of separating argon and oxygen and because of the relatively small argon input in the crude argon column in comparison with the required total liquid argon hold-up the packings and distributors.

[0007] During the operation, the main risk of in the crude argon column is a high N₂ ingress from the second column. This will reduce the temperature at the top of the crude argon column and block the top condenser thus interrupting the flow from the second column to the crude argon column and the crude argon column will stop.

[0008] The key points to keep in mind regarding the crude argon column are that:

- typically the crude argon column cannot run without the double column of the air separation unit
- the crude argon column is sensitive to upsets in the second column

- the crude argon column requires a long restart time

[0009] The proposed idea here is a configuration, which allows at least part of the crude argon column to run at a reduced load in closed loop mode while the remaining part of the air separation unit is either in cold stand-by or suffering a major upset. The major advantage is that the crude argon column is ready to produce whenever the cold stand-by or the upset is over.

[0010] "Cold stand-by" means that the distillation column does not function but remains at a cryogenic temperature, either because the stand-by duration is so short that the temperature does not rise above cryogenic temperatures or because refrigeration is added to the column, for example by adding a cryogenic liquid to the column from an outside source.

[0011] Having an air separation unit in cold stand-by for few hours may become the rule in the future, since the energy supply from certain sources, such as renewables, is intermittent. The proposed solution here aims at adapting the air separation unit for such conditions, keeping the argon part in operation and ready to use during a stoppage of the column feeding the crude argon column, at reasonable energy cost. According to the invention, the crude argon column is kept running using a cycle to provide reboil to the column, the cycle fluid being an argon enriched gas from the top of the crude argon column. This presents the advantage of using a fluid present in the crude argon column, rather than importing a fluid from an external source.

[0012] According to our calculations, comparing the cost of keeping the crude argon column running and the cost of restarting the crude argon column, it is economical to run the crude argon column on its own for several days, rather than to restart it. According to the invention, there is provided a process for the separation of air by cryogenic distillation in which :

- i) During a first operation mode, compressed and purified air is sent to a first distillation column operating at a first pressure, an oxygen enriched liquid is sent from the first distillation column to a second distillation column operating at a second pressure lower than the first pressure, a nitrogen enriched liquid is sent from the first distillation column to the second distillation column, the first distillation column being thermally linked with the second distillation column via a bottom reboiler of the second distillation column, an oxygen enriched fluid is removed from the bottom of the second distillation column, a nitrogen enriched gas is removed from the top of the second distillation column, a feed stream containing between 5 and 15% mol argon is sent from the second column to the bottom of a crude argon column and a stream containing proportionally more argon than the feed stream is removed from the top of the crude argon column and
- ii) During a second operation mode, no air is sent

the first distillation column, no liquids are sent from the first to the second distillation column, no feed stream is sent from the second distillation column to the crude argon column and no stream containing more argon than the feed stream is removed from the top of the crude argon column wherein during the second operation mode, a cycle stream is removed from the top of the crude argon column, at least part of the cycle stream is used to warm a bottom reboiler of the crude argon column being thereby condensed to form a condensed cycle stream, the condensed cycle stream being sent to the top of the crude argon column and the stream, if present, containing proportionally more argon than the feed stream removed from the top of the upper section constitutes at most 20% of the flow of that stream during the first operation mode.

[0013] According to other optional features:

- during the first operation mode, a nitrogen enriched gas or liquid is removed from the top of the second distillation column,
- during the first operation mode, a stream containing proportionally more argon than the feed stream is removed from the top of the crude argon column for further purification or for direct use as a product stream
- during the first operation mode, no stream is used to warm the bottom reboiler of the crude argon column.
- in the second operation mode, the stream containing proportionally more argon than the feed stream removed from the top of the crude argon column constitutes at most 10% of the flow of that stream during the first operation mode
- during the second operation mode at least part of the cycle stream is compressed in a compressor having a cryogenic inlet temperature upstream of the reboiler.
- during the second operation mode the condensed cycle stream is pressurized by a pump before being sent to the top of the crude argon column.
- during the second operation mode, part of the cycle stream is vented or used as a product.
- during the second operation mode, part of the cycle stream is condensed in a condenser other than the bottom reboiler and then sent back to the crude argon column.
- during the second operation mode, any valves on conduits between the second column and the crude argon column are closed.

[0014] According to another object of the invention there is provided a process for the separation of air by cryogenic distillation in which :

- i) During a first operation mode, compressed and

purified air is sent to a first distillation column operating at a first pressure, an oxygen enriched liquid is sent from the first distillation column to a second distillation column operating at a second pressure lower than the first pressure, a nitrogen enriched liquid is sent from the first distillation column to the second distillation column, the first distillation column being thermally linked with the second distillation column via a bottom reboiler of the second distillation column, an oxygen enriched fluid is removed from the bottom of the second distillation column, a nitrogen enriched gas is removed from the top of the second distillation column, a feed stream containing between 5 and 15% mol argon is sent from the second column to the bottom of a crude argon column having a lower section and an upper section and a stream containing proportionally more argon than the feed stream is removed from the top of the upper section of crude argon column and

ii) During a second operation mode, no air is sent to the first distillation column, no liquids are sent from the first to the second distillation column, no feed stream is sent from the second distillation column to the crude argon column in two sections and no stream containing more argon than the feed stream is removed from the top of upper section, the crude argon column wherein during the second operation mode, a cycle stream is removed from the top of the crude argon column, at least part of the cycle stream is used to warm a bottom reboiler of the upper or lower section of the crude argon column being thereby condensed to form a condensed cycle stream, the condensed cycle stream being sent to the top of the upper section of the crude argon column. and the stream, if present, containing proportionally more argon than the feed stream removed from the top of the crude argon column constitutes at most 20% of the flow of that stream during the first operation mode.

[0015] According to another object of the invention there is provided an apparatus for the separation of air by cryogenic distillation comprising a first distillation column operating at a first pressure, a second distillation column operating at a second pressure lower than the first pressure, the second distillation column having a bottom reboiler, a crude argon column having a top condenser and a bottom reboiler, a conduit for sending compressed and purified air to the first distillation column operating at a first pressure, a conduit for sending an oxygen enriched liquid from the first distillation column to the second distillation column operating at a second pressure lower than the first pressure, a conduit for sending a nitrogen enriched liquid from the first distillation column to the second distillation column, the first distillation column being thermally linked with the second distillation column via the bottom reboiler of the second distillation column, a conduit for removing an oxygen enriched fluid from the

bottom of the second distillation column, a conduit for removing nitrogen enriched gas from the top of the second distillation column, a conduit for sending a feed stream containing between 5 and 15% mol argon from the second column to the bottom of a crude argon column, possibly a conduit for removing a stream containing more argon than the feed stream from the top of the crude argon column characterized in that it comprises a conduit for sending at least part of a cycle stream to the reboiler and a conduit for sending a condensed cycle stream from the reboiler to the top of the crude argon column and means for closing the conduit for sending air to the first distillation column and/or means for closing the conduit for sending the feed stream from the second distillation column to the crude argon column.

[0016] The apparatus optionally comprises:

- a compressor for compressing the cycle stream downstream of the removal from the crude argon column and upstream of the bottom reboiler of the crude argon column.
- a pump for sending at least part of the cycle stream in condensed form back to the crude argon column.
- a conduit from sending bottom liquid from the crude argon column to the second distillation column, said conduit being closable via closure means.
- means for venting at least part of the cycle stream.

[0017] The invention will be described in detail with reference to the figures, Figure 1 representing schematically the first and second columns of an apparatus according to the invention and Figures 2 to 6 representing a crude argon column of an apparatus according to the invention.

[0018] Figure 1 shows the first and second columns of an apparatus for the separation of air by cryogenic distillation, designed to be connected to a crude argon column according to one of Figures 2 to 6.

[0019] During a first operation mode corresponding to normal operation of the air separation unit, air 101 is compressed in a compressor C01, purified from water and carbon dioxide in adsorbers R01, R02, cooled in heat exchanger 103 and divided in two. One part of the compressed and purified air is compressed in a cold compressor V02, cooled in heat exchanger 103 and divided in two. One part is expanded in a Claude turbine D01 and sent to the first distillation column as gaseous stream 105. The rest 107 is cooled completely in heat exchanger 103 and sent as a liquid to first column K01.

[0020] Other types of feed air treatment are of course possible (no booster, no cold booster with a warm booster etc).

[0021] Compressed and purified air 105 is sent to the first distillation column K01 operating at a first pressure, generally between 4 and 15 bars abs, and is separated in the first distillation column to form an oxygen enriched liquid at the bottom of the column and a nitrogen enriched gas at the top of the column. Oxygen enriched liquid 109 is sent from the first distillation column to a second dis-

5 tillation column K02 operating at a second pressure lower than the first pressure, generally between 1.2 and 4 bars abs. Part 113A of the oxygen enriched liquid is sent to a top condenser E1 of the crude argon column to be vaporized. A nitrogen enriched liquid is sent from the first distillation column K01 to the second distillation column K02. The first distillation column K01 is thermally linked with the second distillation column K02 via a bottom reboiler of the second distillation column. A feed stream 10 containing between 5 and 15% mol argon removed from an intermediate point of the second column K02 is sent to the bottom of a crude argon column K10, as illustrated in any one of Figures 2 to 6.

[0022] Oxygen enriched liquid 31 from the bottom of the crude argon column is sent back to the second column K02.

[0023] Oxygen rich liquid 27 is removed from the bottom of the second column K02 and vaporized in exchanger 103. Nitrogen rich gas 23 from the top of the second column K02 is also warmed in exchanger 103.

[0024] In Figure 2, column K10 operates at just above atmospheric pressure and stream 3 is compressed to 2.3 bara. During the first operation mode, in normal operation, the feed stream 1 containing between 5 and 15% mol argon removed from an intermediate point of the second column K02 to the bottom of the crude argon column K10 in which it is separated, forming an oxygen enriched stream at the bottom of crude argon column K10 and an argon enriched stream at the top of the crude argon column.

[0025] The crude argon column K10 has a top condenser E1 and a bottom reboiler E2. The bottom reboiler E2 does not function during the first operation mode, i.e. during normal operation. The top condenser E1 functions during the first operation mode, i.e. during normal operation but not in the second operation mode.

[0026] The top condenser E1 condenses a top gas of the crude argon column and sends the liquid formed back to the column as a reflux.

[0027] The argon enriched stream at the top of the crude argon column may be in liquid or gaseous form. It may be removed as a final product (not shown) or it may be sent to a pure argon column for further purification. Part of stream 3 may be produced as a product stream or may be further purified, this stream, if present, containing constituting at most 20% of the flow of that stream during the first operation mode. Cooling for the condenser E1 is provided by stream 113A from the bottom of the first distillation column K01 or another appropriate fluid.

[0028] During a second operation mode, the double column K01, K02 of Figure 1 does not function. This may be because of an accidental or deliberate stoppage of the column. For example, the main air compressor C01 may break down and cause an accidental stoppage. In the case of deliberate stoppage, when energy for the compressor C01 is not available, the double column K01, K02 may be closed down in a programmed way, whilst remaining preferably at cryogenic temperature or at least

temperatures below zero, this situation being known as a "cold standby". In this mode, no air is sent the first distillation column, no liquids are sent from the first to the second distillation column, no feed stream is sent from the second distillation column to the crude argon column and no stream containing more argon than the feed stream is removed from the top of the crude argon column as a product. Alternatively an argon enriched stream representing at most 20% of the argon enriched stream removed from the argon column K10 in the first operation mode is removed from the argon column in liquid or gaseous form.

[0029] Specific means, such as valves, may be provided for closing a conduit to prevent air being sent to the first distillation column and/or a conduit for sending the feed stream from the second distillation column to the crude argon column and/or a conduit for sending bottom liquid from the crude argon column to the second distillation column, during the second operation mode.

[0030] Stream 113A is not sent to the condenser E1 during the second operating mode. During shut-down of a double column (K01, K02), the argon column (K10) keeps functioning or is in a ready to function state, using a cycle stream (3) to heat a bottom reboiler (E2) of the argon column.

[0031] However, if the crude argon column remains in a state of operation, the purities of the fluids within the column remain stable or at least do not increase beyond an acceptable extent. A cycle stream 3 containing more argon than the feed stream, for example 80 to 99,5% mol argon or even up to 99,9% argon and less than 1ppm O₂, is removed from the top of the crude argon column K10, is sent to a heat exchanger E4, compressed in a compressor C, cooled in a cooler R using cooling water forming a cooled stream 5, cooled in heat exchanger E4 and then used to warm a bottom reboiler E2 of the crude argon column. The cycle stream is condensed in the bottom reboiler E2 to form a condensed cycle stream, the condensed cycle stream being sent to the top of the crude argon column after pressurization in a pump P.

[0032] If this cycle stream were not used, the liquid hold-up on the distributors and packings of the crude argon column would fall to the bottom of the column and time would be required to reestablish the liquid levels and the purity profile on restarting the crude argon column K10.

[0033] The use of the cycle stream to provide reboil for the crude argon column ensures that the liquid levels are maintained within the column at least to some extent. The use of the cycle stream to provide vapor and reflux to the crude argon column ensures that the purity profile is maintained along the crude argon column

[0034] Part of stream 3 may be produced as a product stream or may be further purified, this stream, if present, containing constituting at most 20% of the flow of that stream during the first operation mode.

[0035] It may be necessary to provide some additional refrigeration during the second operation mode to com-

pensate for E4 and P loss of cold and to subcool stream 8 in order to limit the downstream flash of stream 9

[0036] The crude argon column K10 of Figure 3 differs from that of Figure 2 in that part 3A of the cycle gas 3 warmed in heat exchanger E3 is sent to the compressor C and part 3B is vented. Thus in the second operation mode, the gas is warmed and then divided in two, one part being compressed and the rest vented. In Figure 3, there is a vent due to the fact that the cold losses and the flash are not compensated. This specific run case of Fig3 will last until a low level at the bottom of the K10 column is reached in the second operating mode.

[0037] In FIG 4 in both modes, column K10 operates at just above atmospheric pressure and stream 3 is compressed to 2.3 bara in compressor C so to be able to be condensed in reboiler E2. Typically stream 3 is compressed to between 0,2 and 2 bar above the pressure of the argon column K10.

[0038] In Figure 4, the exchanger E3 is not used. In the second operation mode, the cycle gas 3 is sent directly to the compressor C via valve V2 and then divided into parts 13A, 13B. Stream 13A is condensed in condenser E5 against a liquid nitrogen stream, by opening valve V3 using for example a PIC at the top of the column K10. Streams 13A downstream the condenser E5 and 13B are sent to a common phase separator S and gas from the phase separator is sent to the bottom reboiler E2 to be condensed. The condensed stream from E2 is sent to the separator S and the liquid 8 from the separator is pressurized in the pump P before being sent back to the column K10 via valve V4 as two-phase stream 9. The pump P is required to overcome the hydrostatic pressure, column K10 being over 60m in height.

[0039] During the first operation mode, the cycle gas is not used to reboil the crude argon column K10 and so the compressor C and pump P do not operate, for all of Figures 2 to 4.

[0040] Typically valve V2 control the flow 3, the column pressure is controlled via valve V3 and the level of separator S is controlled with valve V4.

[0041] A stream of argon enriched gas or liquid is produced from the top of column K10 (not shown).

[0042] During the second operation mode, the crude argon column K10 may be separated from the double column K01,K02 by closing valves on the conduit for sending the feed gas 1 to the crude argon column K10 and on the conduit for sending the bottom liquid from the crude argon column K10 to the second column K02.

[0043] It may be possible to operate during the second operation mode without closing any valve on the conduit for sending the feed gas 1 to the crude argon column K10, thus the valve will not necessarily be present or may have a particularly low pressure drop.

[0044] Part of stream 3 may be produced as a product stream or may be further purified, this stream, if present, containing constituting at most 20% of the flow of that stream during the first operation mode.

[0045] In these examples, the crude argon column K10

is comprised of a single column section, however it will be appreciated that the crude argon column may comprise two to four column sections.

[0046] Figures 5 and 6 show the example where the crude argon column is divided in two sections K10.1 and K10.2, K10.1 corresponding to the lower part of the crude argon column and K10.2 the upper part.

[0047] During the first operation mode for Figures 5 and 6, argon enriched gas 1 is sent from the second column K02 to the bottom of column K10.1, gas from the top of column K10.1 is sent to the bottom of column K10.2, liquid from the bottom of column K10.2 is sent to the top of column K10.1 and an argon rich product is removed at the top of column K10.2, under the top condenser E1. During the first operation mode, bottom reboiler E2 at the bottom of column K10.1 in Fig 5 and the bottom of column K10.2 in Fig 6 does not operate. A stream of argon enriched gas or liquid is produced from the top of column K10.2 (not shown).

[0048] During the second operation mode, for Figure 5, argon enriched gas 1 is not sent from the second column K02 to the bottom of column K10.1, however gas from the top of column K10.1 is sent to the bottom of column K10.2, liquid from the bottom of column K10.2 is sent to the top of column K10.1. No argon rich product is removed at the top of column K10.2, under the top condenser E1. During the second operation mode, bottom reboiler E2 at the bottom of column K10.1 in Fig 5 operates. Gas from the top of the column K10.2 is compressed in compressor C condensed in reboiler E2 to form condensed stream 7 which is pumped in pump P and sent to the top of column K10.2. Thus both columns K10.2 and K10.1 are in operation. During the second operation mode, for Figure 6, argon enriched gas 1 is not sent from the second column K02 to the bottom of column K10.1, gas from the top of column K10.1 is not sent to the bottom of column K10.2, liquid from the bottom of column K10.2 is not sent to the top of column K10.1. No argon rich product is removed at the top of column K10.2, under the top condenser E1. During the second operation mode, bottom reboiler E2 at the bottom of column K10.2 in Fig 6 operates. Gas 3 from the top of the column K10.2 is compressed in compressor C condensed in reboiler E2 to form condensed stream 7 which is pumped in pump P and sent to the top of column K10.2. Thus only column K10.2 is in operation.

[0049] Part of stream 3 may be produced as a product stream or may be further purified, this stream, if present, containing constituting at most 20% of the flow of that stream during the first operation mode.

Claims

1. Process for the separation of air by cryogenic distillation in which :

i) During a first operation mode, compressed

and purified air is sent to a first distillation column (K01) operating at a first pressure, an oxygen enriched liquid (109) is sent from the first distillation column to a second distillation column (K02) operating at a second pressure lower than the first pressure, a nitrogen enriched liquid (115) is sent from the first distillation column to the second distillation column, the first distillation column being thermally linked with the second distillation column via a bottom reboiler (43) of the second distillation column, an oxygen enriched fluid (27) is removed from the bottom of the second distillation column, a nitrogen enriched gas (23) is removed from the top of the second distillation column, a feed stream (1) containing between 5 and 15% mol argon is sent from the second column to the bottom of a crude argon column (K10) and a stream (3) containing proportionally more argon than the feed stream is removed from the top of the crude argon column and

iii) During a second operation mode, no air is sent the first distillation column, no liquids are sent from the first to the second distillation column and no feed stream is sent from the second distillation column to the crude argon column wherein during the second operation mode, a cycle stream (3) is removed from the top of the crude argon column, at least part of the cycle stream is used to warm a bottom reboiler (E2) of the crude argon column being thereby condensed to form a condensed cycle stream (7), the condensed cycle stream being sent to the top of the crude argon column and the stream, if present, containing proportionally more argon than the feed stream removed from the top of the crude argon column constitutes at most 20% of the flow of that stream during the first operation mode.

2. Process according to Claim 1 wherein during the first operation mode, no stream is used to warm the bottom reboiler (E2) of the crude argon column.
3. Process according to Claim 1 or 2 wherein during the second operation mode at least part of the cycle stream is compressed in a compressor (C) having a cryogenic inlet temperature upstream of the reboiler (E2).
4. Process according to any preceding claim wherein during the second operation mode the condensed cycle stream (7) is pressurized by a pump before being sent to the top of the crude argon column (K10).
5. Process according to any preceding claim wherein during the second operation mode, part (3B) of the

cycle stream (3) is vented or used as a product.

6. Process according to any preceding claim wherein during the second operation mode, part (13A) of the cycle stream is condensed in a condenser (E5) other than the bottom reboiler (E2) and then sent back to the crude argon column.

7. Process according to any preceding claim wherein during the second operation mode, any valves on (V1) conduits between the second column (K02) and the crude argon column (K10) are closed.

8. Process for the separation of air by cryogenic distillation in which :

i) During a first operation mode, compressed and purified air (105, 107) is sent to a first distillation column (K01) operating at a first pressure, an oxygen enriched liquid (109) is sent from the first distillation column to a second distillation column (K02) operating at a second pressure lower than the first pressure, a nitrogen enriched liquid (115) is sent from the first distillation column to the second distillation column, the first distillation column being thermally linked with the second distillation column via a bottom reboiler (43) of the second distillation column, an oxygen enriched fluid (27) is removed from the bottom of the second distillation column, a nitrogen enriched gas (23) is removed from the top of the second distillation column, a feed stream (1) containing between 5 and 15% mol argon is sent from the second column to the bottom of a crude argon column (K10) having a lower section (K10.1) and an upper section (K10.2) and a stream (3) containing proportionally more argon than the feed stream is removed from the top of the upper section of crude argon column and

iii) During a second operation mode, no air is sent to the first distillation column, no liquids are sent from the first to the second distillation column, no feed stream is sent from the second distillation column to the crude argon column in two sections and no stream containing more argon than the feed stream is removed from the top of upper section, the crude argon column wherein during the second operation mode, a cycle stream (3) is removed from the top of the crude argon column, at least part of the cycle stream is used to warm a bottom reboiler (E2) of the upper or lower section of the crude argon column being thereby condensed to form a condensed cycle stream (7), the condensed cycle stream being sent to the top of the upper section of the crude argon column and the stream, if present, containing proportionally more argon

than the feed stream removed from the top of the upper section constitutes at most 20% of the flow of that stream during the first operation mode.

9. Apparatus for the separation of air by cryogenic distillation comprising a first distillation column (K01) operating at a first pressure, a second distillation column (K02) operating at a second pressure lower than the first pressure, the second distillation column having a bottom reboiler (43), a crude argon column (K10) having a top condenser (E1) and a bottom reboiler (E2), a conduit for sending compressed and purified air (105, 107) to the first distillation column operating at a first pressure, a conduit for sending an oxygen enriched liquid (109) from the first distillation column to the second distillation column operating at a second pressure lower than the first pressure, a conduit for sending a nitrogen enriched liquid (115) from the first distillation column to the second distillation column, the first distillation column being thermally linked with the second distillation column via the bottom reboiler (43) of the second distillation column, a conduit for removing an oxygen enriched fluid (27) from the bottom of the second distillation column, a conduit for removing nitrogen enriched gas (23) from the top of the second distillation column, a conduit for sending a feed stream (1) containing between 5 and 15% mol argon from the second column to the bottom of a crude argon column, possibly a conduit for removing a stream containing more argon than the feed stream from the top of the crude argon column **characterized in that** it comprises a conduit for sending at least part of a cycle stream to the reboiler and a conduit for sending a condensed cycle stream from the reboiler to the top of the crude argon column and means for closing the conduit for sending air to the first distillation column and/or means for closing the conduit for sending the feed stream from the second distillation column to the crude argon column.

10. Apparatus according to Claim 9 comprising a compressor (C) for compressing the cycle stream (3) downstream of the removal from the crude argon column (K10) and upstream of the bottom reboiler (E2) of the crude argon column.

11. Apparatus according to Claim 9 or 10 comprising a pump for sending at least part of the cycle stream (7) in condensed form back to the crude argon column (K10).

12. Apparatus according to Claim 9, 10 or 11 comprising a conduit from sending bottom liquid from the crude argon column (K10) to the second distillation column (K10), said conduit being closable via closure means.

13. Apparatus according to any of Claims 9 to 12 including means for venting at least part (3B) of the cycle stream.

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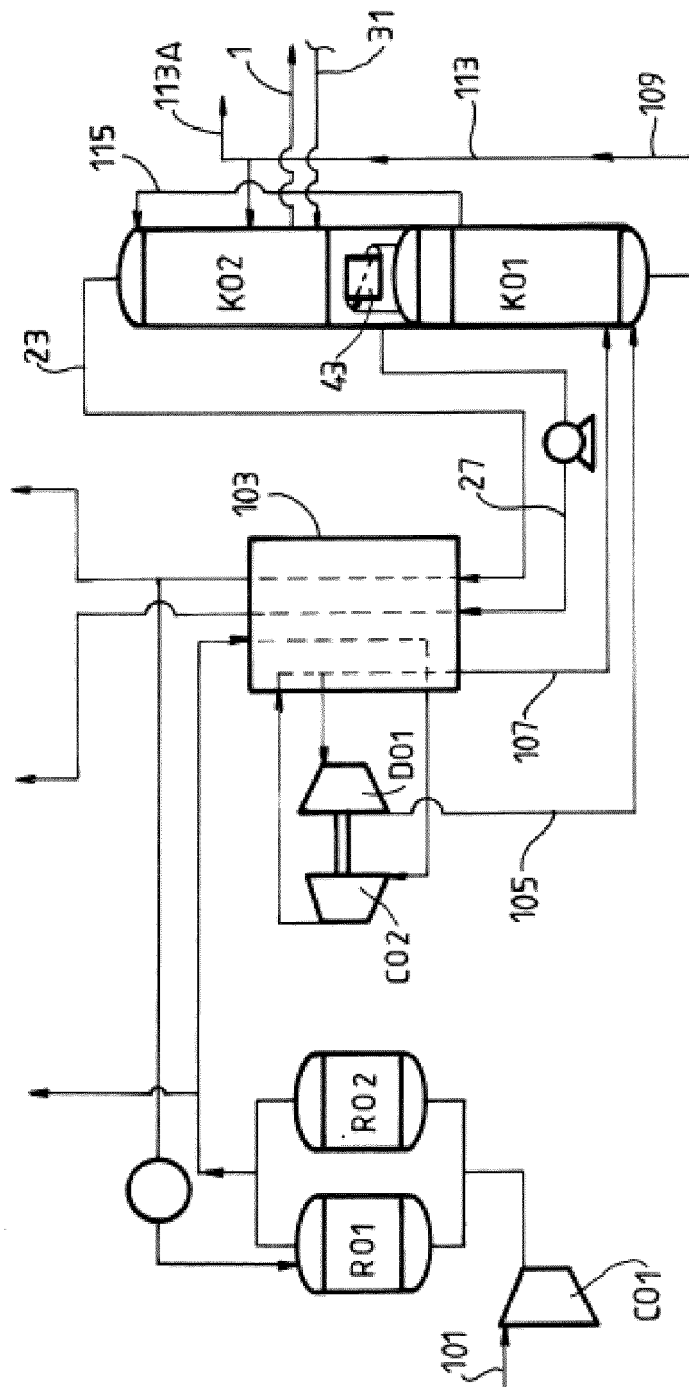


FIG.1

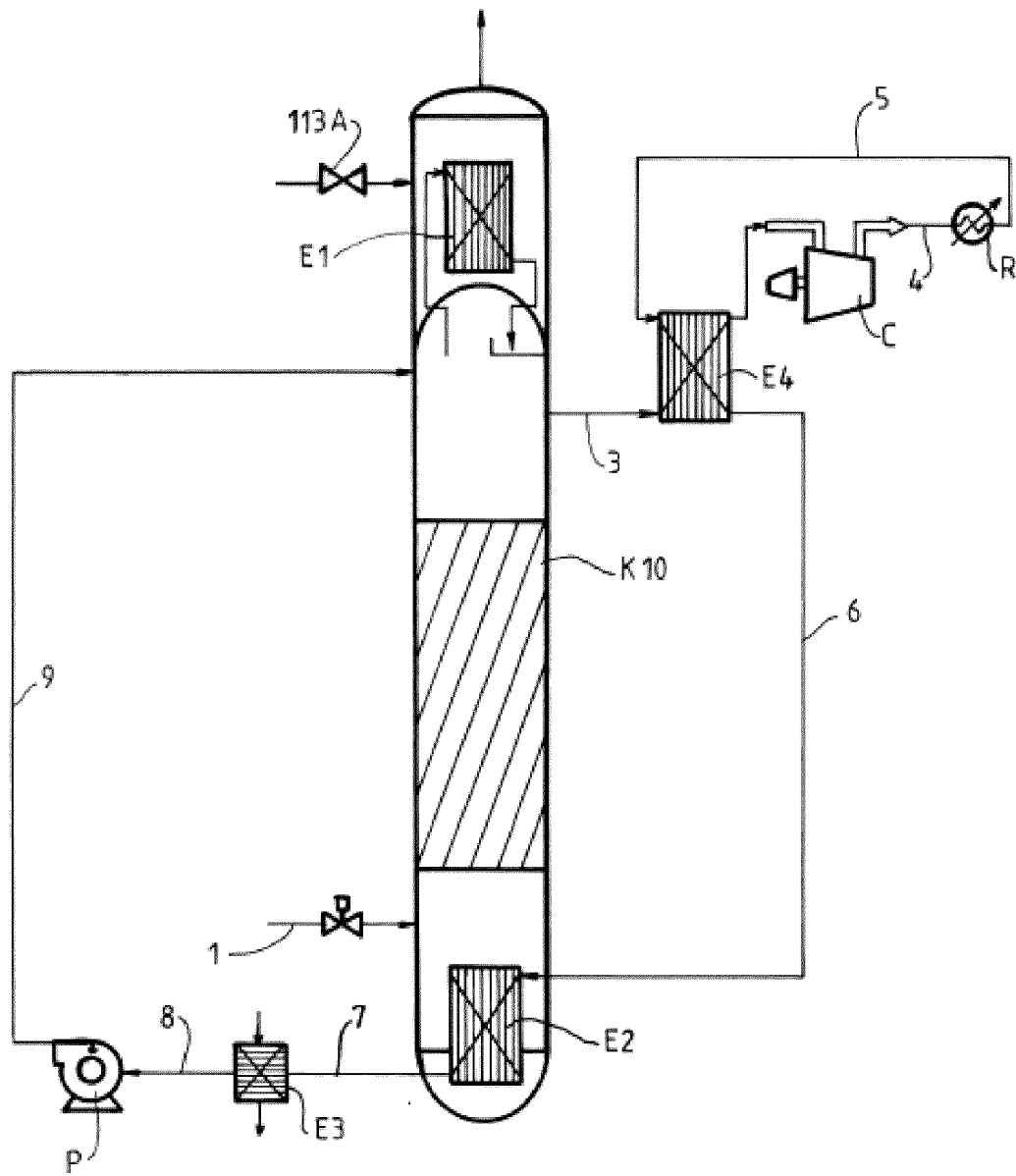


FIG. 2

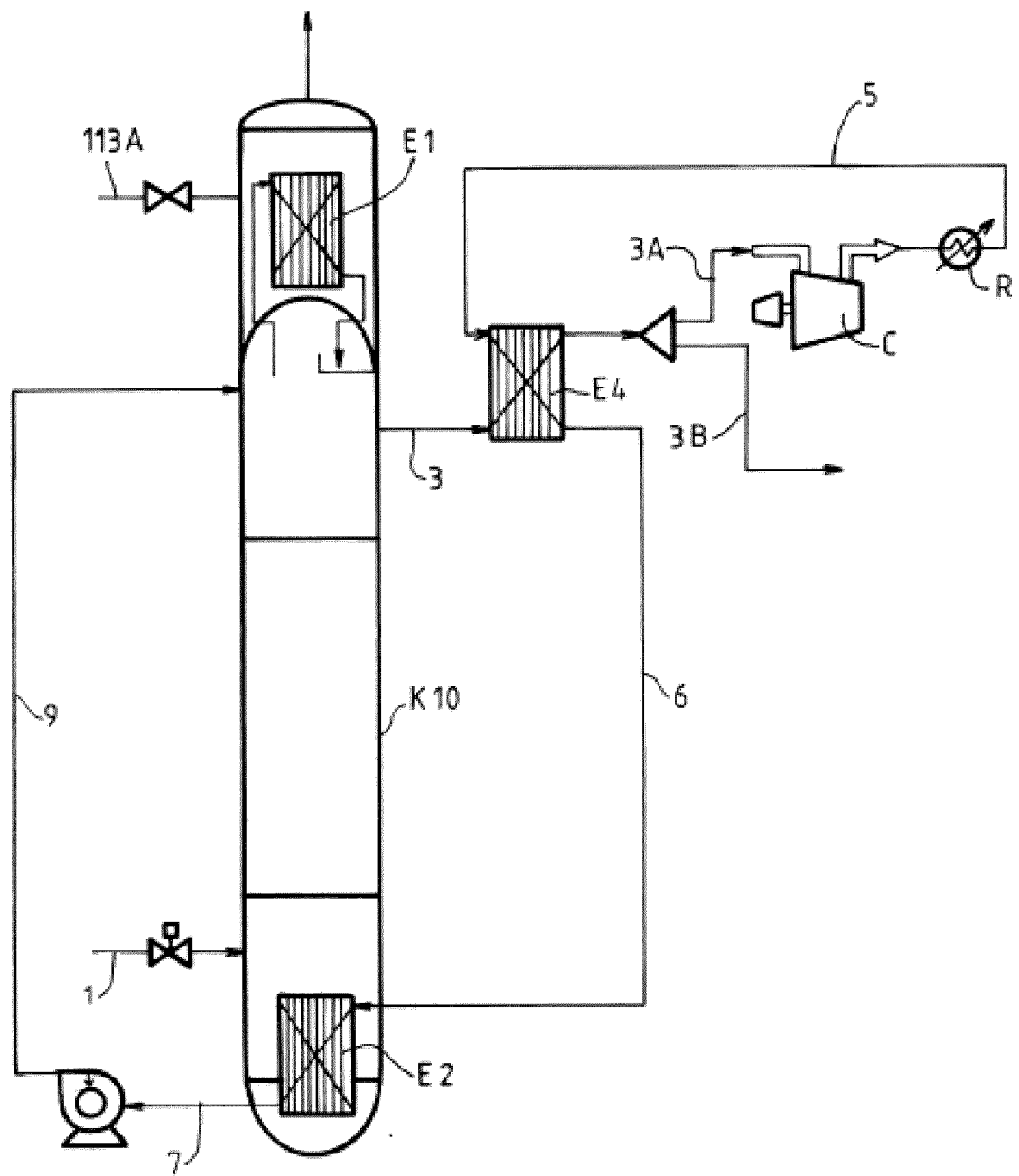


FIG. 3

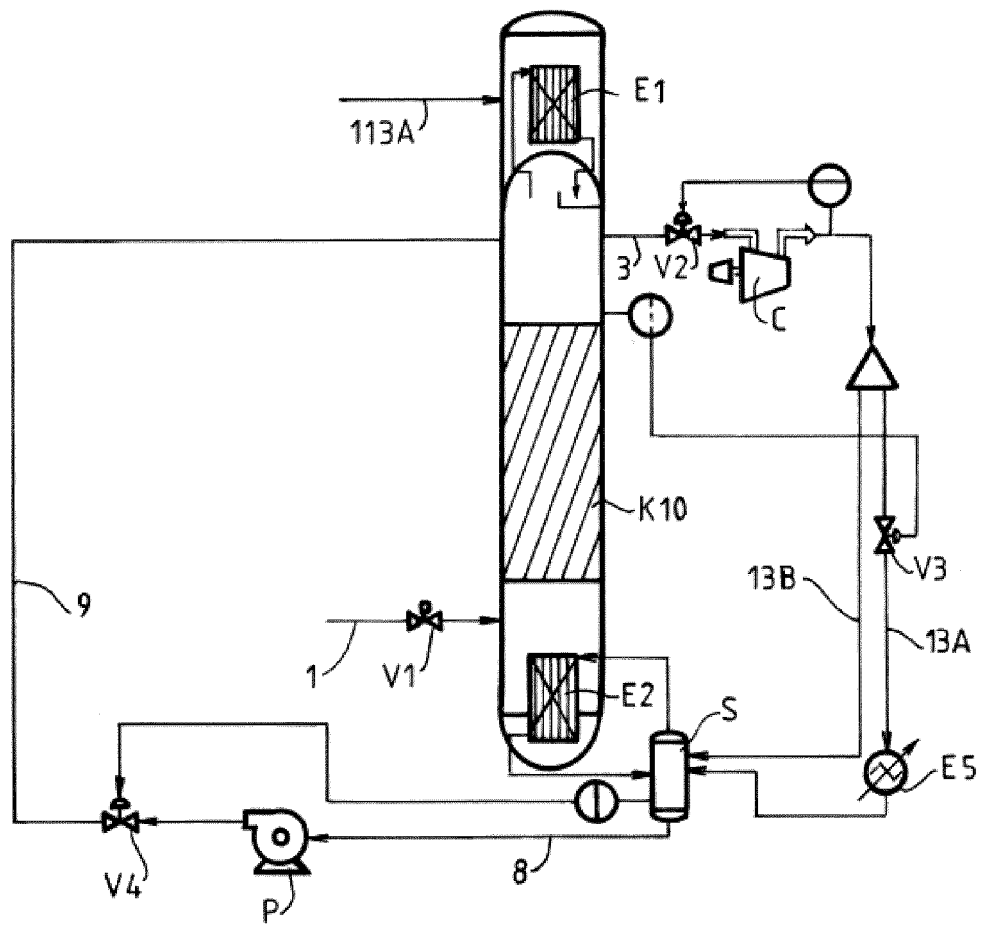


FIG. 4

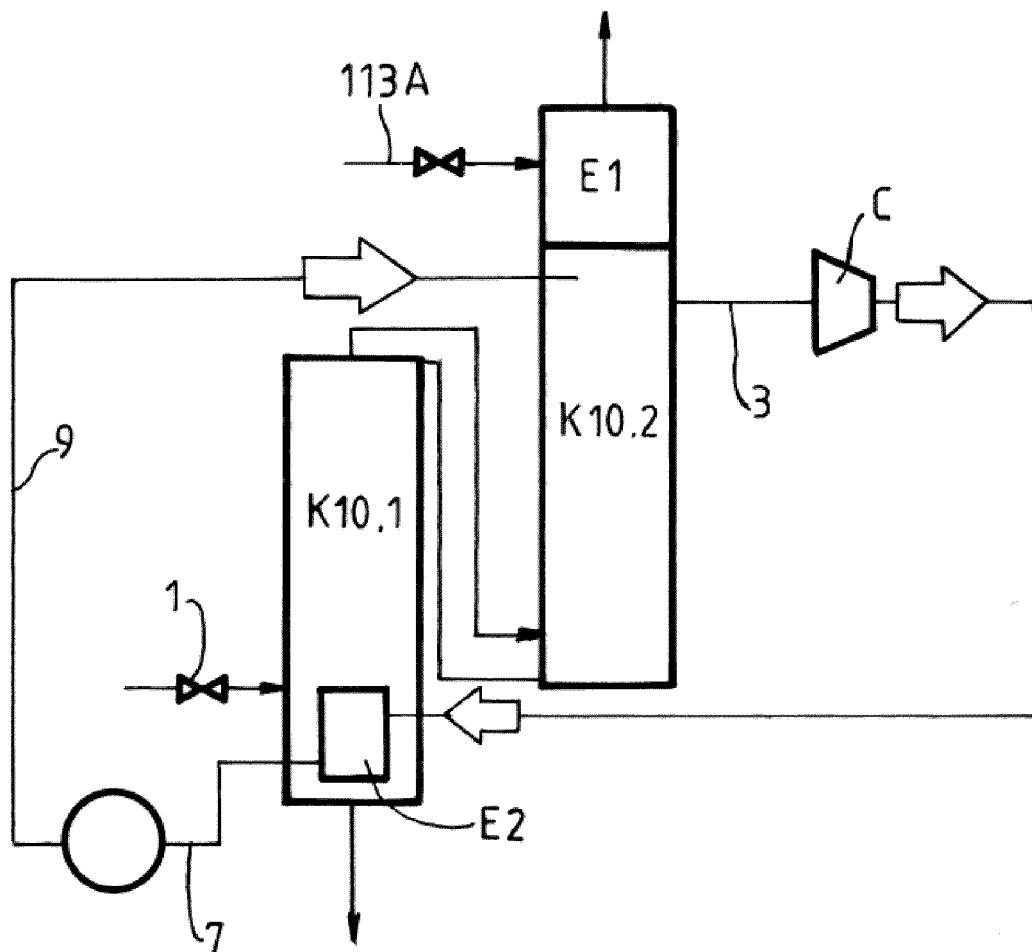


FIG. 5

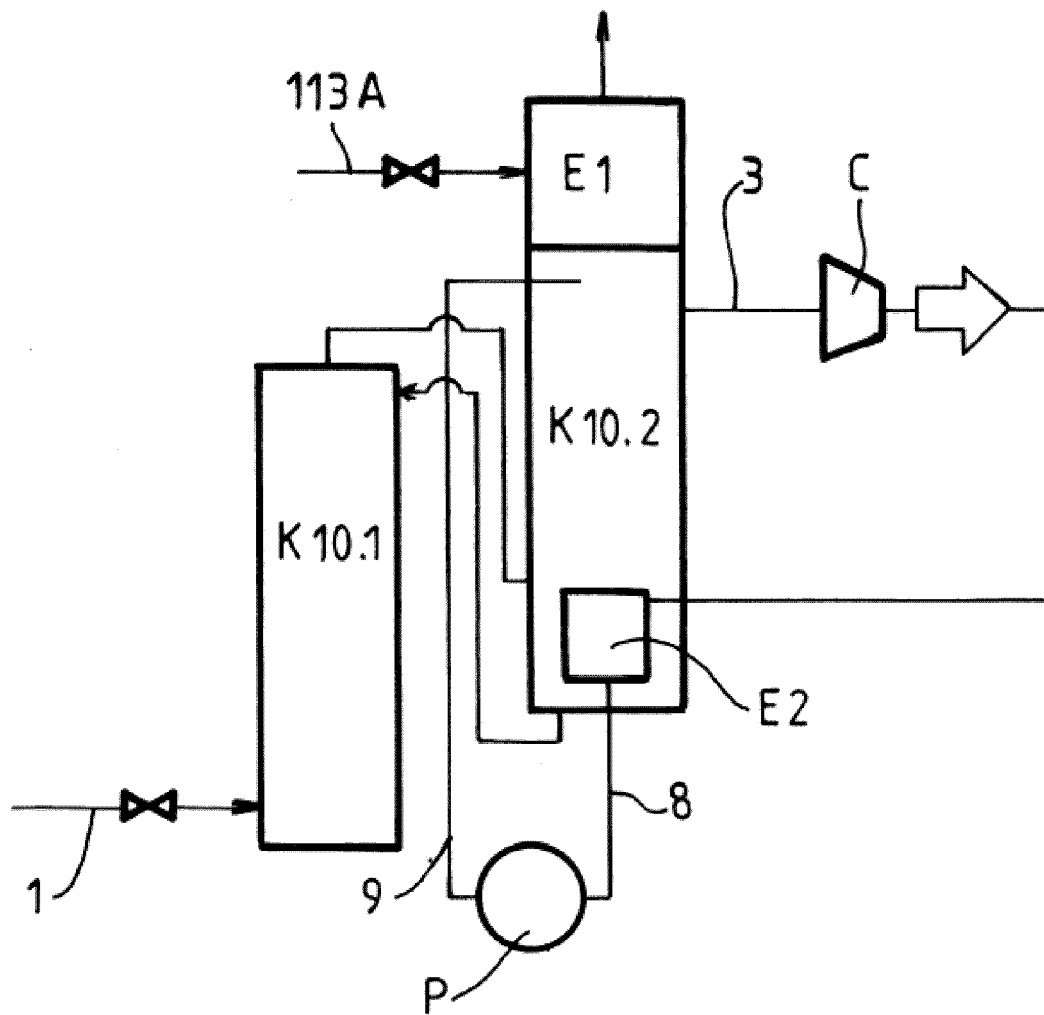


FIG. 6



EUROPEAN SEARCH REPORT

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Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
Y	US 4 057 407 A (BIGI EMANUELE) 8 November 1977 (1977-11-08) * column 2, line 59 - column 3, line 44; figure *	1-8	INV. F25J3/04
Y	DE 197 24 287 A1 (LINDE AG [DE]) 26 February 1998 (1998-02-26) * column 1, lines 28-48; figure 4 * * column 2, lines 40-66 * * column 5, line 56 - column 6, line 14 *	1-8	
Y	US 4 533 375 A (ERICKSON DONALD C [US]) 6 August 1985 (1985-08-06) * column 5, lines 28-35; figure 3 *	3,4	
Y	CN 115 682 630 A (SHAANXI JUNENG XINCHUANG COAL CHEMICAL TECH CO LTD) 3 February 2023 (2023-02-03) * figure 4 *	3	
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Y	JP H07 318241 A (NIPPON OXYGEN CO LTD) 8 December 1995 (1995-12-08) * figures 7,8 *	8	
The present search report has been drawn up for all claims			
Place of search Munich		Date of completion of the search 24 October 2023	Examiner Göritz, Dirk
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	



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CLAIMS INCURRING FEES

The present European patent application comprised at the time of filing claims for which payment was due.

☐ Only part of the claims have been paid within the prescribed time limit. The present European search report has been drawn up for those claims for which no payment was due and for those claims for which claims fees have been paid, namely claim(s):

☐ No claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for those claims for which no payment was due.

LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirements of unity of invention and relates to several inventions or groups of inventions, namely:

see sheet B

☐ All further search fees have been paid within the fixed time limit. The present European search report has been drawn up for all claims.

☐ As all searchable claims could be searched without effort justifying an additional fee, the Search Division did not invite payment of any additional fee.

☐ Only part of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the inventions in respect of which search fees have been paid, namely claims:

☒ None of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims, namely claims:

1-8

☐ The present supplementary European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims (Rule 164 (1) EPC).



LACK OF UNITY OF INVENTION **SHEET B**

Application Number

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The Search Division considers that the present European patent application does not comply with the requirements of unity of invention and relates to several inventions or groups of inventions, namely:

1. claims: 1-8

A process for the separation of air by cryogenic distillation in which :

i) During a first operation mode, compressed and purified air is sent to a first distillation column operating at a first pressure, an oxygen enriched liquid is sent from the first distillation column to a second distillation column operating at a second pressure lower than the first pressure, a nitrogen enriched liquid is sent from the first distillation column to the second distillation column, the first distillation column being thermally linked with the second distillation column via a bottom reboiler of the second distillation column, an oxygen enriched fluid is removed from the bottom of the second distillation column, a nitrogen enriched gas is removed from the top of the second distillation column, a feed stream containing between 5 and 15% mol argon is sent from the second column to the bottom of a crude argon column and a stream containing proportionally more argon than the feed stream is removed from the top of the crude argon column and

ii) During a second operation mode, no air is sent the first distillation column, no liquids are sent from the first to the second distillation column and no feed stream is sent from the second distillation column to the crude argon column wherein during the second operation mode, a cycle stream is removed from the top of the crude argon column, at least part of the cycle stream is used to warm a bottom reboiler of the crude argon column being thereby condensed to form a condensed cycle stream, the condensed cycle stream being sent to the top of the crude argon column.

2. claims: 9-13

An apparatus for the separation of air by cryogenic distillation comprising

- a first distillation column operating at a first pressure,
- a second distillation column operating at a second pressure lower than the first pressure, the second distillation column having a bottom reboiler,
- a conduit for sending compressed and purified air to the first distillation column,
- a conduit for sending an oxygen enriched liquid from the first distillation column to the second distillation column,
- a conduit for sending a nitrogen enriched liquid from the first distillation column to the second distillation column,
- the first distillation column being thermally linked with the second distillation column via the bottom reboiler of the second distillation column,
- a conduit for removing an oxygen enriched fluid from the



LACK OF UNITY OF INVENTION
SHEET B

Application Number

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The Search Division considers that the present European patent application does not comply with the requirements of unity of invention and relates to several inventions or groups of inventions, namely:

bottom of the second distillation column,
- a conduit for removing nitrogen enriched gas from the top
of the second distillation column,
- a crude argon column having a top condenser and a bottom
reboiler,
- a conduit for sending a feed stream containing between 5
and 15% mol argon from the second column to the bottom of a
crude argon column,
- a conduit for sending at least part of a cycle stream to
the reboiler and
- a conduit for sending a condensed cycle stream from the
reboiler to the top of the crude argon column
and further comprising means for closing the conduit for
sending air to the first distillation column and/or means
for closing the conduit for sending the feed stream from the
second distillation column to the crude argon column.

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

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This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

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