(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 3 916 330 A1

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:
01.12.2021 Bulletin 2021/48

(51) Int Cl.:
F25J 3/04 *(2006.01)*

(21) Application number: **20212567.0**

(22) Date of filing: **08.12.2020**

(84) Designated Contracting States:
AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR
Designated Extension States:
BA ME
Designated Validation States:
KH MA MD TN

(71) Applicant: **L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCEDES GEORGES CLAUDE**
75007 Paris (FR)

(72) Inventors:
- **SUDRE, Terry**
1130 BRUSSELS (BE)
- **HENDRICKX, Eric**
1130 BRUSSELS (BE)
- **BUYSSE, Cédric**
1130 BRUSSELS (BE)
- **STUER, Sieger**
1130 BRUSSELS (BE)
- **MEERT, Kurt**
1130 BRUSSELS (BE)
- **MARCON, Luigi**
1130 BRUSSELS (BE)
- **COCK, Felix**
1130 BRUSSELS (BE)

(74) Representative: **Air Liquide**
L'Air Liquide S.A.
Direction de la Propriété Intellectuelle
75, Quai d'Orsay
75321 Paris Cedex 07 (FR)

(54) PROCESS FOR START-UP OF A CRYOGENIC DISTILLATION AIR SEPARATION UNIT

(57) In a process for restarting an air separation unit for the separation of air by cryogenic distillation, the unit comprising a first column (K1) operating in normal operation at a first pressure and a second column (K2) operating in normal operation at a second pressure lower than the first pressure, the first and second columns being thermally linked and provided with conduits (2,4) for transferring liquid enriched in oxygen and liquid enriched in nitrogen from the first column to the second column under normal operation, conduits for removing oxygen enriched fluid (7) and nitrogen enriched gas (8) from the second column in normal operation and a conduit for feeding gaseous air (1) to the first column under normal operation, during restart of the air separation unit, liquid (3) is removed from the bottom of the second column and sent to the bottom of the first column and liquid oxygen (5) from a second external source (S) is sent to the bottom of the second column.

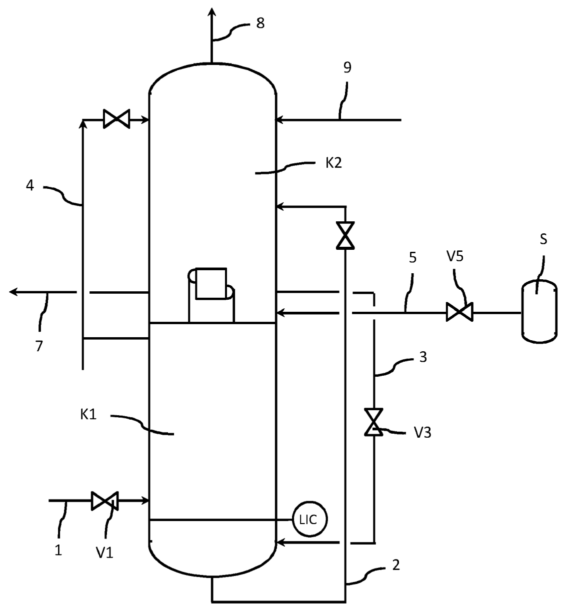

Figure 1

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Description

[0001] The present invention relates to a process for the start up of a cryogenic distillation air separation unit. It also relates to a cryogenic distillation air separation unit capable of being started up according to the process.

[0002] All air separation units for the cryogenic distillation of air need to be closed down from time to time, either for maintenance work or because their products are not required. If the unit no longer operates, heat penetrates into the cold box and the columns warm up. This means that once the unit restarts, considerable time is wasted while the cold box cools down in order for distillation to resume and for the necessary purities to be established.

[0003] For this reason, it is known to maintain the cold box at cryogenic temperatures by processes known as "cold standby" or "cold hold".

[0004] The EIGA document 65/30 "Safe Operation of Reboilers/Condensers in Air Separation Unit" sets out a definition of cold standby as being the condition where equipment is held at cryogenic conditions for immediate service on demand.

[0005] The section on "cold standby" reads as follows: "Following a plant shutdown, the liquid contained in the packing or trays drains to the low point of the column. At this point, the level in the reboilers can be well greater than 100% of the level indicators. This liquid is generally preserved to permit a quick restart of the plant. This mode is referred to as cold standby.

[0006] During a one day or two day cold standby period, flow is circulated through liquid oxygen adsorbers if installed. The following shall be followed to maintain a more extended cold standby in a safe manner:

- close inlet and outlet valves of the recirculation pumps;
- drain pumps and suction filters; and
- drain and regenerate liquid oxygen adsorbers. The regeneration should be done soon after complete draining of the liquid and purging of the adsorber with nitrogen to avoid the release of contaminants in gaseous oxygen atmosphere.

[0007] Due to the heat leak into the coldbox, vaporization eventually reduces the amount of liquid in the reboiler to well less than the normal operating level.

[0008] When the liquid has been reduced to half of its normal level, either liquid shall be added using liquid nitrogen or oxygen injection or all liquid in the plant shall be drained.

[0009] When injecting liquid into the plant, it is advisable to use liquid nitrogen rather than liquid oxygen in particular for a long-term cold standby. When liquid oxygen is used, extra hydrocarbons, carbon dioxide, nitrous oxide, and contaminants in general are added to the system and could accumulate to exceed the maximum contaminant levels."

[0010] It is thus known to maintain the temperature in the columns within the coldbox by adding liquid nitrogen to the air separation unit. Since no distillation takes place and no air enters the columns during cold standby, the liquid nitrogen sent to the top of the low pressure column accumulates at the bottom of that column and is vaporised to a certain extent, due to the heat ingress. This vaporization helps to ensure that the columns remain under a positive pressure and limits the possibility that fluids should enter the columns through the conduits normally used to introduce air or remove products.

[0011] The present invention proposes a procedure to speed up the start up process by accelerating the transition from the cold standby where the bottom of the low pressure column contains liquid nitrogen and normal operation where the bottom of the low pressure column contains oxygen enriched liquid.

[0012] According to the present invention, there is provided a process for restarting an air separation unit for the separation of air by cryogenic distillation, the unit comprising a first column operating in normal operation at a first pressure and a second column operating in normal operation at a second pressure lower than the first pressure, the first and second columns being thermally linked and provided with conduits for transferring liquid enriched in oxygen and liquid enriched in nitrogen from the first column to the second column under normal operation, conduits for removing oxygen enriched fluid and nitrogen enriched gas from the second column in normal operation and a conduit for feeding gaseous air to the first column under normal operation wherein

- when the air separation unit is shut down, no air is sent to the first column and liquid nitrogen from a first external source is sent at least occasionally to the second column and
- during restart of the air separation unit:

  a) Liquid is removed from the bottom of the second column and sent to the bottom of the first column,
  b) Liquid nitrogen is no longer sent to the second column and liquid oxygen from a second external source is sent to the bottom of the second column and
  c) Gaseous air is sent to the first column.

[0013] According to other optional features:

- no gaseous air is sent to the first column during restart until a given quantity of liquid has been sent from the bottom of the second column to the bottom of the first column.
- no gaseous air is sent to the first column during restart until liquid oxygen has been sent to the bottom of the second column.
- no liquid oxygen is sent to the bottom of the second column until a given quantity of liquid has been sent

from the bottom of the second column to the bottom of the first column.

- liquid nitrogen is sent to the second column during normal operation.
- the liquid nitrogen sent to the second column during normal operation provides at least 90% of the refrigeration needed for the process.
- steps a) and b) do not take place during a common time period.

[0014] According to another object of the invention, there is provided an air separation unit for the separation of air by cryogenic distillation, the unit comprising a first column operating in normal operation at a first pressure and a second column operating in normal operation at a second pressure lower than the first pressure, the first and second columns being thermally linked and provided with a conduit for transferring liquid enriched in oxygen from the first column to the second column under normal operation and a conduit for transferring liquid enriched in nitrogen from the first column to the second column under normal operation, conduits for removing oxygen enriched fluid and nitrogen enriched gas from the second column in normal operation and a conduit for feeding gaseous air to the first column under normal operation characterized in that it comprises means for sending liquid oxygen from an external source connected to the bottom of the second column and a conduit for sending liquid from the bottom of the second column to the bottom of the first column.

[0015] According to optional features, the unit comprises:

- means for detecting the amount of liquid sent from the bottom of the second column to the bottom of the first column and means for sending liquid oxygen to the bottom of the second column once a given amount of liquid has been sent from the bottom of the second column to the bottom of the first column.
- means for detecting the liquid level at the bottom of the first column and means for sending air to start up the distillation in response to a signal from the means for detecting the liquid level.
- means for detecting the pressure difference between the first and second columns and and means for sending liquid oxygen to the bottom of the second column once the pressure difference is sufficient.

[0016] The invention will be described in greater detail with reference to the figure.

[0017] Figure 1 shows a cryogenic air separation unit comprising a first column K1 operating in normal operation at a first pressure and a second column K2 operating in normal operation at a second pressure lower than the first pressure.

[0018] The first and second columns are thermally linked and provided with a conduit 2 for transferring liquid enriched in oxygen from the first column to the second column under normal operation and a conduit 4 for transferring liquid enriched in nitrogen from the first column to the second column under normal operation.

[0019] Conduit 7 serves to remove oxygen enriched fluid from the bottom of the second column K2 and conduit 8 removes nitrogen enriched gas from the second column K2 in normal operation.

[0020] Conduit 3 does not serve during normal operation.

[0021] A conduit 1 feeds gaseous air to the first column K1 under normal operation. The air in conduit 1 has previously been purified and cooled. It is possible to cool the air using a refrigeration unit upstream of the purification step.

[0022] When the air separation unit is shut down, no air is sent to the first column K1 and liquid nitrogen 9 from a first external source is sent at least occasionally to the second column K2. This allows the columns K1, K2 to remain at a low temperature even though distillation is not taking place. Whilst the air separation unit is shut down, liquid from within the column drains to the bottom of the column and accumulates. This liquid is much richer in nitrogen than the liquid which accumulates at the bottom of the column during normal operation.

[0023] During restart of the air separation unit, valve V3 is opened, liquid is removed from the bottom of the second column K2 via conduit 3 and sent to the bottom of the first column K1.

[0024] Once sufficient liquid has been sent to the bottom of the first column K1, valve V3 is closed and no more liquid is removed via conduit 3. Instead liquid oxygen from a second external source S is sent via conduit 5 to the bottom of the second column K2 by opening valve V5.

[0025] The amount of liquid drained via conduit 3 depends on the purity of the liquid drained and the purity of the liquid oxygen to be added by conduit 5. The aim is to establish a liquid bath at the bottom of the second column K2 having an oxygen purity greater than that existing during shutdown so as to approach to some degree the oxygen purity existing during distillation. For example, the liquid purity at the bottom of the column could reach between 40 and 50% mol oxygen due to the addition of liquid oxygen. This value will depend on the type of air separation unit.

[0026] The temperature of the liquid in the bath will increase with the oxygen content. For a given Delta T (design value) of the bottom condenser of the second column K1, the temperature on the nitrogen side (column K1) will also increase. During start up, the product that will be condensed is air. The temperature on the nitrogen side must be high enough so that the corresponding pressure (point on the boiling curve of air) is high enough to transfer the condensed liquids from K1 to K2 by pressure differential.

[0027] Consequently the sending of liquid oxygen to the bottom of second column K2 continues until the pressure in the first column K1 is sufficient to ensure that the liquid reflux flows can overcome the hydrostatic pressure and arrive in the second column K2.

[0028] This is checked by measuring the liquid level at the bottom of the first column K1 using level detector LIC. If, on opening the valve V3, the liquid level at the bottom of the first column K1 decreases, then it can be concluded that the pressure is sufficient to cause the bottom liquid to rise through conduit 2 to the second column K2.

[0029] Finally once a certain level of liquid oxygen is established at the bottom of the second column K2, valve V1 opens and gaseous air is sent to column K1 to enable distillation to begin.

[0030] Thus the unit includes means for detecting the liquid level at the bottom of the first column and means for sending air to start up the distillation in response to a signal from the means for detecting the liquid level.

[0031] Optionally, the unit may comprise means for detecting the pressure difference between the first and second columns and means for sending liquid oxygen to the bottom of the second column once the pressure difference is sufficient.

[0032] It is useful before starting the distillation to send an air stream to the refrigeration unit in series with the purification unit. Since the minimum flow for the refrigeration unit to operate is higher than the minimum flow for the purification unit to operate, the excess air from the refrigeration unit can be sent to the atmosphere.

Claims

1. Process for restarting an air separation unit for the separation of air by cryogenic distillation, the unit comprising a first column (K1) operating in normal operation at a first pressure and a second column (K2) operating in normal operation at a second pressure lower than the first pressure, the first and second columns being thermally linked and provided with conduits (2,4) for transferring liquid enriched in oxygen and liquid enriched in nitrogen from the first column to the second column under normal operation, conduits for removing oxygen enriched fluid (7) and nitrogen enriched gas (8) from the second column in normal operation and a conduit for feeding gaseous air (1) to the first column under normal operation wherein

    - when the air separation unit is shut down, no air is sent to the first column and liquid nitrogen (9) from a first external source is sent at least occasionally to the second column
    - and during restart of the air separation unit:

        a) Liquid (3) is removed from the bottom of the second column and sent to the bottom of the first column,
        b) Liquid nitrogen is no longer sent to the second column and liquid oxygen (5) from a second external source (S) is sent to the bottom of the second column and
        c) Gaseous air is sent to the first column.

2. Process according to Claim 1 wherein no gaseous air is sent to the first column (K1) during restart until a given quantity of liquid (3) has been sent from the bottom of the second column to the bottom of the first column.

3. Process according to Claim 1 or 2 wherein no gaseous air (1) is sent to the first column (K1) during restart until liquid oxygen (5) has been sent to the bottom of the second column (K2).

4. Process according to any preceding claim wherein no liquid oxygen (5) is sent to the bottom of the second column (K2à until a given quantity of liquid has been sent from the bottom of the second column (3) to the bottom of the first column (K1).

5. Process according to any preceding claim wherein liquid nitrogen (9) is sent to the second column during normal operation.

6. Process according to any preceding claim wherein the liquid nitrogen (9)sent to the second column during normal operation provides at least 90% of the refrigeration needed for the process.

7. Process according to any preceding claim wherein steps a) and b) do not take place during a common time period.

8. Air separation unit for the separation of air by cryogenic distillation, the unit comprising a first column (K1) operating in normal operation at a first pressure and a second column (K2) operating in normal operation at a second pressure lower than the first pressure, the first and second columns being thermally linked and provided with a conduit for transferring liquid enriched in oxygen (2) from the first column to the second column under normal operation and a conduit (4) for transferring liquid enriched in nitrogen from the first column to the second column under normal operation, conduits for removing oxygen enriched fluid (7) and nitrogen enriched gas (8) from the second column in normal operation and a conduit for feeding gaseous air (1) to the first column under normal operation **characterized in that** it comprises means for sending liquid oxygen (5) from an external source (S) connected to the bottom of the second column and a conduit (3) for sending liquid from the bottom of the second column to the bottom of the first column.

9. Unit according to Claim 8 comprising means for detecting the amount of liquid (3) sent from the bottom

of the second column (K2) to the bottom of the first column (K1) and means for sending liquid oxygen (5) to the bottom of the second column once a given amount of liquid has been sent from the bottom of the second column to the bottom of the first column.

10. Unit according to Claim 8 or 9 including means (LIC) for detecting the liquid level at the bottom of the first column and means for sending air (1) to start up the distillation in response to a signal from the means for detecting the liquid level.

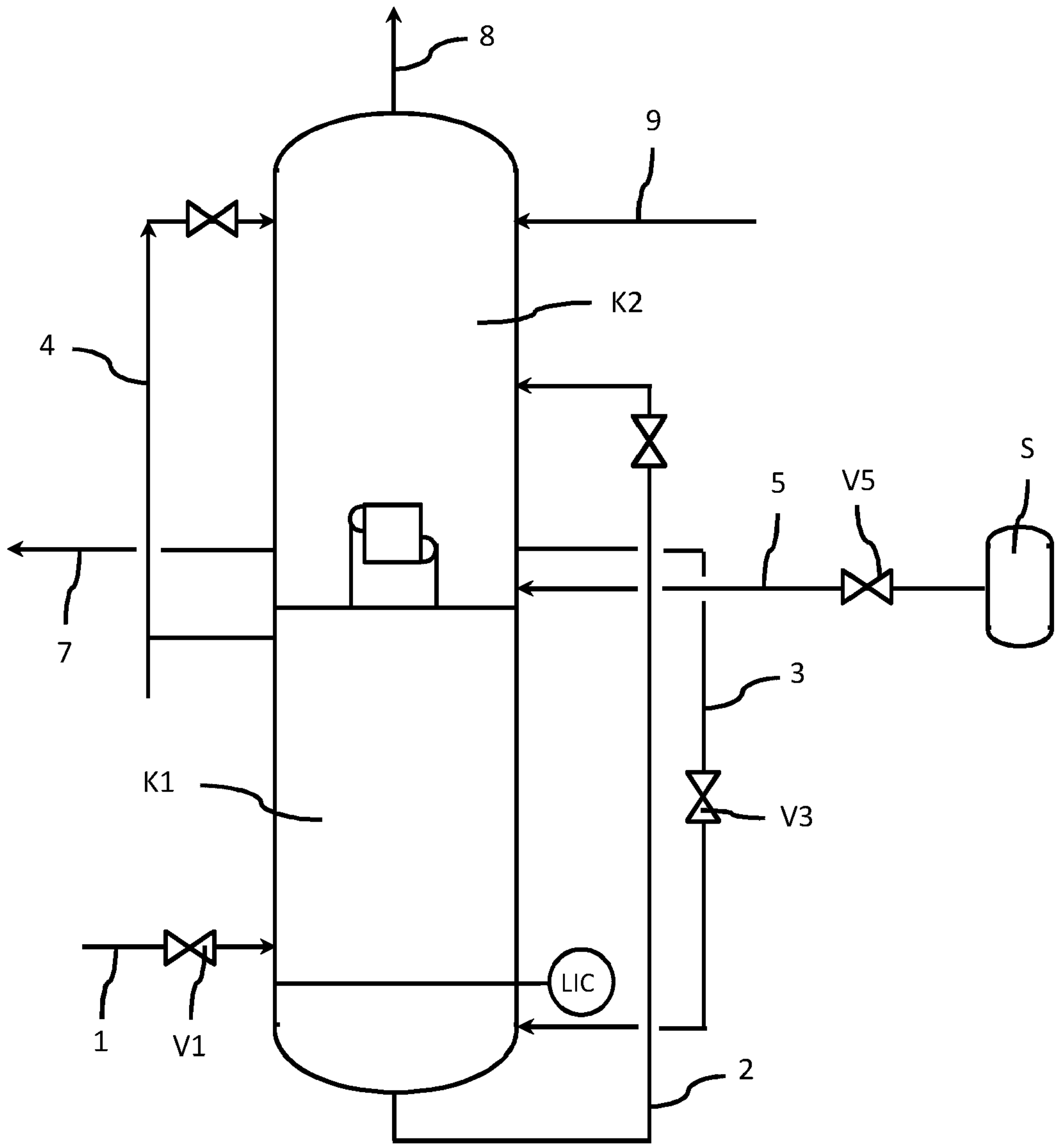

Figure 1

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EUROPEAN SEARCH REPORT

Application Number
EP 20 21 2567

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
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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	28 April 2021	Göritz, Dirk

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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Application Number
EP 20 21 2567

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
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TECHNICAL FIELDS SEARCHED (IPC)

2

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