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(54) **AIR SEPARATION PROCESS USING CRYOGENIC DISTILLATION**

KRYOGENE DESTILLATION VERWENDENDER LUFTTRENNPROZESS

PROCEDE DE SEPARATION DE L'AIR PAR DISTILLATION CRYOGENIQUE

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(73) Proprietor: **L'AIR LIQUIDE, Société Anonyme pour l'Etude et l'Exploitation des Procédés Georges Claude**
**75007 Paris (FR)**

(72) Inventors:
- **BRUDER, Olivier**
  **F-75321 PARIS CEDEX 07 (FR)**
- **LE BOT, Patrick**
  **F-94300 VINCENNES (FR)**
- **DUBETTIER-GRENIER, Richard**
  **F-94210 LA VARENNE SAINT-HILAIRE (FR)**

(74) Representative: **Mercey, Fiona Susan**
**L'Air Liquide SA,**
**Département Propriété Intellectuelle,**
**75 quai d'Orsay**
**75321 Paris Cedex 07 (FR)**

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## Description

**[0001]** The present invention relates to an air separation process using cryogenic distillation. In particular, it relates to air separation processes for the production of large quantities of oxygen with a content of greater than 90 mol% with a yield of greater than 90% in a unit comprising at least one column operating at low pressure and at least one column operating at high pressure, at least one column of which containing at least one section of structured packings of the cross-corrugated type.

**[0002]** Cross-corrugated packings are described in EP-A-0 367 817 and EP-A-0 491 591.

**[0003]** US-A-5653126 describes the use of cross-corrugated structured packings having a density of at least 500 $m^2/m^3$ with a low HETP for the separation of air.

**[0004]** Typically, cross-corrugated packings have corrugations at 45° with the axis of the column in which they are installed. The packings may or may not be perforated and are generally formed by folded metal foil strips.

**[0005]** The increase in demand for gas separation units of large capacity (production greater than 2000 tonnes of gas produced per day) has led to the need to design distillation columns of increasing diameter, despite the frequent use of pressurized distillation processes (with columns operating at pressures above 2 bar abs).

**[0006]** Transport problems become significant when the diameter of the elements exceeds 3m, or even 4.5m, and these are sometimes insurmountable for columns greater than 6m in diameter. If it was desired to increase the ratio of oxygen production (in t/d of pure product) to cross section (in $m^2$) of the low-pressure column, that is to say to reduce the diameter of the low-pressure column for constant oxygen production (or given air throughput), for a unit comprising a medium-pressure column thermally coupled to a low-pressure column or for a unit comprising three columns, including a medium-pressure column thermally coupled to a low-pressure column and an intermediate-pressure column fed from the medium-pressure column, the only solutions were:

- to increase the withdrawal of nitrogen from the medium-pressure column (or from the intermediate-pressure column), but above a level of withdrawal representing between 15-20% of the air throughput, it is found that:
  - ○ either the oxygen yield of the unit drops dramatically for a given oxygen purity
  - ○ or the oxygen purity drops dramatically for a given oxygen withdrawal;
- to increase the pressure of the unit (low pressure > 2 bar abs), but this is highly penalizing from the energy standpoint if the pressure of the waste nitrogen is not utilized (that is to say by expansion in order to make liquid, or as product pressure).

**[0007]** In an air separation unit producing large quantities of oxygen and nitrogen, if it was desired to reduce the diameter of this column, the only solutions were:

- to increase the withdrawal of lean liquid and of liquid nitrogen, but above a certain ratio it is found that the purity of the nitrogen and of the lean liquids dramatically drops and that the oxygen yield of the unit collapses;
- to increase the flow of blown air, but above a certain ratio the performance of the unit is greatly degraded - reduction in oxygen yield (or in oxygen purity), reduction in medium-pressure gaseous nitrogen (or in purity of medium-pressure gaseous nitrogen);
- to increase the liquid air flow, but this flow is set by the production of pumped products and the production of liquid products (liquid oxygen, liquid nitrogen, liquid argon), or a substantial loss of energy;
- to increase the pressure of the unit (with a medium pressure > 8 bar abs), but this is highly disadvantageous from the energy standpoint if the pressure of the waste nitrogen is not utilized (that is to say by expansion in order to make liquid, or as product pressure).

**[0008]** The object of the present invention is to alleviate the problems of the prior art by allowing the column diameters to be reduced for a given oxygen production.

**[0009]** One object of the invention is to provide a process according to Claim 1.

**[0010]** The yield is the percentage number of oxygen molecules in all the oxygen-rich products relative to the number of oxygen molecules in the feed air for the separation unit.

**[0011]** The capacity per unit area of the column is the ratio of the molar volume of gas sent into the column to the mean cross section of the column.

**[0012]** According to other optional features:

- the largest-diameter column of the column system contains at least one section filled with a gas/liquid contactor allowing material exchange between the gaseous phase and the liquid phase such that its effectiveness is defined by a TUH (Transfer Unit Height) of less than 350 mm (preferably less than 300 mm);
- the gas/liquid contactor is composed of cross-corrugated structured packings having a density of less than 500 $m^2/m^3$, preferably less than 400 $m^2/m^3$;
- the column system comprises at least one double column formed by a medium-pressure column and a low-pressure column, the medium-pressure column being thermally coupled to the low-pressure column, and the low-pressure column constituting the largest-diameter column;
- the ratio of the oxygen produced to the cross section of the low-pressure column is greater than or equal to 150 $t/d/m^2$;

- the ratio of the flow rate of air sent to the column system to the cross section of the low-pressure column is not less than 22153 $Nm^3/h/m^2$ (21000 $Sm^3/h/m^2$), preferably not less than 24263 $Nm^3/h/m^2$ (23000 $Sm^3/h/m^2$) and even more preferably not less than 26372 $Nm^3/h/m^2$ (25000 $Sm^3/h/m^2$);
- the ratio of the gaseous air sent to the medium-pressure column to the cross section of the low-pressure column is not less than 18988 $Nm^3/h/m^2$ (18000 $Sm^3/h/m^2$) (preferably not less than 21098 $Nm^3/h/m^2$ (20000 $Sm^3/h/m^2$) and even more preferably not less than 23208 $Nm^3/h/m^2$ (22000 $Sm^3/h/m^2$)); and
- at least one section of the medium-pressure column contains cross-corrugated structured packings with a density not exceeding 500 $m^2/m^3$, or even 400 $m^2/m^3$.

**[0013]** A fluid will be called a "product" if it is output from the air separation unit with the content of one of its constituents being:

- greater than 85 mol%, if the constituent is oxygen;
- greater than 95 mol%, if the constituent is nitrogen.

**[0014]** The extraction efficiency of a constituent will be defined by the ratio of the molar quantity of the said constituent entering the separation unit via the feed fluids to the molar quantity of the same constituent leaving via the product(s), the content of this constituent being greater than or equal to those mentioned above.

**[0015]** Cross-corrugated structured packings with a density not exceeding 500 $m^2/m^3$, or even 400 $m^2/m^3$, may be installed in the medium-pressure column and/or the low-pressure column of a double column, in the medium-pressure column and/or the low-pressure column and/or the intermediate-pressure column of a triple (Etienne) column, an argon column, a mixing column or any other type of air gas separation column.

**[0016]** A packing has a density of less than 500 $m^2/m^3$ if its mean density (without deducting the area of any perforations) does not exceed 500 $m^2/m^3$.

**[0017]** The invention will be described in greater detail with reference to the figure.

**[0018]** Figure 1 shows a process according to the invention.

**[0019]** In a unit according to the invention, a double column comprises a medium-measure column 1 operating at between 5 bar abs and 20 bar abs and a low-pressure column 3 operating at apressure between 1.2 bar abs and 8 bar abs. The low-pressure column is larger in diameter than the medium-pressure column, but it is possible in some cases for the medium-presure column to be larger in diameter than the low pressure column. The two columns are thermally coupled to each other by a reboiler/condensor 5, for example of the bath type.

**[0020]** The low-pressure column 3 contains only sections of structured packings 2 having a density not exceeding 500 $m^2/m^3$, for example having a density of 400 $m^2/m^3$.

**[0021]** The medium-pressure column 1 contains only sections of structured packings 2 having a density close or identical to the above values.

**[0022]** A stream of cooled and purified air 7 is sent to the medium-pressure column 1 and oxygen-enriched and nitrogen-enriched liquid streams 9, 11 are sent from the medium-pressure column to the low-pressure column after an expansion step.

**[0023]** Withdrawn from the bottom of the low-pressure column is a stream of gaseous oxygen 13 as product, with an oxygen yield of greater than 90% (preferably greater than 95%). The oxygen purity is greater than 90 mol% oxygen (preferably greater than 95 mol% oxygen or even 99 mol% oxygen).

**[0024]** At the top of the low-pressure column, gaseous nitrogen 15 is withdrawn with a nitrogen yield of greater than 85% (preferably greater than 90%). The purity of the nitrogen is greater than 99 mol% nitrogen (preferably greater than 99.99 mol% nitrogen or even 99.999 mol% nitrogen).

**[0025]** A stream of waste nitrogen 17 is withdrawn from an upper level of the low-pressure column.

**[0026]** The diameter (in metres) of the low-pressure column is less than:

$$\left[\frac{4Q_{air}}{\pi K\left(\frac{P}{P_0}\right)^{0.35}}\right]^{1/2}$$

where:

$Q_{air}$ is the sum of the flow rates in $Sm^3/h$ of the air feeding the column system;

K is the capacity per unit area of the column and equals at least 22153 $Nm^3/m^2$ (21000 $Sm^3/m^2$) (preferably at least 24263 $Nm^3/m^2$ (23000 $Sm^3/m^2$) or even at least 26372 $Nm^3/m^2$ (25000 $Sm^3/m^2$)); and

$P_0$ is equal to 2 bar abs.

**[0027]** To give a numerical example, it will be assumed that an installation has to treat an air throughput of 527450 $Nm^3/h$ (500000 $Sm^3/h$). According to the invention, if the low-pressure column is operated at a pressure below 2 bar abs, the cross section of the low-pressure column will therefore be less than:

$$(500000/21000)\,m^2$$

(preferably less than (500000/23000) $m^2$ or even less

than (500000/25000) $m^2$)
and the diameter less than:

$$\left[\frac{4(500000/21000}{\pi}\right]^{1/2}$$

i.e. 5.5m (preferably 5.26m, or even at most 5.05m).

**[0028]** If the low-pressure column is operated at 3 bar abs, the cross section of the low-pressure column will therefore be less than:

$$\left(\frac{500000}{21000}\right) m^2$$

(preferably less than (500000/23000) $m^2$ or even less than (5000000/25000) $m^2$)
and the diameter less than:

$$\left(\frac{4\left(\frac{500000}{21000}\right)}{\pi(3/2)^{0.35}}\right)^{1/2}$$

i.e. 5.13m (preferably 4.9 m, or even at most 4.7 m).

## Claims

1. Process for the separation of air into at least one constituent by cryogenic distillation of air in a system of columns (1, 3) comprising at least one column (1, 3)

   a) for producing at least gaseous oxygen as product, such that the oxygen yield is greater than 90% (preferably 95%) and the oxygen purity is greater than 90 mol% oxygen (preferably 95 mol% oxygen or even 99 mol% oxygen) and/or
   b) for producing at least nitrogen such that the nitrogen yield is greater than 85% (preferably greater than 90%) and the nitrogen purity is greater than 99 mol% nitrogen (preferably 99.99 mol% nitrogen or even 99.999 mol% nitrogen)

   **characterized in that:**

   • if the operating pressure of the largest-diameter column (3) is P<2 bar abs, then the diameter (in metres) of this column is less than:

$$\left(\frac{4Q_{air}}{\pi K}\right)^{1/2}$$

   if the operating pressure of the largest-diameter column is P > 2 bar abs, then the diameter (in metres) of this column is less than:

$$\left[\frac{4Q_{air}}{\pi K\left(\frac{P}{P_0}\right)^{0.35}}\right]^{1/2}$$

   where:

   $Q_{air}$ is the sum of the flow rates in $Nm^3/h$ of the air feeding the column system;
   K is the capacity per unit area of the column and equals at least 22153 $Nm/m^2$ (21000 $Sm^3/m^2$) (preferably at least 24263 $Nm^3/m^2$ (23000 $Sm^3/m^2$) or even at least 26372 (25000 $Nm^2/m^2$ $Sm^3/m^2$)); and
   $P_0$ is equal to 2 bar abs.

2. Process according to Claim 1, in which the largest-diameter column (3) of the column system contains at least one section (2) filled with a gas/liquid contactor allowing material exchange between the gaseous phase and the liquid phase such that its effectiveness is defined by a TUH (Transfer Unit Height) of less than 350 mm (preferably less than 300 mm).

3. Process according to Claim 2, in which the gas/liquid contactor is composed of cross-corrugated structured packings having a density of less than 500 $m^2/m^3$, preferably less than 400 $m^2/m^3$.

4. Process according to Claim 1, 2 or 3, in which the column system comprises at least one double column formed by a medium-pressure column (3) and a low-pressure column (1), the medium-pressure column being thermally coupled to the low-pressure column, and the low-pressure column constituting the largest-diameter column.

5. Process according to Claim 4, producing oxygen in which the ratio of the oxygen produced to the cross section of the low-pressure column is greater than or equal to 150 $t/d/m^2$.

6. Process according to Claim 4 or 5, in which the ratio of the flow rate of air sent to the column system to the cross section of the low-pressure column is not less than 22153 $Nm^3/h/m^2$ (21000 $sm^3/h/m^2$), pref-

erably not less than 24263 $Nm^3/h/m^2$ (23000 $Sm^3/h/m^2$) and even more preferably not less than 26372 $Nm^3/h/m^2$ (25000 $Sm^3/h/m^2$).

**7.** Process according to Claim 4, 5 or 6, in which the ratio of the gaseous air sent to the medium-pressure column (1) to the cross section of the low-pressure column is not less than 18988 $Nm^3/h/m^2$ (18000 $Sm^3/h/m^2$) (preferably not less than 21098 $Nm^3/h/m^2$ (20000 $Sm^3/h/m^2$) and even more preferably not less than 23208 $Nm^3/h/m^2$ (22000 $Sm^3/h/m^2$)).

**8.** Process according to Claim 7, in which at least one section (2) of the medium-pressure column (1) contains cross-corrugated structured packings with a density not exceeding 500 $m^2/m^3$, or even 400 $m^2/m^3$.

**Patentansprüche**

**1.** Verfahren zur Zerlegung von Luft in mindestens einen Bestandteil durch Tieftemperaturdestillation von Luft in einem System von Säulen (1, 3) mit mindestens einer Säule (1, 3)

a) zur Herstellung von mindestens gasförmigem Sauerstoff als Produkt mit einer Sauerstoffausbeute von mehr als 90% (vorzugsweise 95%) und einer Sauerstoffreinheit von mehr als 90 Mol-% Sauerstoff (vorzugsweise 95 Mol-% Sauerstoff oder sogar 99 Mol-% Sauerstoff) und/oder
b) zur Herstellung von mindestens Stickstoff mit einer Stickstoffausbeute von mehr als 85% (vorzugsweise mehr als 90%) und einer Stickstoffreinheit von mehr als 99 Mol-% Stickstoff (vorzugsweise 99,99 Mol-% Stickstoff oder sogar 99,999 Mol-% Stickstoff),

**dadurch gekennzeichnet, daß:**

■ dann, wenn der Betriebsdruck der den größten Durchmesser aufweisenden Säule (3) P < 2 bar abs. beträgt, der Durchmesser (in Meter) dieser Säule kleiner als:

$$\left(\frac{4Q_{Luft}}{\pi K}\right)^{1/2}$$

ist,
dann, wenn der Betriebsdruck der den größten Durchmesser aufweisenden Säule P > 2 bar abs. beträgt, der Durchmesser (in Meter) dieser Säule kleiner als:

$$\left[\frac{4Q_{Luft}}{\pi K\left(\frac{P}{P_0}\right)^{0,35}}\right]^{1/2}$$

ist,
wobei:

$Q_{Luft}$ die Summe der Strömungsraten der das Säulensystem speisenden Luft in $Nm^3/h$ ist;
K die Kapazität der Säule pro Flächeneinheit ist und gleich mindestens 22153 $Nm^3/m^2$ (21.000 $Sm^3/m^2$) (vorzugsweise mindestens 24263 $Nm^3/m^2$ (23.000 $Sm^3/m^2$) oder sogar mindestens 26372 $Nm^3/m^2$ (25.000 $Sm^3/m^2$) ist und
$P_0$ gleich 2 bar abs. ist.

**2.** Verfahren nach Anspruch 1, bei dem die den größten Durchmesser aufweisende Säule (3) des Säulensystems mindestens einen Abschnitt (2) enthält, der mit einem Stoffaustausch zwischen der Gasphase und der Flüssigphase erlaubenden Gas/Flüssigkeit-Kontaktor gefüllt ist, so daß seine Effektivität durch eine TUH (Höhe einer Übertragungseinheit) von weniger als 350 mm (vorzugsweise weniger als 300 mm) definiert ist.

**3.** Verfahren nach Anspruch 2, bei dem der Gas/Flüssigkeit-Kontaktor aus gekreuzt-gewellten geordneten Packungen mit einer Dichte von weniger als 500 $m^2/m^3$, vorzugsweise weniger als 400 $m^2/m^3$, besteht.

**4.** Verfahren nach Anspruch 1, 2 oder 3, bei dem das Säulensystem mindestens eine aus einer Mitteldrucksäule (3) und einer Niederdrucksäule (1) gebildete Doppelsäule umfaßt, wobei die Mitteldrucksäule thermisch mit der Niederdrucksäule gekoppelt ist und die Niederdrucksäule die Säule mit dem größten Durchmesser darstellt.

**5.** Verfahren nach Anspruch 4, das Sauerstoff produziert, bei dem das Verhältnis des produzierten Sauerstoffs zum Querschnitt der Niederdrucksäule größer gleich 150 $t/d/m^2$ ist.

**6.** Verfahren nach Anspruch 4 oder 5, bei dem das Verhältnis von Strömungsrate der dem Säulensystem zugeführten Luft zu Querschnitt der Niederdrucksäule mindestens 22153 $Nm^3/h/m^2$ (21. 000

$Sm^3/h/m^2$), vorzugsweise mindestens 24263 $Nm^3/h/m^2$ (23.000 $Sm^3/h/m^2$) und noch weiter bevorzugt mindestens 26372 $Nm^3/h/m^2$ (25.000 $Sm^3/h/m^2$) beträgt.

**7.** Verfahren nach Anspruch 4, 5 oder 6, bei dem das Verhältnis von der Mitteldrucksäule (1) zugeführter gasförmiger Luft zu Querschnitt der Niederdrucksäule mindestens 18988 $Nm^3/h/m^2$ (18.000 $Sm^3/h/m^2$) (vorzugsweise mindestens 21098 $Nm^3/h/m^2$ (20.000 $Sm^3/h/m^2$) und noch weiter bevorzugt mindestens 23208 $Nm^3/h/m^2$ (22.000 $Sm^3/h/m^2$)) beträgt.

**8.** Verfahren nach Anspruch 7, bei dem mindestens ein Abschnitt (2) der Mitteldrucksäule (1) gekreuztgewellte geordnete Packungen mit einer Dichte von höchstens 500 $m^2/m^3$ oder sogar 400 $m^2/m^3$ enthält.

**Revendications**

**1.** Procédé de séparation d'air en au moins un constituant par distillation cryogénique de l'air dans un système de colonnes (1, 3) comprenant au moins une colonne (1, 3)

a) pour produire au moins de l'oxygène gazeux comme produit tel que le rendement en oxygène est supérieur à 90 % (préferablement 95 %) et la pureté d'oxygène est supérieure à 90 % mol. d'oxygène (préférablement 95 % mol. d'oxygène, voire 99 % mol. d'oxygène) et/ou
b) pour produire au moins de l'azote tel que le rendement en azote est supérieur à 85 % (préférablement supérieur à 90 %) et la pureté de l'azote est supérieure à 99 % mol. d'azote (préférablement à 99.99 % mol. d'azote, voire à 99,999 % mol. d'azote)

**caractérisé en ce que :**

• si la pression de fonctionnement de la plus large des colonnes (3) est P < 2 bars abs, alors le diamètre (en mètres) de cette colonne est inférieur à :

$$\left(\frac{4Q_{air}}{\pi K}\right)^{1/2}$$

si la pression de fonctionnement de la plus large des colonnes est P > 2 bars abs, alors le diamètre (en mètres) de cette colonne est inférieur à :

$$\left[\frac{4Q_{air}}{\pi K\left(\frac{P}{P_0}\right)^{0,35}}\right]^{1/2}$$

où :

$Q_{air}$ est la somme des débits d'air alimentant le système de colonnes, en $Nm^3/h$ ;
K est la capacité surfacique de la colonne et vaut au moins 22 153 $Nm^3/m^2$ (21 000 $Nm^3/m^2$) (préférablement au moins 24 263 $Nm^3/m^2$ (23 000 $Nm^3/m^2$), voire au moins 26 372 $Nm^3/m^2$ (25 000 $Nm^3/m^2$); et
$P_0$ est égale à 2 bars abs.

**2.** Procédé selon la revendication 1 dans lequel la colonne de plus grand diamètre (3) du système de colonnes contient au moins un tronçon (2) rempli avec un contacteur gaz/liquide permettant l'échange de matière entre la phase gazeuse et la phase liquide tel que son efficacité sera définie par une hauteur par unité de transfert (HUT) inférieure à 350 mm (préférablement à 300 mm).

**3.** Procédé selon la revendication 2 dans lequel le contacteur gaz/liquide est composé de garnissages structurés ondulés-croisés ayant une densité de moins de 500 $m^2/m^3$, de préférence de moins de 400 $m^2/m^3$.

**4.** Procédé selon la revendication 1, 2 ou 3 dans lequel le système de colonnes comprend au moins une double colonne constituée par une colonne moyenne pression (3) et une colonne basse pression (1), la colonne moyenne pression étant thermiquement reliée à la colonne basse pression et la colonne basse pression constituant la colonne la plus large.

**5.** Procédé selon la revendication 4 produisant de l'oxygène dans lequel le rapport entre l'oxygène produit et la section de la colonne basse pression est supérieur ou égal à 150 $t/j/m^2$.

**6.** Procédé selon la revendication 4 ou 5 dans lequel le rapport entre le débit d'air envoyé au système de colonnes et la section de la colonne basse pression est supérieur ou égal à 22 153 $Nm^3/m^2$ (21 000 $Nm^3/h/m^2$), préférablement à 24 263 $Nm^3/m^2$ (23 000 $Nm^3/h/m^2$) et encore plus préférablement à 26 372 $Nm^3/m^2$ (25 000 $Nm^3/h/m^2$).

**7.** Procédé selon la revendication 4, 5 ou 6 dans lequel le rapport entre l'air gazeux envoyé à la colonne

moyenne pression (1) et la section de la colonne moyenne pression est supérieur ou égal à 18 988 $Nm^3/h/m^2$ (18 000 $Nm^3/h/m^2$) (préférablement à 21 098 $Nm^3/h/m^2$ (20 000 $Nm^3/h/m^2$), encore plus préférablement à 23 208 $Nm^3/h/m^2$ (22 000 $Nm^3/h/m^2$).

**8.** Procédé selon la revendication 7 dans lequel au moins un tronçon (2) de la colonne moyenne pression (1) contient des garnissages structurés ondulés-croisés de densité inférieure à 500 $m^2/m^3$, voire à 400 $m^2/m^3$.

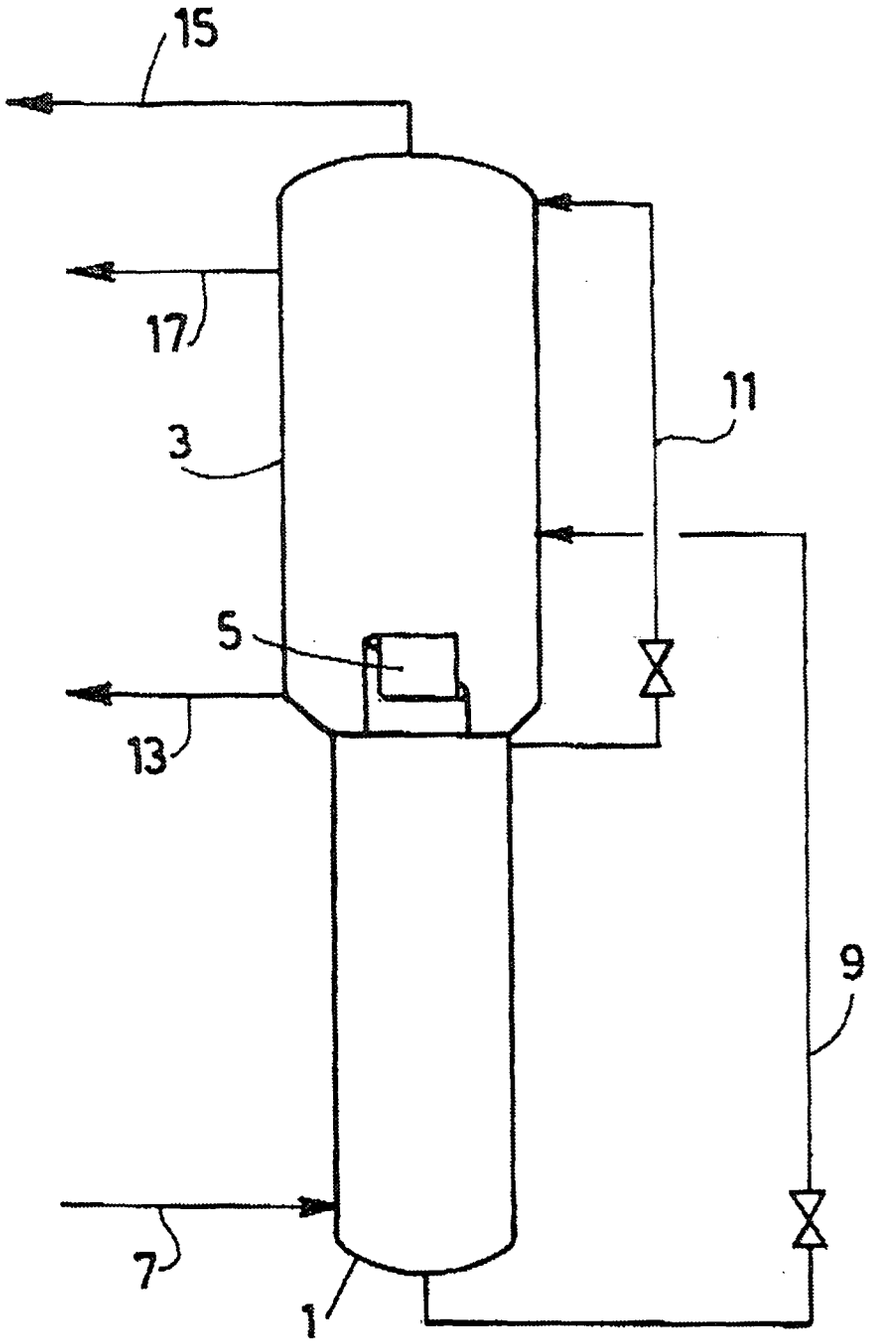



FIG.1

## REFERENCES CITED IN THE DESCRIPTION

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- EP 0491591 A **[0002]**
- US 5653126 A **[0003]**