

12

EUROPEAN PATENT APPLICATION

21 Application number: **89401358.0**

51 Int. Cl.⁴: **F 25 J 3/04**

22 Date of filing: **17.05.89**

30 Priority: **19.05.88 JP 122681/88**

43 Date of publication of application:
23.11.89 Bulletin 89/47

84 Designated Contracting States: **DE ES FR IT NL**

71 Applicant: **L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCÉDES GEORGES CLAUDE**
75, Quai d'Orsay
F-75321 Paris Cédex 07 (FR)

72 Inventor: **Takagi, Harumitsu L'Air Liquide**
Service des Relations Ind. 75, Quai d'Orsay
F-75321 Paris Cedex 07 (FR)

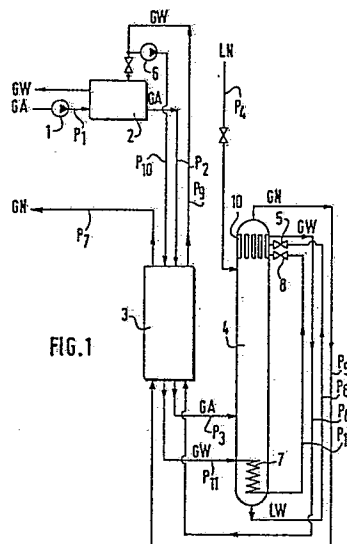
Nagamura, Takashi L'Air Liquide
Service des Relations Ind. 75, Quai d'Orsay
F-75321 Paris Cedex 07 (FR)

Yamamoto, Takao L'Air Liquide
Service des Relations Ind. 75, Quai d'Orsay
F-75321 Paris Cedex 07 (FR)

74 Representative: **Vesin, Jacques et al**
L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCÉDES GEORGES CLAUDE
75, quai d'Orsay
F-75321 Paris Cédex 07 (FR)

54 Method of producing nitrogen gas:

57 Method of producing high purity nitrogen gas from compressed air by utilizing a single fractionating tower (4). According to the invention the oxygen rich gas taken out of the condenser (10) is used as a coolant in the main heat exchanger (3) and thereafter compressed, cooled and fed to the bottom of the fractionating tower (4) for heating the oxygen rich liquid in the bottom of the fractionating tower (4). Moreover, the oxygen rich liquid in the bottom of the fractionating tower (4) is used as a cold source for producing the recirculation liquid.



Description

Method of Producing Nitrogen Gas

DETAILED DESCRIPTION OF THE INVENTION

(Industrial Field of Application)

The present invention relates to a method of producing nitrogen gas from compressed air by utilizing a single fractionating tower.

(Prior Art)

As a nitrogen gas producing method of this type utilizing a single fractionating tower, a method is known wherein nitrogen gas taken out of a top position of the fractionating tower is used as a coolant in a main heat exchanger, and the nitrogen gas heated to room temperature is taken out as a low pressure nitrogen gas product having approximately the same pressure as the raw material air (Patent Publication No. 54-39830, for example).

It is possible with this known method to fractionate and separate nitrogen gas from the raw material air by partial condensation through contact between the raw material air fed to a lower position of the fractionating tower and recirculation liquid descending from the top of the fractionating tower.

In the known method, oxygen-rich liquid having a large nitrogen content is collected in a sump at the bottom of the fractionating tower. The oxygen-rich liquid in the sump is taken out as it is and led to a condenser in the top of the fractionating tower to be used as a coolant therein. This liquid is vaporized into oxygen-rich air through heat exchange in the condenser, which air is thereafter used as a coolant in the main heat exchanger and is then released as exhaust gas.

(Problem to be Solved by Invention)

In practice, the oxygen-rich gas is released as exhaust gas as noted above without its effective use being attained to the full extent although it is possible to make effective use of the oxygen-rich gas.

The present invention has been made having regard to the above state of the art, and its object is to provide a method of producing nitrogen gas with improved yield and with low manufacturing cost per unit amount, which is achieved by making effective use of the oxygen-rich gas which has been disposed of as exhaust gas as noted above.

(Means for Solving Problem)

A method of producing nitrogen gas according to the present invention comprises the steps of removing impurities such as moisture and carbon dioxide from a raw material consisting of compressed air, feeding the impurity-free raw material, after cooling the same to a temperature close to a liquefying point through a main heat exchanger, to a lower position of a fractionating tower for fractionating the raw material, withdrawing nitrogen gas from a top position of the fractionating tower and leading the nitrogen gas to the main heat exchanger for use

as a coolant, and heating the nitrogen gas to room temperature by heat exchange therein to obtain nitrogen gas product,

characterized in that oxygen-rich liquid is taken out of a bottom position of the fractionating tower and, while being expanded, is fed to a condenser disposed in a top position of the fractionating tower for use as a coolant therein, said liquid being vaporized in said condenser into oxygen-rich gas, said gas is taken out of said condenser and led to said main heat exchanger for use as a coolant therein, said gas being heated to room temperature by said main heat exchanger and taken out therefrom, at least part thereof being recirculated by expanding and returning it to said main heat exchanger, cooling it through heat exchange in said main heat exchanger, and leading it to the bottom of said fractionating tower, and expanding and leading it to said condenser, and an additional coolant is separately replenished in any one of the cooling processes.

(Function)

In producing nitrogen gas by the method according to the present invention, the cold energy of the oxygen-rich gas taken out of the condenser is first used as a cold source in the main heat exchanger, whereby the oxygen-rich gas is heated to room temperature. At least part of this oxygen-rich gas is compressed and returned to the main heat exchanger where it is cooled, and is thereafter fed to the bottom of the fractionating tower (to a reboiler disposed therein, for example). Then a heat exchange takes place in the bottom of the fractionating tower between the compressed oxygen-rich gas and the oxygen-rich liquid. The oxygen-rich liquid is thereby heated and the compressed oxygen-rich gas is liquefied. The gas evaporated as the oxygen-rich liquid is heated ascends in counter current contact with a recirculation liquid (liquid nitrogen, for example) descending through the fractionating tower. Fractionation is thereby effected with oxygen becoming liquefied and descending, and nitrogen-rich gas ascending. On the other hand, the oxygen-rich liquid collected in the bottom of the fractionating tower is taken out of the bottom, expanded and fed to the condenser to act as a coolant. In other words, the oxygen-rich liquid is fed to the top of the fractionating tower to produce the recirculation liquid necessary for separating the nitrogen content from the raw material air by liquefying the nitrogen gas ascending through the fractionating tower.

(Effect of the Invention)

In the method according to the present invention, the oxygen-rich gas taken out of the condenser is used as a coolant in the main heat exchanger, and thereafter compressed, cooled and fed to the bottom of the fractionating tower for heating the oxygen-rich liquid in the bottom of the fractionating tower. Moreover, the oxygen-rich liquid in the

bottom of the fractionating tower is used as a cold source for producing the recirculation liquid. This feature realizes improved yield of nitrogen gas and low manufacturing cost per unit amount compared with the known method.

(Embodiments)

The present invention will be described further with reference to the drawings illustrating embodiments thereof.

As shown in Fig. 1, raw material air GA stripped of dust by an air filter (not shown) is compressed by a compressor 1 to a nitrogen gas product pressure and pressure necessary for operating an air separator (9.5kg/cm²G, for example). The compressed raw material air GA is fed through a piping P1 to a drying and carbon removing unit 2. In the drying and carbon removing unit 2, the compressed raw material air GA is fed to one of two molecular sieve towers where moisture and carbon dioxide are removed from the raw material air GA through adsorption. Meanwhile, oxygen-rich gas GW having passed through a main heat exchanger 3 to be described later is fed to the other molecular sieve tower to regenerate this tower.

The raw material air GA stripped of moisture and carbon dioxide at the drying and carbon removing unit 2 is fed through a piping P2 to the main heat exchanger 3 to be cooled to a temperature close to the liquefying point. Thereafter the air GA is fed through a piping P3 to a lower position of a fractionating tower 4. This fractionating tower 4 receives liquid nitrogen LN, which is one example of cold source, delivered through a piping P4 to an upper position thereof. In the fractionating tower 4, the raw material air GA ascending from below and the liquid nitrogen (recirculation liquid) descending from above contact each other in counter current, whereby oxygen in the raw material air GA is liquefied to fractionate and separate nitrogen gas GN.

The nitrogen gas GN taken out of the top of the fractionating tower 4 is fed through a piping P5 to the main heat exchanger 3 so that the cold energy of nitrogen gas GN is used as a coolant in the main heat exchanger 3 and that the nitrogen gas GN is heated to room temperature. The nitrogen gas GN at room temperature taken out of the main heat exchanger 3 through a piping P7 is supplied as a nitrogen gas product having an appropriate pressure (9.0kg/cm²G, for example).

Oxygen-rich liquid LW is collected in the bottom of the fractionating tower 4. This liquid LW is taken out of the bottom and is led through a piping P6 having an expansion valve 5 to a condenser 10 disposed in the top position of the fractionating tower 4. The liquid LW is expanded by the expansion valve 5 to an appropriate pressure (3.5kg/cm²G, for example) and is led into the condenser 10 to be used as a coolant therein. In the condenser 10 the liquid LW is vaporized into oxygen-rich gas GW.

The oxygen-rich gas GW, after being taken out of the condenser 10, is led through a piping P8 to the main heat exchanger 3 to be used as a coolant therein. This gas GW is heated to room temperature

at the main heat exchanger 3, and is thereafter led through a piping P9 to the drying and carbon removing unit 2 and a compressor 6. Part of the gas GW is released as exhaust gas GW after being used for regenerating the drying and carbon removing unit 2 as described hereinbefore. The remainder is compressed by the compressor 6 (to a pressure of 3.5kg/cm²G to 10.0kg/cm²G, for example), and returned through a piping P10 to the main heat exchanger 3. The gas GW is cooled through heat exchange in the main heat exchanger 3. The cooled gas GW is led through a piping P11 to a reboiler 7 disposed in the bottom of the fractionating tower 4 to give off heat. Then the gas GW is cooled therein and expanded to a pressure of 3.5kg/cm²G, for example, through a piping P12 having an expansion valve 8 at an intermediate position thereof. Thereafter expanded gas GW is led to the compressor 10 disposed in the top position of the fractionating tower 4 to join the oxygen-rich gas GW.

Thus, in producing nitrogen gas, the oxygen-rich gas GW taken out of the condenser 10 is used as a coolant in the main heat exchanger 3. After being taken out of the main heat exchanger 3, the gas GW is compressed, cooled and fed to the reboiler 7 for heating the oxygen-rich liquid LW collected in the bottom of the fractionating tower 4. Moreover, the oxygen-rich liquid LW which has been liquefied in the reboiler 7 is used as a cold source in the condenser 10 for producing the recirculation liquid. Thus, effective use is made of the oxygen-rich gas GW, whereby the yield of nitrogen gas is improved to about 88% compared with less than 50% of nitrogen gas heretofore obtained from nitrogen contained in the air.

In the described embodiment, part of the oxygen-rich gas taken out of the condenser 10 and heated to room temperature by the main heat exchanger 3 is utilized for regenerating the drying and carbon removing unit 2. This feature promotes the effective use of the oxygen-rich gas.

As shown in Fig. 2, the oxygen-rich gas GW taken out of the compressor 10 may be taken out at an intermediate position of the main heat exchanger 3 through a piping P13. Part of the gas GW is adiabatically expanded by an expansion turbine 11 and returned through a piping P14 to the main heat exchanger 3 to be used as a coolant in the main heat exchanger 3. The gas GW used as a coolant may be taken out of the main heat exchanger 3 and led through a piping P15 to the drying and carbon removing unit 2 for regenerating this unit 2. In this case, the gas GW led through the piping P9 need not be used as the regenerating gas. This method provides an even more effective use of the oxygen-enriched gas GW.

Further, as shown in Figs. 3 and 4, the oxygen-rich gas GW returned to the main heat exchanger 3, as in the above embodiment, may be led through a piping 16 directly to the bottom of the fractionating tower 4 after being cooled by the main heat exchanger 3 to a temperature adjacent the liquefying point.

Although the claims include reference numbers for convenience of comparison with the drawings, the present invention is not limited to the construc-

tion illustrated in the accompanying drawings.

4. BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a view of a piping system illustrating execution of a nitrogen gas producing method according to the present invention, and

Figs. 2-4 are views of modified piping systems, respectively.

2 ... drying and carbon removing unit, 3 ... main heat exchanger, 4 ... fractionating tower, 7 ... reboiler, 10 ... condenser, 11 ... expansion turbine.

Claims

1. A method of producing nitrogen gas comprising the steps of removing impurities such as moisture and carbon dioxide from a raw material consisting of compressed air, feeding the impurity-free raw material, after cooling the same to a temperature close to a liquefying point through a main heat exchanger (3), to a lower position of a fractionating tower (4) for fractionating the raw material, withdrawing nitrogen gas from a top position of the fractionating tower (4) and leading the nitrogen gas to the main heat exchanger (3) for use as a coolant, and heating the nitrogen gas to room temperature by heat exchange therein to obtain nitrogen gas product, characterized in that oxygen-rich liquid is taken out of a bottom position of the fractionating tower (4) and, while being expanded, is fed to a condenser (10) disposed in a top position of the fractionating tower (4) for use as a coolant therein, said liquid being vaporized in said condenser (10) into oxygen-rich gas, said gas is taken out of said condenser (10) and led to said main heat exchanger (3) for use as a coolant therein, said gas being heated to room temperature by said main heat exchanger (3) and taken out therefrom, at least part thereof being recirculated by expanding and returning it to said main heat exchanger (4), cooling it through heat exchange in said main heat exchanger (4), and leading it to the bottom of said fractionating tower (4), and expanding and leading it to said condenser (10), and an additional coolant is separately replenished in any one of the cooling processes.

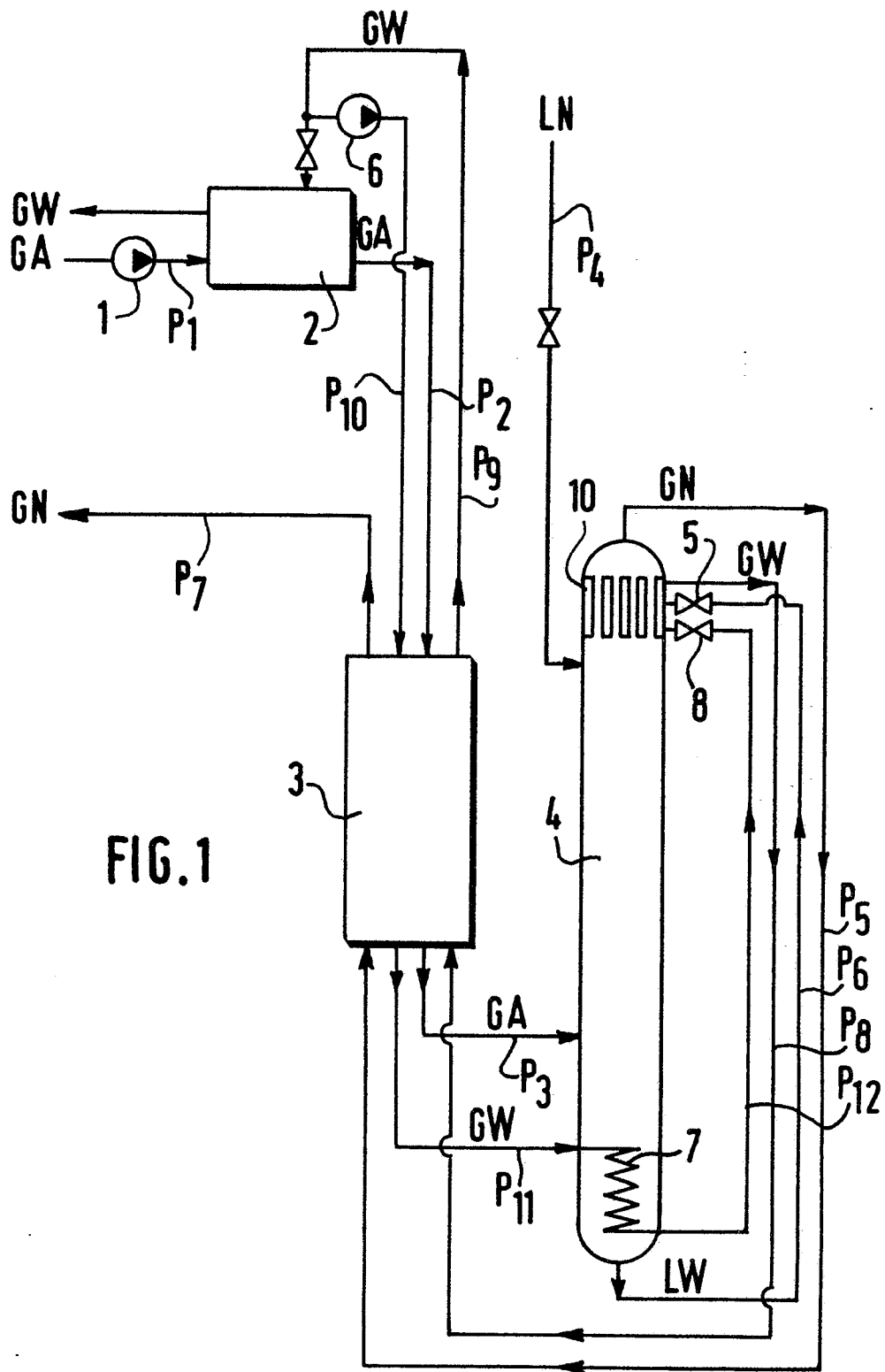
2. A method of producing nitrogen gas as defined in Claim 1, wherein a drying and carbon removing unit (2) is used for removing moisture and carbon dioxide from the raw material, and part of the oxygen-rich gas taken out of said condenser (10) and heated to room temperature through said main heat exchanger (3) is used for regenerating said drying and carbon removing unit (2).

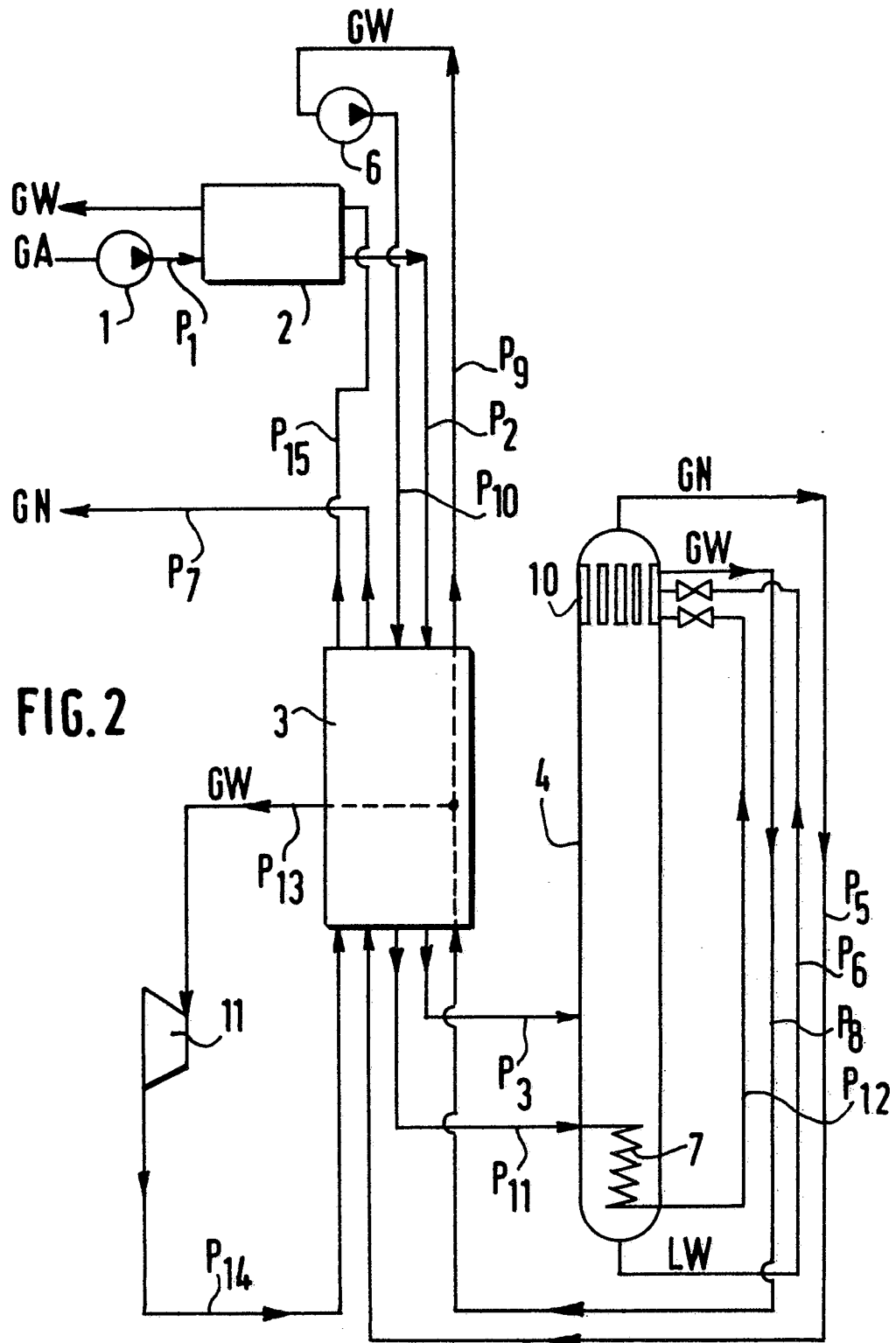
3. A method of producing nitrogen gas as defined in Claim 1 or 2, wherein the oxygen-rich

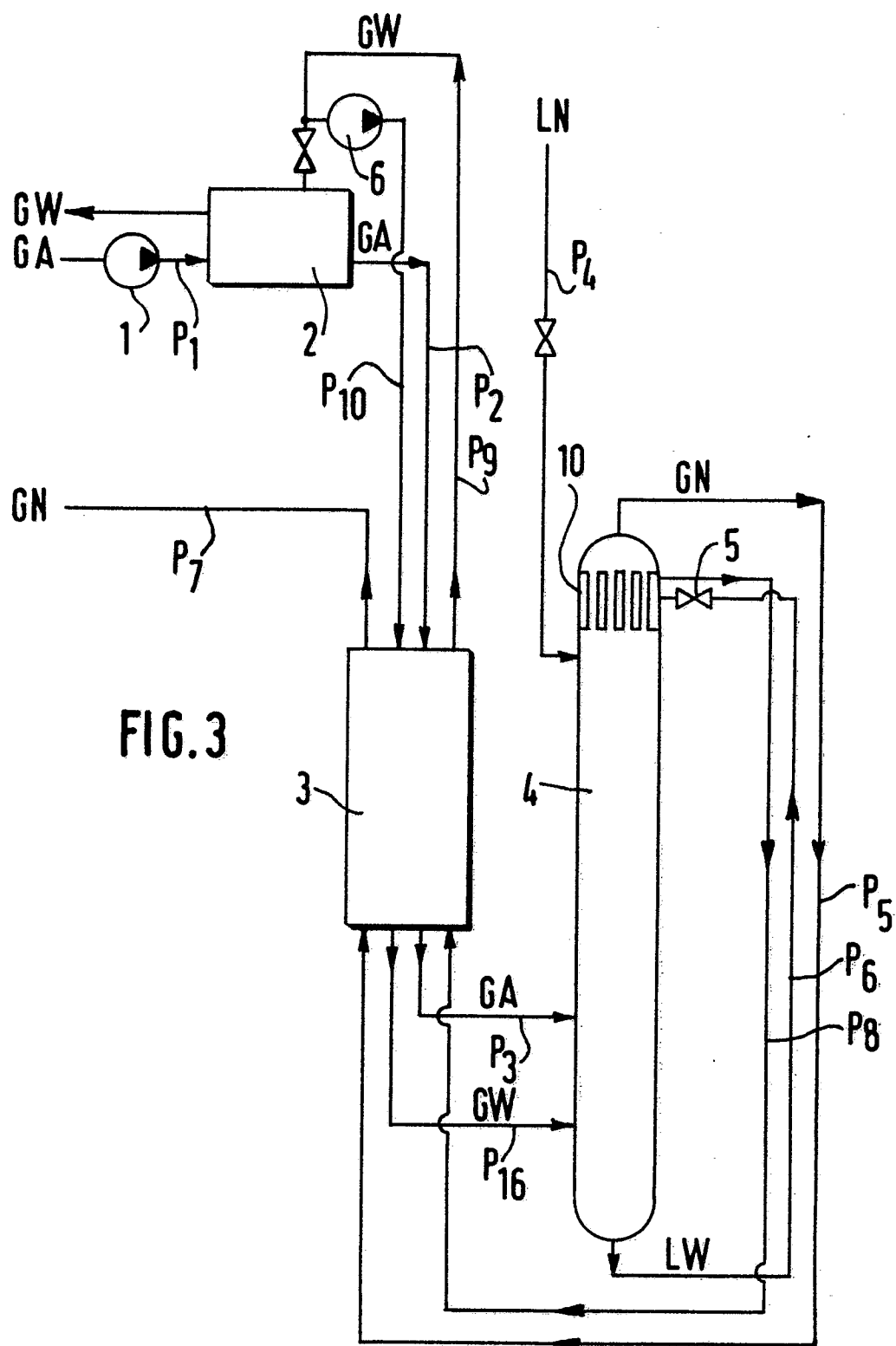
gas returned to said main heat exchanger (3) and cooled through heat exchange in said main heat exchanger (3) is fed to a reboiler (7) disposed in the bottom of said fractionating tower (4) and, after passing through said reboiler (7), expanded and led to said condenser (10).

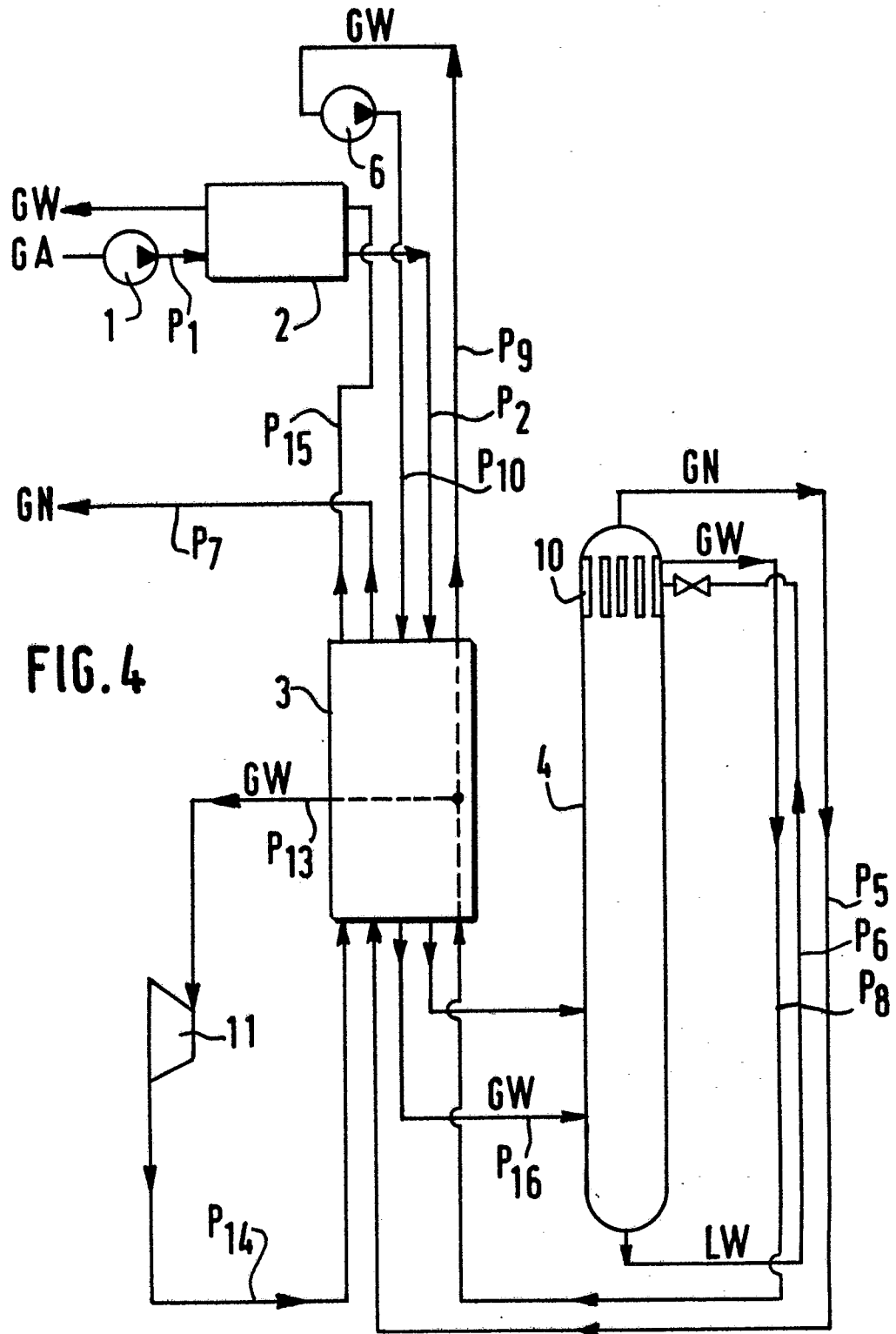
4. A method of producing nitrogen gas as defined in Claim 2, wherein the oxygen-rich gas taken out of said condenser (10) is taken out of an intermediate position of said main heat exchanger (3), expanded by an expansion turbine (11), used as a coolant in said main heat exchanger (3), and thereafter used for regenerating said drying and carbon removing unit (2).

5. A method of producing nitrogen gas as defined in Claim 1, 2 or 4, wherein the oxygen-rich gas returned to said main heat exchanger (3) is cooled by the main heat exchanger (3) to a temperature adjacent the liquefying point, and thereafter directly fed to the bottom of said fractionating tower (4).











DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
Y	EP-A-0 241 817 (LINDE) * Abstract; column 3, line 26 - column 4, line 38; figure *	1	F 25 J 3/04
A	---	2	
Y	FR-A-2 225 705 (CRYOPLANTS) * Page 1, lines 1-7; page 2, lines 1-14; page 4, lines 12-19; page 5, line 15 - page 6, line 7; figure * -----	1	
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)
			F 25 J
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 28-08-1989	Examiner SIEM T.D.
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			