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Description

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

Japanese Patent Publication 54-39830 describes 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, to a lower position of a fractionating tower and leading the nitrogen gas to the main heat exchanger for use as a coolant,

heating the nitrogen gas to room temperature by heat exchange therein to obtain nitrogen gas product,

taking oxygen-rich liquid out of a bottom position of the fractionating tower and, while being expanded, feeding it 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,

taking said gas out of said condenser and leading it to said main heat exchanger for use as a coolant therein, said gas being heated to room temperature by said main heat exchanger.

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.

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.

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, heating the nitrogen gas to room temperature by heat exchange therein to obtain nitrogen gas product,

taking oxygen-rich liquid out of a bottom position of the fractionating tower and, while being expanded, feeding it 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,

taking said gas out of said condenser and leading it to said main heat exchanger for use as a coolant therein, said gas being heated to room temperature by said main heat exchanger

characterized in that at least part of said oxygen-rich gas is recirculated after compression to said main heat exchanger, cooled through heat exchange in said main heat exchanger, and led to the condenser either via a reboiler provided at the bottom of the fractionating column or by introducing it directly into the bottom of the fractionating column and

a coolant means is associated to any one of the cooling processes.

According to one embodiment, the coolant means is an additional coolant, as already shown in FR-A-2.225.705.

According to another embodiment, said coolant means is obtained by taking oxygen-rich gas out of an intermediate position of the main heat exchanger, expanded by an expansion turbine, used as a coolant in said heat exchanger and thereafter used for regenerating said drying and carbon removing unit.

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.

In one embodiment of 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.

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

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.

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 9.3 bar, 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. There after 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 (8.82 bar, 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 3.43 bar, 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 3.43 to 9.8 bar, 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 3.43 bar, for example, through a piping P12 having an expansion valve 8 at an intermediate position thereof. Thereafter expanded gas GW is led to the condenser 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.

ore 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 condenser 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.

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 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 exchanger (3) for use as a coolant, and
 heating the nitrogen gas to room temperature by heat exchange therein to obtain nitrogen gas product,
 taking oxygen-rich liquid out of a bottom position of the fractionating tower (4) and, while being expanded, feeding it 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,
 taking said gas out of said condenser (10) and leading it 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),
 characterized in that at least a part of said oxygen rich gas is recirculated after compression (6) to said main heat exchanger (3), cooled through heat exchange in said main heat exchanger (3), and led to the condenser (10) either via a reboiler (7) provided at the bottom of the fractionating column, or by introducing it directly into the bottom of the fractionating column and
 a coolant means is associated to 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 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).

4. A method of producing nitrogen gas as defined in Claim 1, 2 or 3, 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).

Patentansprüche

1. Stickstoffgasherstellungsverfahren, das folgende Schritte umfasst :
 Entfernen von Verunreinigungen, wie Feuchtigkeit und Kohlendioxid, aus einem aus Druckluft bestehenden Rohmaterial,
 Zuführung des von Verunreinigungen freien Rohmaterials, nachdem dieses auf eine nahe am Verflüssigungspunkt liegende Temperatur über einen Hauptwärmetauscher (3) abgekühlt wurde, zu einer unteren Position eines Fraktionierturmes (4) zwecks Fraktionierung des Rohmaterials,
 Abziehen von Stickstoffgas aus einer oberen Position des Fraktionierturmes (4) und Führen des Stickstoffgases zum Hauptwärmetauscher (3), zwecks Verwendung als Kühlmittel, und
 Erhitzen des Stickstoffgases auf Raumtemperatur mittels in diesem erfolgenden Wärmeaustausch zwecks Erzielung des Stickstoffgaserzeugnisses, Entnahme einer sauerstoffreichen Flüssigkeit aus einer untersten

Position des Fraktionierturmes (4) und Zuführung desselben zu einem Kondensator (10), der sich in einer oberen Position des Fraktionierturmes (4) befindet, um dort als Kühlmittel verwendet zu werden, wobei die Flüssigkeit im Kondensator (10) zu einem sauerstoffreichen Gas verdampft wird,

Entnahme des Gases aus dem Kondensator (10) und Zuführen desselben zum Hauptwärmetauscher (3), um es in diesem als Kühlmittel zu verwenden, wobei das Gas durch den Hauptwärmetauscher (3) auf Raumtemperatur erwärmt wird, dadurch **gekennzeichnet**, dass mindestens ein Teil des sauerstoffreichen Gases nach Verdichtung (6) zum Hauptwärmetauscher (3) rezirkuliert wird, durch Wärmeaustausch im Hauptwärmetauscher (3) gekühlt und dem Kondensator (10) entweder über ein am untersten Teil der Fraktioniersäule vorgesehenes Destillationsgefäß (7) zugeführt wird, oder er unmittelbar in den untersten Teil der Fraktioniersäule eingeführt wird, und ein Kühlmittel jedem der Kühlvorgänge zugeordnet ist.

2. Stickstoffgasherstellungsverfahren nach Anspruch 1, dadurch **gekennzeichnet**, dass eine Trocken- und Kohlenstoffentnahmeeinheit (2) verwendet wird, um Feuchtigkeit und Kohlendioxid aus dem Rohmaterial zu entnehmen, und dass ein Teil des sauerstoffreichen Gases, das aus dem Kondensator (10) entnommen und über den Hauptwärmetauscher (3) auf Raumtemperatur erwärmt wurde, dazu verwendet wird, um die Trocken- und Kohlenstoffentnahmeeinheit (2) zu regenerieren.

3. Stickstoffgasherstellungsverfahren nach Anspruch 2, dadurch **gekennzeichnet**, dass das aus dem Kondensator (10) entnommene sauerstoffreiche Gas aus einer Zwischenposition des Hauptwärmetauschers (3) entnommen wird, durch eine Expansionsturbine (11) entspannt wird, als Kühlmittel im Hauptwärmetauscher (3) verwendet wird und anschließend zur Regenerierung der Trocken- und Kohlenstoffentnahmeeinheit (2) verwendet wird.

4. Stickstoffgasherstellungsverfahren nach Anspruch 1, 2 oder 3, dadurch **gekennzeichnet**, dass das zum Hauptwärmetauscher (3) zurückgeführte, sauerstoffreiche Gas durch den Hauptwärmetauscher (3) auf eine nahe am Verflüssigungspunkt liegende Temperatur gekühlt wird, und anschließend unmittelbar dem untersten Teil des Fraktionierturmes (4) zugeführt wird.

Revendications

1. Procédé de production d'azote gazeux comprenant les étapes suivantes :
on enlève les impuretés notamment l'humidité et

le gaz carbonique d'un matériau brut constitué par de l'air comprimé,

on alimente le matériau brut libre d'impuretés, après refroidissement de ce dernier à une température proche d'un point de liquéfaction à travers un échangeur de chaleur principal (3), en position basse d'une tour de fractionnement (4) afin de fractionner le matériau brut,

on retire de l'azote gazeux en tête de la tour de fractionnement (4) et on envoie l'azote gazeux à l'échangeur principal pour utilisation en tant qu'agent de refroidissement, et

on chauffe l'azote gazeux à température ambiante par échange de chaleur de façon à obtenir un produit constitué d'azote gazeux,

on soutire un liquide enrichi en oxygène en cuve de la tour de fractionnement (4) et tout en le dilatant, on l'alimente à un condenseur (10) disposé en tête de la tour de fractionnement (4) pour utilisation en tant qu'agent de refroidissement, ledit liquide étant vaporisé dans ledit condenseur (10) pour former un gaz enrichi en oxygène,

on retire le gaz dudit condenseur (10) et on l'envoie audit échangeur de chaleur principal (3) pour utilisation en tant qu'agent de refroidissement, ledit échangeur de chaleur principal (3), chauffant ledit gaz à température ambiante, caractérisé en ce qu'au moins une partie dudit gaz enrichi en oxygène est recirculée après compression (6) vers ledit échangeur de chaleur principal (3), refroidie par échange de chaleur dans ledit échangeur de chaleur principal (3), et envoyée au condenseur (10) soit par l'entremise d'une rebouilloire (7) prévue en cuve de la colonne de fractionnement ou en l'introduisant directement en cuve de la colonne de fractionnement et

un moyen refroidisseur est associé à l'un quelconque des étapes de refroidissement.

2. Procédé de production d'azote gazeux selon la revendication 1, caractérisé en ce que l'on utilise un ensemble (2) de séchage et d'enlèvement de carbone pour enlever l'humidité et le gaz carbonique du matériau brut, et une partie du gaz enrichi en oxygène que l'on retire dudit condenseur (10) et que l'on chauffe à température ambiante en se servant de l'échangeur de chaleur principal (3) est utilisé pour régénérer ledit ensemble de séchage et d'enlèvement de carbone (2).

3. Procédé de production d'azote gazeux selon la revendication 2, caractérisé en ce que l'on soutire le gaz enrichi en oxygène retiré dudit condenseur (10) d'une position intermédiaire dudit échangeur de chaleur principal, on le dilate dans une turbine de dilatation (11), on l'utilise comme réfrigérant dans ledit échangeur de chaleur principal et on l'utilise ensuite pour régénérer ledit ensemble de séchage et d'enlèvement de carbone (2).

4. Méthode de production d'azote gazeux selon les revendications 1, 2 ou 3, caractérisé en ce que le gaz enrichi en oxygène que l'on retourne audit échangeur de chaleur principal (3) est refroidi dans l'échangeur de chaleur principal (3) à une température voisine du point de liquéfaction et qu'on l'envoie ensuite en cuve de la tour de fractionnement (4).

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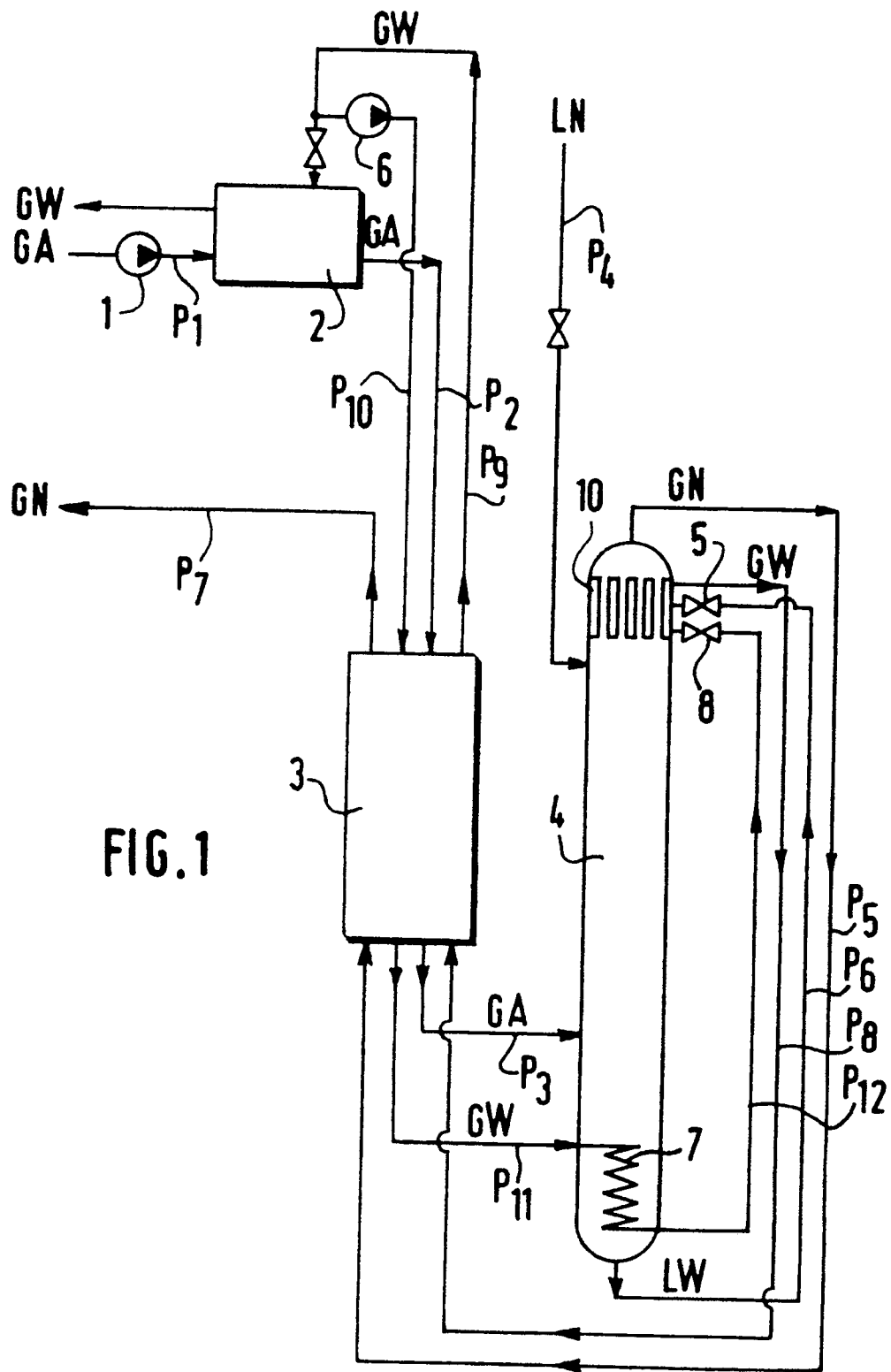


FIG. 1

