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

(11) EP 0 279 500 B2

(12) NEW EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention of the opposition decision:
04.11.1998 Bulletin 1998/45

(51) Int Cl.6: **F25J 3/04**

(45) Mention of the grant of the patent:
23.12.1992 Bulletin 1992/52

(21) Application number: **88200470.8**

(22) Date of filing: **07.03.1984**

(54) Highly pure nitrogen gas producing apparatus

Sehr reiner Stickstoffgaserzeugungsapparat

Dispositif de production de gaz d'azote à haute pureté

(84) Designated Contracting States:
DE FR GB NL

(30) Priority: **08.03.1983 JP 38050/83**
11.01.1984 JP 4123/84

(43) Date of publication of application:
24.08.1988 Bulletin 1988/34

(62) Document number(s) of the earlier application(s) in accordance with Art. 76 EPC:
84901096.2 / 0 144 430

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Printed by Jouve, 75001 PARIS (FR)

Description

This application is divided from European Patent Application No. 84 901 096.2, published on 19th June 1985 as EP-A1-0 144 430.

This invention relates to a trouble-free nitrogen gas producing apparatus which can produce pure nitrogen gas at a low cost.

In the electronics industry, quite a large quantity of nitrogen gas is used and very high purity is now demanded to maintain and improve the accuracy of parts.

Nitrogen gas has been produced so far by low temperature separation methods by which air as the raw material is compressed with a compressor, then is put into an adsorption cylinder to eliminate CO_2 gas and moisture content, then is cooled through heat exchange with refrigerant in a heat exchanger, then is turned into nitrogen gas product by low temparature separation in a rectifying column, and the nitrogen gas product is heated close to normal temperature through the said heat exchanger.

In many cases, however, trouble results if the nitrogen gas is used as it is because of oxygen content as an impurity.

To remove oxygen as an impurity, the following two methods are available.

One is to add a small quantity of hydrogen to the nitrogen gas by using Pt catalyst and to turn the oxygen into water by reaction with the hydrogen in atmosphere of about 200°C.

Another method is to put the oxygen in the nitrogen gas in contact with Ni catalyst in an atmosphere at about 200°C and to eliminate oxygen through reaction of $Ni + 1/2O_2 \rightarrow NiO$.

By either method, however, the nitrogen gas must be heated to a high temperature and be put in contact with a catalyst. It is not possible, therefore, to incorporate such apparatus into the nitrogen gas producing apparatus of an ultra-low temperature system. A refining apparatus must be installed separately from the nitrogen gas producing apparatus, which makes the whole system larger.

Moreover, the first method requires a high level of skill for operation since the quantity of hydrogen must be controlled accurately. If the hydrogen added is not in exactly the quantity required for reaction with the oxygen impurity, the oxygen or the added hydrogen is still left as an impurity.

By the second method, the cost of refining is increased by the H_2 re-generation equipment, as it is necessary to regenerate NiO produced through reaction with the oxygen impurity ($NiO + H_2 \rightarrow Ni + H_2O$). It has been demanded, therefore, to solve these problems.

For nitrogen gas producing apparatus of the conventional low temperature separation type, an expansion turbine is used for cooling the refrigerant of the heat exchanger used to cool down the compressed air by heat exchange, and the turbine is driven by the pressure of the gas evaporated from the liquid air accumulated in the rectifying column (nitrogen of low boiling point is taken out as gas by low temperature separation and the residual air is accumulated as oxygen rich liquid air).

Follow-up operation of changing load (changes in the quantity of product nitrogen gas taken out) is difficult since the rotation of the expansion turbine is very fast (several tens of thousand per minute). It is therefore, difficult to change the feeding quantity of liquid air to the expansion turbine exactly according to the changes in the quantity of product nitrogen gas taken out so that the compressed air is cooled to a constant temperature at all times.

As a result, the purity of the product nitrogen gas differs and nitrogen gas of low purity is often produced.

The expansion turbine requires high precision in its mechanical structure because of the highspeed of rotation, the cost is high, and the intricate mechanism is subjected to frequent troubles.

EP 0 107 418, which is prior art to this application under the provisions of Article 54(3) EPC, discloses apparatus of this type, employing an expansion turbine to provide refrigerant for the incoming compressed air.

An apparatus has been proposed in FR 2 225 705, in which no expansion turbine is provided and liquid nitrogen is supplied to a rectifying column. This kind of apparatus does not have the disadvantage of an expansion turbine. However, this apparatus produces nitrogen gas for preventing fire in tankers and the like, and is not required to achieve the constant high purity of nitrogen gas product required by the electronics industry. The apparatus of this document has the disadvantage that there are variations in the purity of the nitrogen gas product. Also, for preventing fire, the demand for nitrogen gas product does not fluctuate so a control means to meet fluctuations of nitrogen gas consumption is not provided. Accordingly, it is impossible for the rate of production to change in accordance with the fluctuations of nitrogen gas consumption without deterioration in the purity of the nitrogen gas.

Fig. 1 shows the nitrogen gas producing apparatus of PSA system. In the drawing, (1) is the air inlet, (2) is the air compressor, (3) is the after cooler, (3a) is the cooling water supply channel, and (4) is the oil-water separator, (5) is the 1st adsorption tank, (6) is the 2nd adsorption tank, and V1, V2 are air operated valves to fee the air compressed by the compressor (2) to the adsorption tank (5) or (6), V3 and V4 are vacuum valves to turn inside of the adsorption tank (5) or (6) to vacuum condition by the operation of the vacuum pump (6a), (6b) is the cooling pipe to supply cooling water to the vacuum pump (6a), (6c) is the silencer, and (6d) is the exhaust pipe, V5, V6, V7, and V9 are air operated valves. (7) is the product tank connected to the adsorption tanks (5) (6) through the pipe (8). (7a) is a product nitrogen gas takeout pipe, (7b) is an impurity analyzer, and (7c) is a flow-meter.

In this nitrogen gas producing apparatus, air is compressed by the air compressor (2), the compressed air is cooled by the after-cooler (3) attached to the air compressor, the condensed water is removed by the separator (4), then the compressed air is supplied into the adsorption tank (5) or (6) through the air operated valve (V1) or (V2).

Two adsorption tanks (5) (6) respectively incorporate a carbon molecular sieve for oxygen adsorption, and the compressed air is supplied into the adsorption tanks (5) (6) alternatively every minute by pressure swing method.

The inside of the adsorption tank (6) or (5) to which no compressed air is supplied is kept under vacuum condition by the vacuum pump (6a). In other words, the air compressed by the air compressor (2) goes into one of the two adsorption tanks (5) or (6) and the oxygen content is adsorbed and removed by the carbon molecular sieve, then the nitrogen gas is supplied into the product tank (7) through the valves (V5, V7, V9), and is taken out through the pipe (7a). At this time, the other adsorption tank (6) or (5) shuts off the air from the air compressor (2) since the valve (V2) closes, and the inside is evacuated by the vacuum pump (6a) as the valve (V4) opens. Accordingly, the oxygen adsorbed by the carbon molecular sieve is removed to re-generate the carbon molecular sieve.

Nitrogen gas is supplied from the adsorption tanks (5) (6) alternatively to the product tank (7) to assure continuous feeding of nitrogen gas.

By this nitrogen gas producing apparatus, the characteristic of carbon molecular sieve of selective adsorption of oxygen is effectively used to produce nitrogen gas at a low cost.

However, a number of valves are necessary, the valve operation is intricate, and trouble tends to happen frequently since the two adsorption tanks (5) (6) are operated alternately for one minute to feed compressed air and one of the tanks is subjected to evacuation.

It is necessary, therefore, to provide two sets of adsorption tanks (5) (6) and to use one set as the spare.

As described above, the nitrogen producing apparatus of PSA method is also subject to frequent trouble due to a large number of valves and requires an extra, spare, apparatus. It was therefore demanded to develop a nitrogen gas producing apparatus which can turn out highly pure nitrogen gas at a low cost.

In view of the foregoing, it is the primary object of this invention to provide a nitrogen gas producing apparatus which can produce highly pure nitrogen gas at a low cost yet without any trouble.

According to the present invention, there is provided apparatus for producing highly pure nitrogen gas as defined in claim 1.

For the apparatus, a liquefied nitrogen storing means is provided independently from the nitrogen gas separating system to separate nitrogen gas from air, the liquefied nitrogen in the storage means is supplied into the liquid nitrogen holder of the rectifying column of the nitrogen gas separating system, the compressed air supplied into the rectifying column is cooled by using evaporation heat of the liquefied nitrogen, a part of the compressed air (oxygen content in the main) is separated by liquefaction and the nitrogen is kept in gas form, then the gas mixed with the gassified liquid nitrogen after used as the cooling source of the rectifying column and is taken out as the product nitrogen gas. Accordingly, nitrogen gas can be obtained at a lower cost.

To be more specific, this apparatus uses liquefied nitrogen as the source of cooling and after use, the liquefied nitrogen is not discarded but is mixed with the nitrogen gas made from air to be turned into product nitrogen gas. Accordinly, the method is free from any waste of natural resources. Since the apparatus is provided with a condenser at the top of the rectifying column, a shortage of reflux produced in the said condenser is made up by supplying liquid nitrogen from the liquid nitrogen storage. The obtained product nitrogen gas is about 10 times of the consumed liquefied nitrogen, the cost of product nitrogen gas can be reduced substantially.

The use of liquefied nitrogen as the cooling source for the compressed air instead of an expansion turbine enables delicate adjustment of it's feeding amount. This apparatus is provided with a means to control the feeding amount of the said liquefied nitrogen from the storage which enables accurate follow-up of changing load (changes in taken-out quantity of nitrogen gas product). The purity is, therefore, stable and highly pure nitrogen gas can be produced.

Moreover, the apparatus is subjected to almost no trouble as no expansion turbine which is susceptible to troubles is used and not many valves are required unlike PSA system.

In other words, the apparatus has almost no moving parts compared with the conventional methods and is, therefore, subjected to little trouble. There is no need to prepare an extra set of adsorption tanks as the spare as it is necessary for PSA system, which can save the equipment cost.

The invention will now be further described, with reference to Figure 2 of the drawings which is a structural drawing of an embodiment of the present invention.

This shows the structure of an embodiment of the present invention. In the drawing, (9) is an air compressor (10) is a drain separator, (11) is a Freon cooler, and (12) is a pair of adsorption cylinders. The adsorption cylinders (12) are filled with molecular sieves to adsorb and remove H_2O and CO_2 in the air compressed by the air compressor (9). (13) is the 1st heat exchanger into which the compressed air after elimination of H_2O and CO_2 by the adsorption cylinders (12) is supplied. (14) is the 2nd heat exchanger, into which the compressed air coming through the 1st heat exchanger is supplied. (15) is a rectifying column provided with a condenser (16) at the top to cool the compressed air cooled down to ultra low temperature by the 1st and 2nd heat exchangers

(13, 14) still further, to turn a part of the compressed air into liquid to be kept on the bottom, and to take out nitrogen only in gas form.

That is, the rectifying column (15) functions to cool the compressed air cooled down to ultra low temperature (about -170°C) through the 1st and the 2nd heat exchangers (13, 14) further by passing through the liquefied air (18) (N_2 50 - 70%, O_2 30 - 50%) kept on the bottom of the rectifying column (15) by a pipe (17), then to jet the air inside through the expansion valve (19), and oxygen is liquefied by the condenser (16) and nitrogen is left in gas form.

The condenser (16) comprises a heat exchanger (16c) in the condenser (16), to cool the heat exchanger (16c) with the liquefied air (18) from the bottom of the tower (22), supplied through valve (19), to return the liquefied portion of the compressed air to the tower (22). Liquefied nitrogen is supplied from the liquefied nitrogen tank (23) through pipe (24) to a liquid nitrogen holder X which accumulates reflux produced in the condenser (16).

A level gauge (25) is provided to detect the level of liquefied air in the condenser (16) and adjust the valve (26) to control the supply of liquefied nitrogen from the liquefied nitrogen storage tank (23) into the column (15) according to the level of liquefied air in the condenser (16).

(27) is an outlet pipe to take out nitrogen gas from the part of the column (15) and functions to guide the nitrogen gas of ultra low temperature into the 2nd and 1st heat exchangers (14,13), to heat the gas to normal temperature by heat exchange with the compressed air supplied into the heat exchangers, and to feed into the main pipe (28). The outlet pipe (27) is provided with an oxygen adsorption cylinder (27a), which incorporate adsorbent that adsorbs oxygen and carbon monoxide selectively at ultra low temperature.

The alternate long and short dash line shows a vacuum cooling box in which the heat exchangers (13,14) and the rectifying column (15) are housed and heat-insulated by vacuum perlite.

(29) is the pipe to feed the gassified portion of the compressed air from the top of the condenser (16) into the 2nd and 1st heat exchangers (14,13), and (29a) is the pressure holding valve. After heat exchange (cooling of compressed air) in the 2nd and 1st heat exchangers (14,13), the air is discharged from the 1st heat exchanger (13) as indicated by the arrow A.

(30) is the line of back-up system to feed the liquefied nitrogen in the liquefied nitrogen storage tank (23) into the main pipe (28) through evaporation of the evaporator (31) should the line of the air compression system go out of order, (32) is an impurity analyzer to analyze the purity of the product nitrogen gas fed out to the main pipe. If the purity is low, the valves (34), (34a) are operated to discard the product nitrogen gas to the outside as shown by the arrow B.

Nitrogen gas is produced by this apparatus through the following processes.

Air is compressed by the air compressor (9) and moisture in the compressed air is removed by the drain separator (10), then the air is cooled by the Freon cooler (11), sent to the adsorption cylinders (12) as being cooled, and H_2O and CO_2 in the air are removed by adsorption.

The compressed air after removal of H_2O and CO_2 is supplied into the 1st and 2nd heat exchangers (13) (14) to be cooled down to ultra low temperature, then is cooled further by the liquid air (18) stored at the bottom of the rectifying column (15) then is ejected into the tower (22) of the rectifying column (15).

Oxygen in the air is liquefied by using the difference in the boiling point between nitrogen and oxygen (oxygen -183°C; nitrogen -196°C), nitrogen is taken out in gas form, supplied into the 1st or the 2nd heat exchanger (13 or 14) to be heated close to the normal temperature, then is taken out as nitrogen gas through the main pipe (28).

In this case, the liquefied air from the bottom of the rectifying column (15) functions as the cooling source of the condenser (16). The liquefied nitrogen itself turns into gas and is supplied into the main pipe (28), mixed with the nitrogen gas in the air from the rectifying column (15), then is taken out as product nitrogen gas.

With this nitrogen gas producing apparatus, highly pure nitrogen gas can be obtained at very low cost because an oxygen and so forth are separated from the compressed air by liquefaction using the evaporation heat of liquefied nitrogen, and only nitrogen is taken out in gas form and mixed with liquefied nitrogen serving as the cooling source (the nitrogen itself is gassified in this stage) to be turned into product nitrogen gas.

In other words, the apparatus can produce highly pure nitrogen gas with 0.3 ppm or less of impurity oxygen by setting the rectifying column (15) at high purity since no expansion turbine is used unlike the case of conventional method.

By the conventional apparatus of low temperature separation type, the nitrogen gas obtained contains oxygen of 5 ppm as impurity and by the nitrogen gas producing apparatus of PSA method, the obtained gas contains so much oxygen as 1000 ppm. Accordingly the apparatus, PSA type in particular, are not applicable as they are to electronic industry where highly pure nitrogen gas is required.

To be used for electronic industry, it is necessary to provide a refining apparatus separately and to remove oxygen (impurity) in nitrogen gas by adding hydrogen and by combining oxygen with hydrogen (into H_2O).

By this process, however, hydrogen goes into nitrogen gas as impurity and the purity is improved only a little even if the nitrogen gas is passed through a refining apparatus. Moreover, the nitrogen gas obtained from the nitrogen gas producing apparatus of PSA type contains CO_2 gas of 5 to 10 ppm as impurity and another adsorption tank to remove CO_2 gas is necessary in addition.

With the nitrogen gas producing apparatus by the present invention, on the other hand, highly pure nitrogen gas which can be used for electronic industry as it is can be obtained. Moreover, the gas does not contain any CO_2 gas (eliminated by liquefaction within the producing apparatus), and there is no need to provide any adsorption tank for CO_2 gas separately. In the condenser (16) of the rectifying column (15), a part of the nitrogen gas rising from the tower (22) and through a first connecting pipe (A') is liquefied and a part of the liquefied nitrogen pours back to the liquid nitrogen holder X in the tower (22) through a second connecting pipe (B') as reflux. At the upper part of the condenser (16), He and H_2 of lower boiling point (-269°C and 253°C) are accumulated and these get mixed if nitrogen gas is taken out from the upper part. To prevent this, it is desirable to take out nitrogen gas from the top of the tower (22) not from the top of the condenser (16).

By the nitrogen gas producing apparatus of the present invention, feeding liquefied nitrogen gas of 100 Nm^3 from the liquefied nitrogen gas tank to the condenser (16) can obtain product nitrogen gas of 1000 Nm^3. That is, the product nitrogen gas obtained is 10 times of the liquefied nitrogen supplied.

Compared with conventional nitrogen gas producing apparatus of PSA type or of low temperature separation type, the apparatus is simple and the whole system can be lower in cost, and reliability of the apparatus is higher as not many valves or no expansion turbine are required.

Moreover, nitrogen gas can be supplied even when the line of air compression system is out of order by the line of back-up system and supply of nitrogen gas is never interrupted.

As the adsorbent, synthetic zeolite 3A, 4A or 5A having pore diameter of 3A, 4A or 5A (molecular sieve 3A, 4A, or 5A made by Union Carbide) is used, for example. These synthetic zeolite 3A, 4A, and 5A respectively show highly selective adsorption property to oxygen and carbon monoxide at ultra low temperature.

Accordingly, the impurity in the discharged nitrogen gas from the upper space of the condenser (16) is eliminated and purity of the product nitrogen gas is improved further. Synthetic Zeolite 13X of Union Carbide is also used in place of the said synthetic zeolite 3A, 4A or 5A.

As a feature of this nitrogen gas producing apparatus, impurities such as oxygen and carbon monoxide are eliminated very easily by effectively using the characteristics of synthetic zeolite.

With this apparatus, the nitrogen gas produced by gassification of the liquefied nitrogen in the nitrogen tank is also passed through the oxygen adsorption cylinder (11) in the same manner as the nitrogen gas obtained from compressed air. Even when the liquefied nitrogen in the nitrogen tank contains impurities such as oxygen and carbon monoxide, therefore, the purity of the obtained product nitrogen gas is not lowered. In this case, the quantity of oxygen and carbon monoxide in the ultra low temperature nitrogen gas guided into the oxygen adsorption cylinder (11) has been reduced to a low level while going through the rectifying column (15). Accordingly, the quantity of oxygen and carbon monoxide adsorbed in the cylinder (11) is minimal. One unit of adsorption cylinder suffices and regeneration of zeolite once a year is sufficient.

Claims

1. Apparatus for producing highly pure nitrogen gas comprising a compressor (9) for compressing air from outside the apparatus, means (12) to remove the carbon dioxide gas and water from the compressed air, a heat exchanger (13,14) for cooling the compressed air to a cryogenic temperature, a rectifying column (15) for separating the air into a liquefied air portion and nitrogen vapour, a liquid nitrogen store (23), a leading channel (24) to supply liquid nitrogen from the liquid nitrogen store (23) to the upper portion of the rectifying column (15) as a refrigerant, and an outlet channel (27) for withdrawing the nitrogen from the upper portion of the rectifying column (15), and a condenser (16) comprising a heat exchanger (16c) for condensing nitrogen vapour supplied thereto from the top of the rectifying column (15) to provide liquid nitrogen reflux to the column (15) by heat exchange with the evaporating liquefied air portion supplied to the heat exchanger (16c) through a pipeline connecting the condenser (16) with the bottom of the column (15), the condenser (16) being formed to accumulate the liquefied air portion therein and liquid level detecting means (25) being provided to detect the level of the liquefied air portion in the condenser (16) and to control the supply of liquid nitrogen from the store (23) into the column (15) according to this level, the liquid nitrogen store (23) being arranged to receive and store liquid nitrogen supplied from outside the apparatus and the column (15) and the heat exchanger (13, 14) for cooling the compressed air being contained in a vacuum cooling box and heat insulated by vacuum perlite.

2. Apparatus according to claim 1 further comprising an oxygen absorption cylinder (27a) into which nitrogen is led by the outlet channel (27).

Patentansprüche

1. Apparat zur Erzeugung von hochreinem Stickstoffgas, umfassend einen Kompressor (9) zur Komprimierung von Luft von außerhalb des Apparates, Mittel (12) zur Entfernung von Kohlendioxidgas und Wasser aus der komprimierten Luft, einen Wärme-

tauscher (13, 14) zur Kühlung der Luft auf Tieftemperatur, eine Rektifikationskolonne (15) zur Trennung der Luft in einen Teil verflüssigter Luft und Stickstoffdampf, einen Flüssigstickstoff-Speicher (23), einen Zufuhrkanal (24) zur Zuführung von Flüssigstickstoff aus dem Flüssigstickstoff-Speicher (23) zum oberen Abschnitt der Rektifikationskolonne (15) als Kühlmittel, einen Auslaßkanal (27) zum Abzug des Stickstoffes aus dem oberen Abschnitt der Rektifikationskolonne (15), und einen, einen Wärmetauscher (16c) enthaltenden Kondensator (16) zur Kondensierung von Stickstoffdampf, der ihm vom oberen Ende der Rektifikationskolonne (15) zugeführt wird, um einen Flüssigstickstoff-Rückfluß zu der Kolonne (15) unter Wärrneaustausch mit dem verdampfenden Teil verflüssigter Luft vorzusehen, der dem Wärmetauscher (16c) durch eine Rohrleitung zugeführt wird, welche den Kondensator mit dem Boden der Kolonne (15) verbindet, wobei der Kondensator (16) dazu ausgebildet ist, den Teil verflüssigter Luft darin zu sammeln und Flüssigkeitsstands-Erfassungsmittel (25) vorgesehen sind, um den Stand des Teiles verflüssigter Luft in dem Kondensator (16) festzustellen und die Zufuhr von Flüssigstickstoff aus dem Speicher (23) in die Kolonne (15) in Abhängigkeit von diesem Stand zu steuern, wobei der Flüssigstickstoff-Speicher (23) zur Aufnahme und Speicherung von Flüssigstickstoff, der von außerhalb des Apparates zugeführt wird, vorgesehen ist und wobei die Kolonne (15) und die Wärmetauscher (13, 14) zur Kühlung der komprimierten Luft in einer Vakuum-Kühlbox untergebracht und durch Vakuum-Perlite gekühlt sind.

2. Apparat nach Anspruch 1, ferner umfassend einen Sauerstoff-Absorptionszylinder (27a), in welchem Stickstoff durch den Auslaßkanal (27) geleitet wird.

Revendications

1. Appareil pour la production d'un azote gazeux de haute pureté comprenant un compresseur (9) destiné à comprimer l'air de provenance extérieure à l'appareil, un moyen (12) pour éliminer le gaz de dioxyde de carbone et l'eau de l'air comprimé, un échangeur de chaleur (13, 14) pour refroidir l'air comprimé à une température cryogénique, une colonne de rectification (15) pour séparer l'air en une portion d'air liquéfié et en vapeur d'azote, un stock d'azote liquide (23), une canalisation d'amenée (24) pour fournir l'azote liquide en provenance du stock d'azote liquide (23) à la portion supérieure de la colonne de rectification (15) en tant que réfrigérant, une canalisation de sortie (27) pour prélever l'azote de la portion supérieure de la colonne de rectification (15), et un condenseur (16) comprenant un échangeur de chaleur (16c) pour condenser la vapeur d'azote fournie à celui-ci par le dessus de la colonne de rectification (15) pour fournir un reflux d'azote liquide à la colonne (15) par échange thermique avec la portion d'air liquéfiant en évaporation fournie à l'échangeur de chaleur (16c) par une conduite reliant le condenseur (16) avec le fond de la colonne (15), le condenseur (16) étant formé pour accumuler la portion d'air liquéfié et un moyen de détection de niveau liquide (25) étant prévu de façon à détecter le niveau de portion d'air liquéfié dans le condenseur (16) et pour commander l'amenée d'azote liquide en provenance du stock (23) dans la colonne (15) en fonction de ce niveau, le stock d'azote liquide (23) étant apte à recevoir et à stocker l'azote liquide fournie depuis l'extérieur de l'appareil et de la colonne (15) et de l'échangeur de chaleur (13, 14) pour refroidir l'air comprimé contenu dans un caisson de refroidissement sous vide et isolé thermiquement par perlite sous vide.

2. Appareil selon la revendication 1, comprenant de plus un cylindre d'absorption d'oxygène (27a) dans lequel est amené de l'azote par le canal de sortie (27).

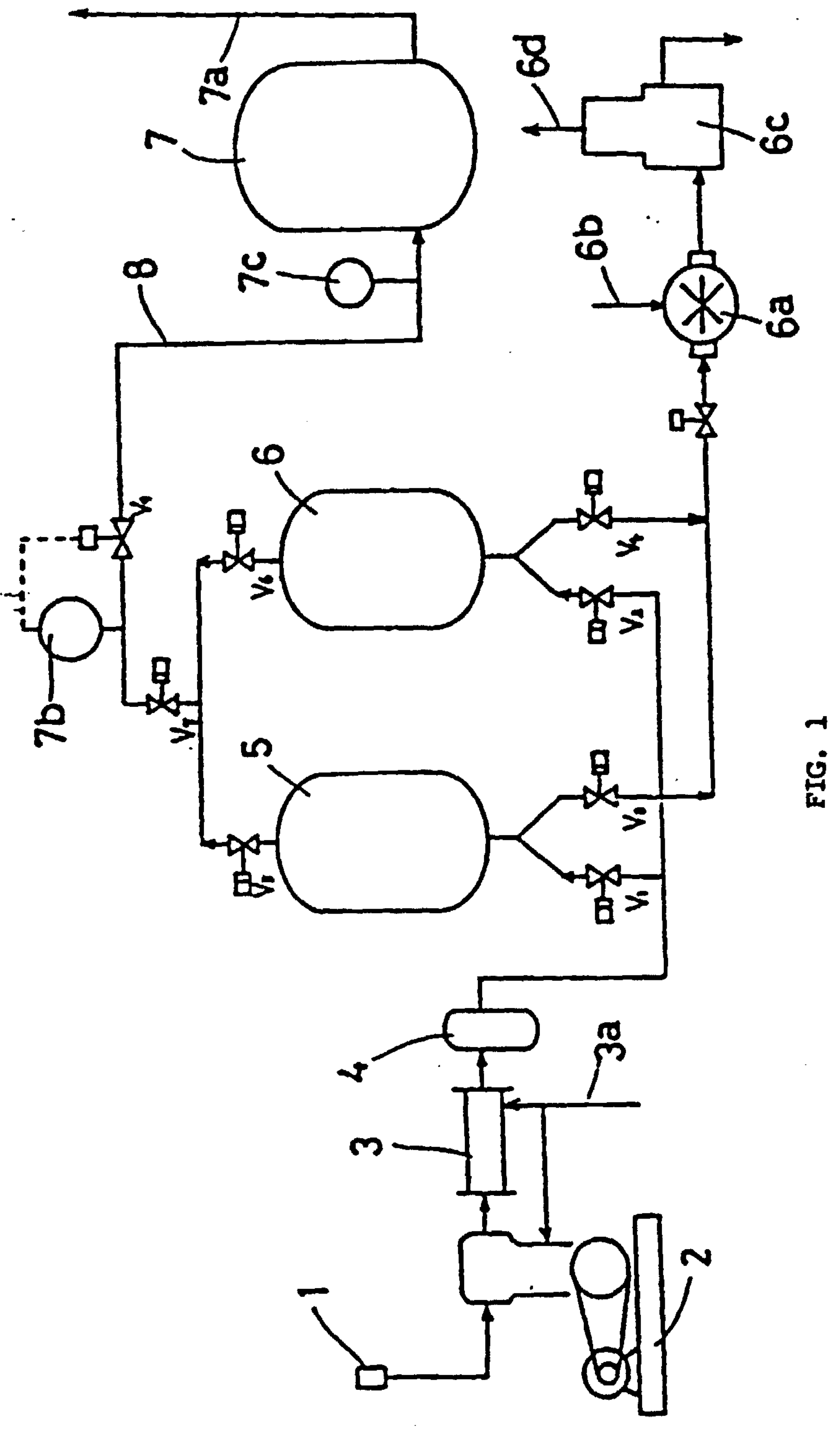

FIG. 1

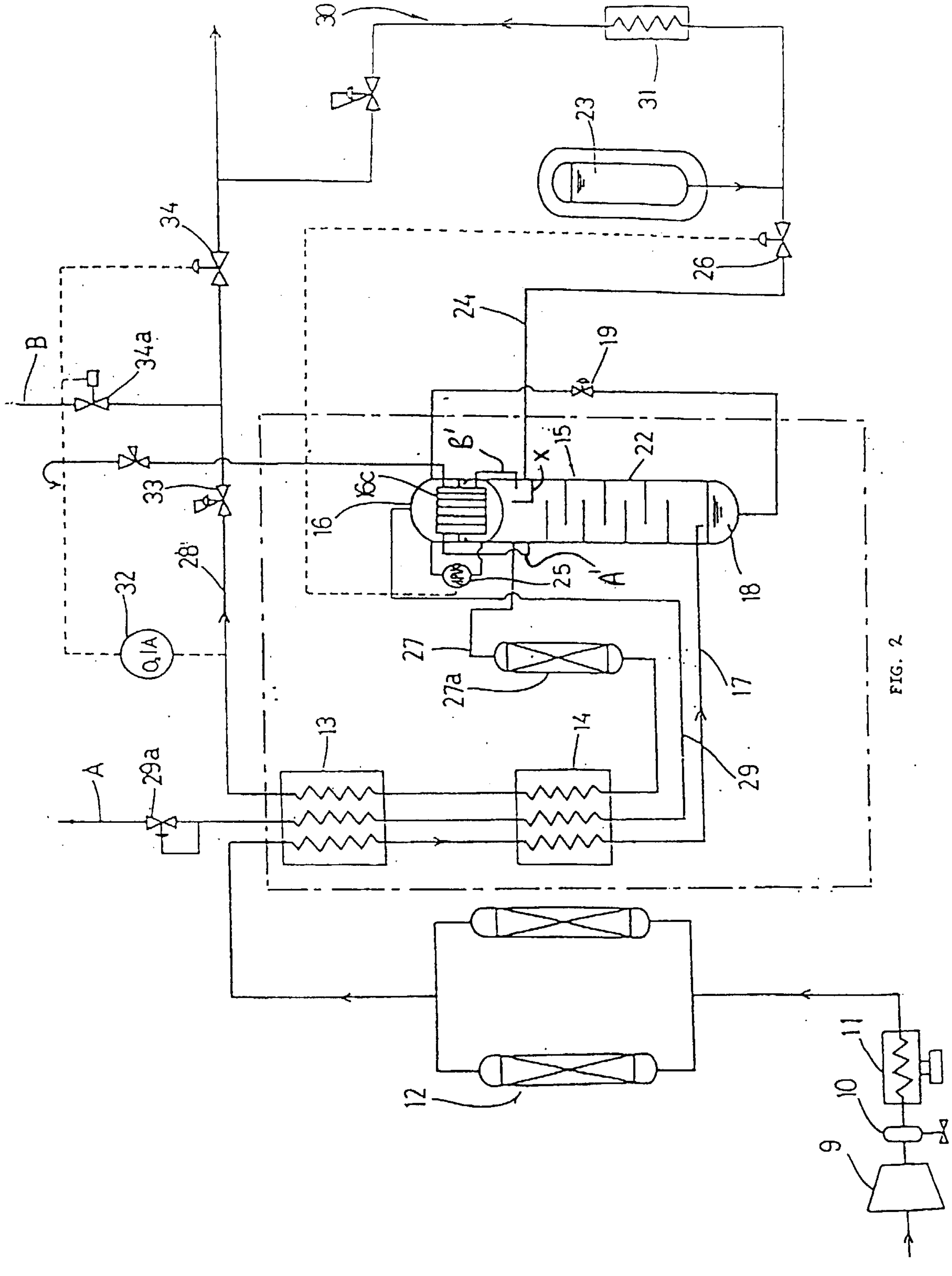
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FIG. 2