

[Document Name] SPECIFICATION

[Title of the Invention] Method and apparatus for
producing product nitrogen gas and product argon

5 [Technical Field]

[0001]

The present invention relates to a nitrogen and argon
production method and apparatus, for producing nitrogen
gas while also producing argon.

10

[Prior Art]

[0002]

A method has been proposed for producing nitrogen gas
by means of a nitrogen production apparatus employing
15 cryogenic separation, wherein energy efficiency is
improved by utilizing, as a cold source, a gas
containing oxygen drawn from a condenser portion of a
rectification column (e.g., Patent Document 1).
According to Patent Document 1, a fluid drawn from an
20 intermediate portion of the rectification column is
introduced into a main heat exchanger where it
undergoes heat exchange with raw air, and the fluid is
thereby utilized as a cold source. According to the
method disclosed, the fluid after having been utilized
25 as a cold source is expanded and cooled in an expansion
turbine, once again introduced into the main heat
exchanger, and the cold thereof is further utilized.

There is a known method in which nitrogen, argon and
30 oxygen are produced by means of an air separation
apparatus employing cryogenic separation. Oxygen and
argon have similar boiling points, so it is necessary
to perform rectification in order to separate oxygen
and argon when argon is to be produced, and high-purity
35 oxygen is also generally produced in that process (e.g.,
Patent Document 2).

[Prior Art Documents]

[Patent Documents]

[0003]

[Patent Document 1] US 4,222,756

[Patent Document 2] JP 8-61844 A

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[Summary of the Invention]

[Problem to be Solved by the Invention]

[0004]

It is difficult to apply the method disclosed in Patent
10 Document 1, in which a gas containing oxygen drawn from
a condenser portion of a rectification column is
utilized as a cold source in order to improve energy
efficiency, to a method for producing not only nitrogen
but also argon. Furthermore, although the method
15 disclosed in Patent Document 1 makes it possible to
produce nitrogen, there is no mention of producing
argon.

However, recent years have seen increasing demand for
20 extracting not only nitrogen but also argon.

[0005]

In light of the circumstances described above, the aim
of the present invention lies in providing a method for
25 producing high-purity nitrogen and argon with high
energy efficiency by utilizing the cold of an oxygen-
containing gas, and also an apparatus employed in said
method.

30 [Means for Solving the Problem]

[0006]

In one aspect, there is provided a method for producing
product nitrogen gas and product argon, comprising:
a cooling step in which raw air from which
35 predetermined impurities have been removed is cooled;
a raw air introduction step in which the raw air cooled
in the cooling step is introduced into a first
rectification column;

a first oxygen-enriched liquid introduction step in
5 which an oxygen-enriched liquid drawn from a column
bottom portion of the first rectification column is
introduced into a second rectification column;
a second oxygen-enriched liquid introduction step in
which at least a portion of the oxygen-enriched liquid
10 drawn from the column bottom portion of the first
rectification column is introduced into a second
condenser disposed in a third rectification column;
a nitrogen-containing liquid introduction step in which
a nitrogen-containing liquid condensed in a first
15 condenser is introduced as a reflux liquid at an upper
portion of the second rectification column;
an expansion step in which at least a portion of a
mixed gas is expanded to generate cold, after being
made to release cold by means of heat exchange with raw
20 air in a main heat exchanger, the mixed gas comprising
an intermediate portion gas drawn from an intermediate
portion of the second rectification column and a
condenser gas drawn from the first condenser which is
configured to perform heat exchange between a gas
25 accumulated in a column top portion of the first
rectification column, and a liquid accumulated in a
column bottom portion of the second rectification
column, and is made to release further cold by means of
reintroduction into the main heat exchanger;
30 an argon-containing gas introduction step in which an
argon-containing gas drawn from a lower portion of the
second rectification column is introduced into the
third rectification column;
a product nitrogen gas drawing step in which product
35 nitrogen gas is drawn from a column top portion of the
second rectification column; and

5 a product argon drawing step in which product argon is drawn from the intermediate portion of the third rectification column.

10 In another aspect, there is provided An apparatus for producing product nitrogen gas and product argon, comprising:
a main heat exchanger for cooling raw air from which predetermined impurities have been removed;
a first rectification column into which the cooled raw
15 air is introduced;
a second rectification column from which product nitrogen gas is drawn;
a third rectification column from which product argon is drawn;
20 a first condenser configured to perform heat exchange between a gas accumulated in a column top portion of the first rectification column, and a liquid accumulated in a column bottom portion of the second rectification column;
25 a nitrogen-containing liquid introduction pipe for introducing at least a portion of a nitrogen-containing liquid condensed in the first condenser into the second rectification column as a reflux liquid;
an argon-containing gas introduction pipe for
30 introducing an argon-containing gas into the third rectification column from a lower portion of the second rectification column;

5 an argon-containing liquid drawing pipe for introducing
an argon-containing liquid into the second
rectification column from a column bottom portion of
the third rectification column;
a condenser gas drawing pipe for drawing a condenser
gas from a gas-phase portion of the first condenser;
10 an intermediate portion gas drawing pipe for drawing an
intermediate portion gas from an intermediate portion
of the second rectification column;
an expansion turbine for expanding and cooling a mixed
gas comprising the condenser gas and the intermediate
15 portion gas, after said mixed gas has been made to pass
through the main heat exchanger; a pipe for introducing
into the main heat exchanger a gas expanded and cooled
in the expansion turbine;
a product nitrogen gas drawing pipe for drawing the
20 product nitrogen gas from the second rectification
column; and
a product argon drawing pipe for drawing the product
argon from an intermediate portion of the third
rectification column.

25

(Invention 1)

A method for producing product nitrogen gas and product
argon gas according to the present invention comprises:
a cooling step in which raw air from which
30 predetermined impurities have been removed is cooled;

a raw air introduction step in which the raw air cooled in the cooling step is introduced into a first rectification column;

5 a first oxygen-enriched liquid introduction step in which an oxygen-enriched liquid drawn from a column bottom portion of the first rectification column is introduced into a second rectification column;

10 a second oxygen-enriched liquid introduction step in which at least a portion of the oxygen-enriched liquid drawn from the column bottom portion of the first rectification column is introduced into a second condenser disposed in a third rectification column;

15 a nitrogen-containing liquid introduction step in which a nitrogen-containing liquid condensed in the first rectification column is introduced as a reflux liquid at an upper portion of the second rectification column;

20 an expansion step in which at least a portion of a mixed gas is expanded to generate cold, the mixed gas comprising an intermediate portion gas drawn from an intermediate portion of the second rectification column and a condenser gas drawn from a first condenser configured to perform heat exchange between a gas accumulated in a column top portion of the first rectification column, and a liquid accumulated in a

25 column bottom portion of the second rectification column;

30 an argon-containing gas introduction step in which an argon-containing gas drawn from a lower portion of the second rectification column is introduced into the third rectification column;

a product nitrogen gas drawing step in which product nitrogen gas is drawn from a column top portion of the second rectification column; and

35 a product argon drawing step in which product argon is drawn from the intermediate portion of the second rectification column.

The first rectification column has a higher operating pressure than the second rectification column and is able to separate the raw air into an oxygen-enriched liquid and nitrogen gas. A gas containing argon and oxygen is produced in the second rectification column, and this gas is supplied to the third rectification column. Product argon is produced in the third rectification column.

10 [0008]

Condensed raw air from which predetermined impurities have been removed is first of all cooled by means of the cooling step in the main heat exchanger in order to form low-temperature raw air. This raw air is introduced into the first rectification column in the raw air introduction step. The raw air introduced into the first rectification column comes into contact with liquid nitrogen condensed in the column top portion of the first rectification column, and is thereby rectified and separated into an oxygen-enriched liquid and nitrogen gas.

[0009]

The oxygen-enriched liquid accumulated in the lower portion of the first rectification column is supplied to a predetermined position in the second rectification column in the first oxygen-enriched liquid introduction step. The oxygen-enriched liquid supplied to the second rectification column is a starting material for the product nitrogen and product argon, and is also utilized as a refrigerant in the second rectification column.

Before being supplied to the second rectification column, at least a portion of the oxygen-enriched liquid accumulated in the lower portion of the first rectification column is supplied to the second condenser disposed in the third rectification column.

The oxygen-enriched liquid introduced is utilized as a refrigerant for condensing argon in the second condenser.

5 The oxygen-enriched liquid vaporized in the second condenser is drawn from the third rectification column, after which it is supplied as a refrigerant to a predetermined position in the second rectification column.

10

The oxygen-enriched liquid accumulated in the lower portion of the first rectification column can also be split so that a portion thereof is supplied to the second rectification column and the portion which is
15 not supplied to the second rectification column can be supplied to the second condenser, but it is equally possible to supply all of the oxygen-enriched liquid to the second condenser and then to the second rectification column. The oxygen-enriched liquid which
20 has passed through the second condenser is also supplied to the second rectification column when the oxygen-enriched liquid is split so that a portion thereof is supplied to the second rectification column without passing through the second condenser, so the
25 whole amount of the oxygen-enriched liquid is ultimately supplied to the second rectification column.

[0010]

The nitrogen-containing liquid condensed in the first
30 rectification column is introduced as a reflux liquid into the upper portion of the second rectification column in the nitrogen-containing liquid introduction step.

35 The condenser in the first rectification column is configured to perform heat exchange between the gas accumulated in the column top portion of the first rectification column and the liquid accumulated in the

column bottom portion of the second rectification column. The condenser gas is drawn from said condenser. The gas condensed in the condenser is supplied to the first rectification column as the reflux liquid, and
5 the liquid vaporized in the condenser is supplied to the second rectification column as the condenser gas. Argon-containing gas is drawn from the lower portion of the second rectification column, so the main component of the condenser gas is oxygen. This is because it is
10 necessary to concentrate and recover virtually all of the oxygen to a high degree of purity in order to produce argon by means of cryogenic separation. Otherwise, since argon and oxygen have very close boiling points, argon readily mixes with the oxygen
15 stream and flows out therewith, and it is no longer possible to recover argon. The condenser gas drawn from the lower portion of the second rectification column is therefore almost 100% pure oxygen gas.

20 [0011]

When the cold of the condenser gas is to be utilized in the main heat exchanger, it is therefore necessary to use a pipe made of a special material able to withstand the use of oxygen gas. In addition, when the condenser
25 gas whereof the cold has been used in the main heat exchanger is expanded and cooled and the cold thereof is further utilized in the main heat exchanger, it is necessary to use an expansion turbine made of a special material able to withstand the use of oxygen gas. It is
30 likewise also necessary to use a special expansion turbine able to handle high-concentration oxygen when the condenser gas is introduced directly into the expansion turbine without passing through the main heat exchanger and is expanded and cooled by means of the
35 expansion turbine. A material such as duralumin may be cited as an example of a special material, but it is difficult to obtain and costly.

[0012]

In this regard, according to the present invention, the intermediate portion gas drawn from the intermediate portion of the second rectification column is made to
5 merge with the condenser gas, after which the cold is released therefrom and expansion and cooling are performed, and cold is further generated. The intermediate portion gas contains a large amount of nitrogen gas, so when this is mixed with the condenser
10 gas, it is possible to reduce the oxygen concentration contained in the gas. As a result, it is possible to use normal pipes and a normal expansion turbine which do not employ a special material able to withstand the use of oxygen gas. It is a simple matter to obtain an
15 expansion turbine and pipes which do not employ a special material, and there is also an advantage in that the cost is low. It is also possible to reduce the oxygen concentration of the gas introduced into the expansion turbine by mixing the condenser gas with the
20 product nitrogen gas, instead of the intermediate portion gas, but this is undesirable because it reduces the amount of product nitrogen gas.

[0013]

25 When the oxygen-enriched liquid is introduced from the column bottom portion of the first rectification column into the second rectification column, the oxygen-enriched liquid may be cooled before it is introduced into the second rectification column by passage through
30 a sub-cooler. This is because it is possible to suppress a phenomenon in which the rectification efficiency decreases as a result of a large amount of the oxygen-enriched liquid being vaporized in the interior of the second rectification column, and the
35 rectification efficiency is further improved. In the sub-cooler, the fluid passing through the oxygen-enriched liquid drawing pipe and the nitrogen-containing liquid introduction pipe is cooled as a

result of undergoing heat exchange with the product nitrogen gas passing through the product nitrogen gas drawing pipe.

5 [0014]

According to the present invention, it is possible to utilize the cold of the oxygen gas to cool the raw air. The condenser gas having oxygen as the main component thereof is introduced into the main heat exchanger
10 together with the intermediate portion gas, whereby the cold of the condenser gas and the intermediate gas can be utilized, and after expansion and cooling, the cold thereof can be further utilized. It is therefore possible to utilize the cold of the oxygen which is not
15 required as a gas product in particular for applications where oxygen gas is not required but nitrogen and argon are required, so it is possible to provide a method for producing product nitrogen gas and product argon with high energy efficiency.

20

[0015]

In the second rectification column, the gas containing nitrogen comes into contact with liquid nitrogen supplied to the upper portion of the second
25 rectification column, while rising up in the interior of the second rectification column, and said gas is rectified. In this step, a portion of the argon and oxygen accompanying the gas containing nitrogen also rises in the interior of the second rectification
30 column. The accompanying argon and oxygen are mixed in with the product nitrogen gas and therefore cause a reduction in the purity of the product nitrogen gas. It would also be feasible to provide multiple distillation stages in order to completely separate the oxygen-
35 nitrogen-argon components to produce high-purity product nitrogen gas, but this would be a problem in terms of increased costs, while at the same time complete distillation and separation of the oxygen-

nitrogen-argon components would require very precise adjustment of the operating control, which would make stable operation of the apparatus difficult during fluctuations in load, such as the amount of raw air
5 supplied.

According to the present invention, the intermediate portion gas containing nitrogen is drawn from the intermediate portion of the second rectification column,
10 whereby it is possible to reduce the amount of nitrogen and oxygen rising up in the interior of the second rectification column. As a result, it is possible to reduce the amount of argon and oxygen contained in the product nitrogen gas and to increase the purity of the
15 product nitrogen gas without providing a large number of distillation stages.

[0016]

(Invention 2)

20 An apparatus (100; 101; 102; 103) for producing product nitrogen gas and product argon gas according to the present invention comprises:
a main heat exchanger (1) for cooling raw air from which predetermined impurities have been removed;
25 a first rectification column (2) into which the cooled raw air is introduced;
a second rectification column (5) from which product nitrogen gas is drawn;
a third rectification column (6) from which product
30 argon is drawn;
a first condenser (3) configured to perform heat exchange between a gas accumulated in a column top portion of the first rectification column, and a liquid accumulated in a column bottom portion of the second
35 rectification column;
a nitrogen-containing liquid introduction pipe (11) for introducing at least a portion of a nitrogen-containing

liquid condensed in the first condenser (3) into the second rectification column as a reflux liquid;
an argon-containing gas introduction pipe (17) for introducing an argon-containing gas into the third
5 rectification column from a lower portion of the second rectification column;
an argon-containing liquid drawing pipe (19) for introducing an argon-containing liquid into the second rectification column from a column bottom portion of
10 the third rectification column;
a condenser gas drawing pipe (14) for drawing a condenser gas from a gas-phase portion of the first condenser (3);
an intermediate portion gas drawing pipe (15) for
15 drawing an intermediate portion gas from an intermediate portion of the second rectification column;
an expansion turbine (8) for expanding and cooling a mixed gas comprising the condenser gas and the
20 intermediate portion gas;
a product nitrogen gas drawing pipe (16) for drawing the product nitrogen gas from the second rectification column; and
a product argon drawing pipe (18) for drawing the
25 product argon from an intermediate portion of the third rectification column.

It should be noted that the symbols given in parentheses in the present specification indicate a
30 mode of embodiment and are not limiting.

[0017]

When the intermediate portion gas drawing pipe (15) according to the present invention is not provided, the
35 oxygen concentration in the condenser gas drawn from the condenser gas drawing pipe (14) becomes extremely high (e.g., 99% or greater). This is because a gas containing argon is drawn by means of the argon-

containing gas introduction pipe (17) from the second rectification column (5) in order to produce argon gas.

According to the present invention, the intermediate
5 portion gas drawing pipe (15) is provided and the intermediate portion gas which contains nitrogen is merged with the condenser gas which contains a high concentration of oxygen, whereby the oxygen
10 concentration is reduced (e.g., the concentration is between 70% and 97%). A special material (e.g., duralumin) which is usable in oxygen gas therefore does not have to be employed for the expansion turbine (8). Accordingly, it is a simple matter to obtain the pipes and expansion turbine and there is also an advantage in
15 terms of a low cost.

The mixed gas comprising the condenser gas and the intermediate portion gas is introduced into the expansion turbine (8) where it is expanded and cooled.
20 The cold generated as a result is introduced into the main heat exchanger (1) where it is used for heat exchange with the raw air.

The mixed gas comprising the condenser gas and the
25 intermediate portion gas may be introduced into the main heat exchanger (1) before it is introduced into the expansion turbine (8). In this case, the mixed gas comprising the condenser gas and the intermediate portion gas undergoes heat exchange with the raw air in
30 the main heat exchanger (1), whereby the cold thereof is released. After further cold has been released from said gas, it is then introduced into the expansion turbine (8) where it is expanded and cooled. The expanded and cooled gas is once again introduced into
35 the main heat exchanger (1) and the cold thereof is utilized for heat exchange with the raw air.

The cold of the gas which contains oxygen is utilized in the manner described above, so it is possible to produce the product nitrogen gas and product argon gas with high energy efficiency in applications which do not especially require oxygen as a product gas.

[0018]

In addition, according to the present invention, by providing the intermediate portion gas drawing pipe (15) it is possible to reduce the amount of argon and oxygen rising up in the interior of the second rectification column (5), accompanied by the nitrogen gas rising up in the interior of the second rectification column (5). Consequently, there is an advantage in terms of higher purity of the product nitrogen gas obtained from the column top portion of the second rectification column (5).

[0019]

(Invention 3)

The apparatus for producing product nitrogen gas and product argon gas according to the present invention described above may further comprise:
an oxygen-enriched liquid drawing pipe (21) for drawing, from a column bottom portion of the first rectification column, an oxygen-enriched liquid accumulated in the column bottom portion of the first rectification column;
a second oxygen-enriched liquid introduction pipe (13) for introducing, into a second condenser (7) disposed in the third rectification column, the oxygen-enriched liquid drawn from the oxygen-enriched liquid drawing pipe (21); and
a third oxygen-enriched liquid introduction pipe (22) for introducing the oxygen-enriched liquid drawn from the second condenser into the second rectification column.

[0020]

(Invention 4)

In accordance with invention 3, the third oxygen-enriched liquid introduction pipe (22) may introduce
5 the oxygen-enriched liquid in a gaseous state from a gas-phase portion of the second condenser (7) into the second rectification column (5).

[0021]

10 The oxygen-enriched liquid is introduced into the second rectification column (5) as a refrigerant and furthermore as a starting material for the product nitrogen and product argon. However, some or all of the oxygen-enriched liquid may be introduced into the
15 second condenser (7) and the cold of the oxygen-enriched liquid may be utilized, after which it may be returned to the second rectification column (5). In this case, the oxygen-enriched liquid vaporized in the second condenser (7) is present in a gaseous state at
20 an upper portion of the second condenser (7), and is returned to the second rectification column (5) by means of the third oxygen-enriched liquid introduction pipe (22) extending from the upper portion of the second condenser (7).

25

[0022]

(Invention 5)

The apparatus for producing product nitrogen gas and product argon gas according to the present invention
30 described above may further comprise:
a fourth rectification column (9) disposed at the upper portion of the second condenser; and
a fourth rectification column column-top-portion gas introduction pipe (23) for introducing, into the second
35 rectification column, a fourth rectification column column-top-portion gas extracted from a column top portion of the fourth rectification column.

The third oxygen-enriched liquid introduction pipe introduces the oxygen-enriched liquid in a liquid state from a liquid-phase portion of the second condenser into the second rectification column.

5

The oxygen-enriched liquid accumulated in the column bottom portion of the first rectification column is introduced into the second condenser via a gas-phase portion of the fourth rectification column.

10

[0023]

By providing the fourth rectification column (9), it is possible to further concentrate the argon contained in the oxygen-enriched liquid in the interior of the fourth rectification column (9), and the argon can be supplied to the second rectification column (5). It is therefore possible to reduce the separation load in the second rectification column (5) and to improve the rectification efficiency, while it is also possible to increase the argon recovery rate.

20

[0024]

(Invention 6)

The apparatus for producing product nitrogen gas and product argon gas according to the present invention described above may also further comprise: the fourth rectification column (9) disposed at the upper portion of the second condenser; and a first oxygen-enriched liquid introduction pipe (12) for introducing, into the second rectification column, at least a portion of the oxygen-enriched liquid accumulated in the column bottom portion of the first rectification column.

30

[0025]

35 (Invention 7)

In the apparatus for producing product nitrogen gas and product argon gas according to the present invention described above, a mixed gas comprising the condenser

gas and the intermediate portion gas is introduced into the expansion turbine (8). This mixed gas may be a mixed gas comprising the condenser gas directly extracted from the gas-phase portion of the first
5 condenser (3), and the intermediate portion gas directly extracted from the intermediate portion of the second rectification column (5).

[0026]

10 The mixed gas comprising the condenser gas and the intermediate portion gas is further cooled by means of the expansion turbine (8), after which the cold thereof may be utilized in the main heat exchanger (1). By efficiently utilizing the cold of the mixed gas, it is
15 possible to improve the energy efficiency.

[0027]

(Invention 8)

Furthermore, said mixed gas may be a mixed gas in which
20 the condenser gas directly extracted from the gas-phase portion of the first condenser, and the intermediate portion gas directly extracted from the intermediate portion of the second rectification column are mixed, after which the mixture is made to pass through the
25 main heat exchanger.

[0028]

The mixed gas comprising the condenser gas and the intermediate portion gas may be introduced into the
30 main heat exchanger (1) before introduction into the expansion turbine (8), the cold thereof may be utilized, and then it may be further expanded and cooled by means of the expansion turbine (8), after which the cold thereof may be utilized in the main heat exchanger (1).
35 By effectively utilizing the cold of the mixed gas, it is possible to improve the energy efficiency.

[0029]

(Invention 9)

In the apparatus for producing product nitrogen gas and product argon gas according to the present invention described above, at least either one of the nitrogen-
5 containing liquid pipe (11) and the oxygen-enriched liquid drawing pipe (21), and the product nitrogen gas drawing pipe may be configured to pass through a sub-cooler (4).

10 [0030]

The nitrogen-containing liquid and/or oxygen-enriched liquid are/is cooled as a result of passing through the sub-cooler (4) and undergoing heat exchange with the low-temperature product nitrogen gas. As a result, it
15 is possible to suppress a phenomenon in which the rectification efficiency decreases as a result of the nitrogen-containing liquid and/or oxygen-enriched liquid introduced into the second rectification column (5) being vaporized in a large amount in the interior
20 of the second rectification column (5).

[0031]

(Invention 10)

In the apparatus for producing product nitrogen gas and
25 product argon gas according to the present invention described above, the intermediate portion gas drawing pipe, after being introduced into the sub-cooler, may be connected to the condenser gas drawing pipe at a first merging point (25). The first merging point (25)
30 is at a stage after the sub-cooler and before the expansion turbine.

[0032]

By introducing the intermediate portion gas into the
35 sub-cooler (4), it is possible to further improve the energy efficiency because the cold of the intermediate portion gas is utilized for cooling the oxygen-enriched liquid and/or the nitrogen-containing liquid.

[0033]

(Invention 11)

In the apparatus for producing product nitrogen gas and product argon gas according to the present invention described above, the intermediate portion of the second rectification column may be below an attachment position of the nitrogen-containing liquid introduction pipe (11) on the second rectification column (5) side, and may be above an attachment position of the first oxygen-enriched liquid introduction pipe (12) on the second rectification column (5) side.

[0034]

The intermediate portion gas drawing pipe (15) is attached below the nitrogen-containing liquid introduction pipe (11) and above the first oxygen-enriched liquid introduction pipe (12), and as a result it is possible to control the oxygen concentration in the gas introduced into the expansion turbine (8) to or below a predetermined concentration (e.g., 97% or less), while maintaining the purity of the product nitrogen gas at a high level.

[0035]

(Invention 12)

In the apparatus for producing product nitrogen gas and product argon gas according to the present invention described above, the ratio of the drawing flow rate of the intermediate portion gas drawn from the intermediate portion gas drawing pipe (15) to the drawing flow rate of the condenser gas drawn from the condenser gas drawing pipe (14) may be between 0.03 and 2. The ratio of the drawing flow rate of the intermediate portion gas to the drawing flow rate of the condenser gas may preferably be between 0.25 and 0.5.

[0036]

By employing the abovementioned flow rate ratio, it is possible to control the temperature to between -185°C and -165°C while maintaining the oxygen concentration
5 contained in the mixed gas comprising the condenser gas and the intermediate portion gas introduced into the main heat exchanger (1) at between 70% and 97%.

[0037]

10 In the apparatus for producing product nitrogen gas and product argon gas according to the present invention described above, the oxygen concentration in the gas introduced into the expansion turbine (8) may be between 70% and 97%. By setting the oxygen
15 concentration at between 70% and 97%, it is possible to use an expansion turbine (8) made from an inexpensive material (e.g., stainless steel).

[0038]

20 By virtue of the apparatus for producing product nitrogen gas and product argon described above, it is possible to produce product nitrogen gas and product argon with high energy efficiency by utilizing the cold of intermediate portion gas and condenser gas
25 containing oxygen, without causing a reduction in the argon recovery rate. Furthermore, utilizing the cold makes it possible to employ pipes and an expansion turbine using normal materials (e.g., stainless steel), without the use of special members which are durable in
30 relation to oxygen gas. Furthermore, it is possible to produce high-purity product nitrogen gas containing little oxygen or argon.

[Brief Description of the Drawings]

35 [0039]

[Fig. 1] shows a configuration example of an apparatus for producing product nitrogen gas and product argon according to Mode of Embodiment 1.

[Fig. 2] shows a configuration example of an apparatus for producing product nitrogen gas and product argon according to Mode of Embodiment 2.

5 [Fig. 3] shows a configuration example of an apparatus for producing product nitrogen gas and product argon according to Mode of Embodiment 3.

[Fig. 4] shows a configuration example of an apparatus for producing product nitrogen gas and product argon according to Mode of Embodiment 4.

10

[Mode of Embodiment of the Invention]

[0040]

Several modes of embodiment of the present invention will be described below. The modes of embodiment described below illustrate examples of the present invention. The present invention is in no way limited to the following modes of embodiment and also includes a number of variant examples implemented within a scope that does not alter the essential point of the present invention. It should be noted that there is no limitation in terms of all of the constituent elements described below being constituent elements which are essential to the present invention.

25 [0041]

The flow of the nitrogen production method according to the present invention will be described.

[0042]

30 (Cooling step)

The cooling step is a step in which raw air is cooled in a heat exchanger. The raw air introduced into the main heat exchanger may be raw air which has passed through a compression step in which raw air taken in from the outside is compressed by means of one or more compressors, and a removal step in which predetermined impurities are removed from the compressed raw air. There is no particular limitation as to the method for

removing impurities in the removal step, and these may be removed by means of a known method such as adsorption or cooling. There is no particular limitation as to the impurities removed, but these may
5 include carbon dioxide and moisture which cause blockage of the heat exchanger etc.

The compression step may include a cooling step in which the compressed raw air is cooled. When the raw
10 air is compressed by means of a plurality of compressors, a plurality of cooling steps for cooling the raw air compressed by each compressor may be included.

15 [0043]

In the cooling step, the raw air is cooled by means of heat exchange with at least any one of a product nitrogen gas, condenser gas and intermediate portion gas which will be described later.

20

The cooling step is implemented by a main heat exchanger 1 in the apparatus 100 for producing product nitrogen gas and product argon gas shown in fig. 1.

25 [0044]

(Raw air introduction step)

The raw air introduction step is a step in which the raw air cooled in the cooling step is introduced into a first rectification column. The raw air may be expanded
30 and cooled before introduction into the first rectification column. The raw air may be expanded and cooled by means of an expansion valve. The temperature of the raw air introduced into the first rectification column is in a range of between -170°C and -155°C, for
35 example, and the pressure is in a range of between 7.0 barA and 15 barA.

The raw air introduced into the first rectification column in the raw air introduction step is separated into an oxygen-enriched liquid and nitrogen gas. The oxygen-enriched liquid is accumulated in a column
5 bottom portion of the first rectification column, and the nitrogen gas is condensed to form liquid nitrogen by a condenser disposed at an upper portion of the first rectification column.

10 [0045]

(First oxygen-enriched liquid introduction step)

The first oxygen-enriched liquid introduction step is a step in which the oxygen-enriched liquid accumulated in the column bottom portion of the first rectification
15 column is introduced into a second rectification column. Before the oxygen-enriched liquid is introduced into the second rectification column, some or all of it may be introduced into a second condenser in a third rectification column. The temperature of the oxygen-
20 enriched liquid introduced into the second rectification column is between -175°C and -160°C , and the oxygen-enriched liquid comes into contact with a gas rising up in the second rectification column while said oxygen-enriched liquid drops down in the interior
25 of the second rectification column, and is accumulated in a condensing portion disposed between the first rectification column and the second rectification column.

30 The oxygen-enriched liquid drawn from a column bottom portion of the first rectification column may be cooled by passage through a sub-cooler before introduction into the second rectification column, but it need not pass through the sub-cooler.

35

[0046]

(Second oxygen-enriched liquid introduction step)

The second oxygen-enriched liquid introduction step is a step in which a part or all of the oxygen-enriched liquid accumulated in the column bottom portion of the first rectification column (e.g., between 10% and 100% of the oxygen-enriched liquid accumulated in the column bottom portion) is introduced into the third rectification column. The oxygen-enriched liquid introduced into the third rectification column undergoes heat exchange with argon gas in a condensing portion disposed in an upper portion of the third rectification column. The vaporized oxygen-enriched liquid drawn from the upper portion of the condensing portion disposed in the upper portion of the third rectification column is returned to the second rectification column.

Here, the vaporized oxygen-enriched liquid comes into contact with the liquid dropping down from the upper portion of the second rectification column and is rectified.

[0047]

(Nitrogen-containing liquid introduction step)

The nitrogen-containing liquid introduction step is a step in which liquid nitrogen obtained as a result of being condensed in a first condenser is introduced as a reflux liquid into an upper portion of the second rectification column. The first condenser is configured to perform heat exchange between the gas accumulated in the column top portion of the first rectification column and the liquid accumulated in the column bottom portion of the second rectification column. The temperature of the nitrogen-containing liquid introduced into the second rectification column is between -192°C and -175°C , for example.

The nitrogen-containing liquid drawn from the first condenser may be cooled by passage through a sub-cooler

before introduction into the second rectification column, but it need not pass through the sub-cooler.

[0048]

5 (Argon-containing gas introduction step)

The argon-containing gas introduction step is a step in which an argon-containing gas drawn from a lower portion of the second rectification column is introduced into the third rectification column. The
10 argon-containing gas introduced into the third rectification column is separated by means of rectification into an oxygen-enriched argon-containing liquid and product argon.

15 [0049]

(Product argon gas drawing step)

The product argon gas drawing step is a step in which the product argon gas obtained in the third rectification column is drawn from the third
20 rectification column. The purity of the product argon gas is 99.9% or greater, for example.

[0050]

(Product nitrogen gas drawing step)

25 The product nitrogen gas drawing step is a step in which the product nitrogen gas is drawn from the column top portion of the second rectification column. The purity of the product nitrogen gas is 99.9999% or greater, for example. The temperature of the product
30 nitrogen gas drawn from the column top portion of the second rectification column may be between -192°C and -175°C, and the product nitrogen gas may cool the oxygen-enriched liquid and/or the liquid nitrogen in a sub-cooler, but a sub-cooler need not be provided. The
35 product nitrogen gas is further introduced into the main heat exchanger from a cold end side and undergoes heat exchange with the raw air, after which it is drawn from a warm end side. The temperature of the product

nitrogen gas drawn from the main heat exchanger may be 0°C or greater, for example.

[0051]

5 (Expansion step)

The expansion step is a step in which a mixed gas comprising the condenser gas and the intermediate portion gas releases the cold therefrom in the main heat exchanger after which it is expanded and cooled,
10 then the expanded and cooled gas once again releases the cold therefrom in the main heat exchanger. The mixed gas comprising the condenser gas and the intermediate portion gas is introduced on the cold end side of the main heat exchanger at a temperature of
15 between -185°C and -165°C, for example. The cold is released from the mixed gas as a result of heat exchange with the raw air therein, and the temperature of the mixed gas is then between -120°C and -80°C, for example. The mixed gas is expanded and cooled by an
20 expansion turbine and the temperature thereof is then between -140°C and -100°C, for example, and it is once again introduced on the cold end side of the main heat exchanger. Here, the mixed gas undergoes heat exchange with the raw air and after the cold been released from
25 the mixed gas, it is released from the warm end side of the main heat exchanger.

The oxygen concentration of the condenser gas is 99% or greater for example, but by mixing the condenser gas
30 with the intermediate portion gas, the oxygen concentration is reduced to between 70% and 97%, for example.

[0052]

35 (Mode of Embodiment 1)

The apparatus for producing product nitrogen gas and product argon according to Mode of Embodiment 1 will be described with reference to fig. 1.

The apparatus 100 for producing product nitrogen gas and product argon according to Mode of Embodiment 1 comprises: the main heat exchanger 1, a first rectification column 2, a second rectification column 5, a third rectification column 6, a nitrogen-containing liquid introduction pipe 11, a first oxygen-enriched liquid introduction pipe 12, a second oxygen-enriched liquid introduction pipe 13, a condenser gas drawing pipe 14, an intermediate portion gas drawing pipe 15, an expansion turbine 8, a product nitrogen gas drawing pipe 16, an argon-containing gas introduction pipe 17, and a product argon drawing pipe 18.

[0053]

The apparatus 100 for producing product nitrogen gas and product argon gas is an apparatus for producing nitrogen gas and argon gas by means of cryogenic separation, and oxygen gas used as a product gas need not be produced by the apparatus.

[0054]

The main heat exchanger 1 is a heat exchanger for cooling the raw air. Before the raw air is introduced into the main heat exchanger (the amount of raw air is 1000 Nm³/h, for example), it is compressed by means of a compressor (not depicted) and predetermined impurities are removed therefrom. There is no particular limitation as to the predetermined impurities, but these may include carbon dioxide and moisture which cause blockage of the heat exchanger etc.

Inside the main heat exchanger 1, the raw air undergoes heat exchange with at least one of the product nitrogen gas, condenser gas and intermediate portion gas which will be described later. The raw air is cooled to close to the liquefaction point thereof as a result. The temperature of the raw air is 20°C, for example, when

it is introduced into the main heat exchanger 1, and it is cooled in the main heat exchanger 1 to between -170°C and -155°C, for example.

5 [0055]

The raw air cooled in the main heat exchanger 1 is introduced into the first rectification column 2 where it is rectified. The number of theoretical plates in the first rectification column 2 is between 30 plates and 80 plates, and may be set at 50 plates, for example. The operating pressure range in the first rectification column 2 is 7 barA-15 barA, and the operating pressure may be set at 9 barA, for example.

15 [0056]

The product nitrogen gas is extracted from the column top portion of the second rectification column 5. The number of theoretical plates in the second rectification column 5 is between 40 plates and 120 plates, and may be set at 80 plates, for example. The operating pressure range in the second rectification column 5 is 1.5 barA-6 barA, and the operating pressure may be set at 2.5 barA, for example.

25 [0057]

The product argon gas is extracted from the third rectification column 6. The number of theoretical plates in the third rectification column 6 is between 100 plates and 300 plates, and may be set at 180 plates, for example. The operating pressure range in the third rectification column 6 is 1.5 barA-6 barA, and the operating pressure may be set at 2.5 barA, for example.

[0058]

35 A first condenser 3 is arranged in such a way as to perform heat exchange between the gas accumulated in the column top portion of the first rectification column and the liquid accumulated in the column bottom

portion of the second rectification column. The raw air is separated into oxygen-enriched liquid and nitrogen gas in the first rectification column 2, and the oxygen-enriched liquid is accumulated in the column
5 bottom portion of the first rectification column 2. The separated nitrogen gas is condensed in the first condenser 3 to form liquid nitrogen. The oxygen-enriched liquid which will be described later is utilized as a refrigerant in the first condenser 3 as a
10 result of dropping down in the interior of the second rectification column 5 arranged above the condenser 3.

At least a portion of the liquid nitrogen obtained as a result of being condensed in the first condenser 3
15 (e.g., between 10% and 97% of the liquid nitrogen condensed in the first condenser 3) passes through the nitrogen-containing liquid introduction pipe 11 and is introduced as a reflux liquid into an upper portion of the second rectification column 5. The upper portion of
20 the second rectification column 5 is above the uppermost plate of a rectification portion in the interior of the second rectification column 5, and is above the 80th plate when the number of theoretical plates in the second rectification column 5 is 80
25 plates.

It should be noted that the second rectification column 5 may be disposed above the first condenser 3, but it may also be disposed to the side of the first condenser
30 3.

[0059]

The oxygen-enriched liquid accumulated in the column bottom portion of the first rectification column 2 is
35 drawn from the column bottom portion of the first rectification column by means of an oxygen-enriched liquid drawing pipe 21. Some or all of the oxygen-enriched liquid (e.g., between 10% and 100% of the

oxygen-enriched liquid accumulated in the column bottom portion) is introduced into the second rectification column 5 via the first oxygen-enriched liquid introduction pipe 12, and the portion of the oxygen-enriched liquid accumulated in the column bottom portion of the first rectification column 2 which is not introduced into the second rectification column 5 is introduced into the third rectification column 6 via the second oxygen-enriched liquid introduction pipe 13.

10

[0060]

An attachment position of the first oxygen-enriched liquid introduction pipe 12 on the second rectification column 5 side is below the nitrogen-containing liquid introduction pipe 11 and the intermediate portion gas drawing pipe 15 which will be described later.

An attachment position of the nitrogen-containing liquid introduction pipe 11 on the second rectification column 5 side is above the position at which the second rectification column is filled with a rectification filler. The attachment position of the first oxygen-enriched liquid introduction pipe 12 on the second rectification column 5 side may be above $2/4$ and below $3/4$ of the height of the second rectification column, for example.

When calculated by the number of theoretical plates, the attachment position of the first oxygen-enriched liquid introduction pipe 12 on the second rectification column 5 side is a position corresponding to a plate number obtained by multiplying the total number of plates by between 0.5 and 0.7. Specifically, when the number of theoretical plates in the second rectification column 5 is 80 plates, the position is above the 40th plate ($80 \times 0.5 = 40$) and below the 56th plate ($80 \times 0.7 = 56$).

The second oxygen-enriched liquid introduction pipe 13 is disposed in such a way that the oxygen-enriched liquid is introduced into the second condenser disposed in the upper region of the third rectification column 6.

- 5 The oxygen-enriched liquid which has passed through the second oxygen-enriched liquid introduction pipe 13 is utilized as a refrigerant in the second condenser 7 in order to cause condensation of the argon gas rising up in the interior of the third rectification column 6.
- 10 The oxygen-enriched liquid vaporized in the second condenser 7 may be expelled from the second condenser 7, after which it may merge in the first oxygen-enriched liquid introduction pipe 12, and may be introduced into the second rectification column 5.

15

[0061]

- The condenser gas drawing pipe 14 is a pipe for drawing the condenser gas expelled from the first condenser 3 which is configured to perform heat exchange between
- 20 the gas accumulated in the column top portion of the first rectification column 2 and the liquid accumulated in the column bottom portion of the second rectification column 5. The oxygen concentration of the condenser gas is 99.9% or greater, for example.

25

[0062]

- The intermediate portion gas drawing pipe 15 is a pipe for drawing the intermediate portion gas from the intermediate portion of the second rectification column
- 30 5. The intermediate portion gas drawing pipe 15 lies below the attachment position of the nitrogen-containing liquid introduction pipe 11 on the second rectification column side and above the attachment position of the first oxygen-enriched liquid
- 35 introduction pipe 12 on the second rectification column side. When the number of theoretical plates in the second rectification column 5 is 80 plates, the attachment position of the intermediate portion gas

drawing pipe 15 is a position between the 56th plate and the 79th plate. The nitrogen concentration of the intermediate portion gas is between 80% and 99%, for example.

5

The condenser gas drawing pipe 14 and the intermediate portion gas drawing pipe 15 merge at a stage before the main heat exchanger 1, and the intermediate portion gas and the condenser gas are mixed at that stage. The
10 oxygen concentration in the mixed gas is between 70% and 97%, for example.

The ratio of the drawing flow rate of the intermediate portion gas to the drawing flow rate of the condenser
15 gas may be between 0.1 and 2, and it may preferably be between 0.2 and 0.5.

[0063]

The expansion turbine 8 is an expansion turbine for
20 expanding and cooling the mixed gas comprising the intermediate portion gas and the condenser gas after said mixed gas has passed through the main heat exchanger 1 and the cold has been released therefrom as a result of undergoing heat exchange with the raw air
25 in the interior of the main heat exchanger 1. The temperature of the mixed gas comprising the intermediate portion gas and the condenser gas when it is first introduced into the main heat exchanger is between -185°C and -165°C, for example, and the
30 temperature before being drawn from the main heat exchanger 1 and introduced into the expansion turbine 8 is between -120°C and -80°C, for example. The mixed gas is expanded and cooled by means of the expansion turbine 8, and the temperature thereof is then between
35 -140°C and -100°C, for example. The mixed gas which has been expanded and cooled is once again introduced into the main heat exchanger 1 where it undergoes heat exchange with the raw air, whereby the cold is released

therefrom, after which the mixed gas is expelled from the main heat exchanger 1.

[0064]

5 The product nitrogen gas drawing pipe 16 is a pipe for drawing the product nitrogen gas from the column top portion of the second rectification column 5. The temperature of the product nitrogen gas which has been drawn is in a range of between -192°C and -175°C , and
10 it may be supplied as nitrogen gas without further treatment, but it may equally be introduced into the main heat exchanger 1 where it may undergo heat exchange with the raw air, whereby the cold may be released therefrom, and said nitrogen gas may be
15 supplied as nitrogen gas at a temperature of between 0°C and 20°C , for example. In addition, said nitrogen gas may also undergo heat exchange in the sub-cooler 4 before introduction into the main heat exchanger 1.

20 [0065]

The product nitrogen gas undergoes heat exchange with the nitrogen-containing liquid and oxygen-enriched liquid in the interior of the sub-cooler 4. That is to say, the cold of the product nitrogen gas is utilized
25 to cool the nitrogen-containing liquid and the oxygen-enriched liquid in the interior of the sub-cooler 4.

By virtue of the nitrogen-containing liquid and the oxygen-enriched liquid being cooled, it is possible to
30 suppress a phenomenon in which the rectification efficiency of the second rectification column 5 decreases as a result of a large amount of the nitrogen-containing liquid and the oxygen-enriched liquid being vaporized in the interior of the second
35 rectification column 5, but the sub-cooler need not be provided.

[0066]

When the sub-cooler 4 is not provided, the nitrogen-containing liquid condensed in the first condenser 3 is directly introduced into the upper portion of the second rectification column 5 by means of the nitrogen-containing liquid introduction pipe 11. The oxygen-enriched liquid drawn from the column bottom portion of the first rectification column 2 via the oxygen-enriched liquid drawing pipe 21 is likewise directly introduced into the intermediate portion of the second rectification column 5. The product nitrogen gas drawn from the column top portion of the second rectification column 5 via the product nitrogen gas drawing pipe 16 is directly introduced into the main heat exchanger 1, and after the cold of the product nitrogen gas has been utilized, the product nitrogen gas is expelled from the main heat exchanger 1.

[0067]

The argon-containing gas introduction pipe 17 is a pipe for introducing the argon-containing gas from the lower portion of the second rectification column 5 into the third rectification column 6. The attachment position of the argon-containing gas introduction pipe 17 on the second rectification column 5 side is below the first oxygen-enriched liquid introduction pipe 12, and is a position between the 20th plate and the 40th plate when the number of theoretical plates in the second rectification column 5 is 80 plates, for example.

[0068]

The argon-containing gas introduced into the third rectification column 6 is separated by means of rectification into an oxygen-enriched argon-containing liquid and product argon gas. The product argon gas is drawn from the product argon gas drawing pipe 18. Meanwhile, the oxygen-enriched argon-containing liquid accumulated in the column bottom portion of the third rectification column 6 is introduced into the second

rectification column 5 via an argon-containing liquid drawing pipe 19. The position of the argon-containing liquid drawing pipe 19 is below the product argon gas drawing pipe 18.

5

[0069]

(Mode of Embodiment 2)

An apparatus 101 for producing product nitrogen gas and product argon according to Mode of Embodiment 2 will be described with reference to fig. 2. Elements bearing the same reference symbols as those of the apparatus for producing product nitrogen gas and product argon according to Mode of Embodiment 1 have the same function and will therefore not be described again.

15

In Mode of Embodiment 2, the intermediate portion gas is introduced into the sub-cooler 4 via an intermediate portion gas drawing pipe 152. The temperature of the intermediate portion gas is raised to around -170°C in the sub-cooler 4, after which it is mixed with the condenser gas.

This makes it possible to further cool the oxygen-enriched liquid and/or the nitrogen-containing liquid, and the rectification efficiency in the second and third rectification columns can be improved.

25

[0070]

The intermediate portion gas drawing pipe 152 is connected via the sub-cooler 4 to the condenser gas drawing pipe 14 at a first merging point 25. The first merging point is positioned at a stage after the sub-cooler 4 and before the expansion turbine 8. When the mixed gas comprising the intermediate portion gas and the condenser gas is introduced into the main heat exchanger 1, before introduction into the expansion turbine, the first merging point is positioned at a

30

35

stage after the sub-cooler 4 and before the main heat exchanger 1.

The intermediate portion gas and the condenser gas are mixed to form the mixed gas at the first merging point 25. The oxygen concentration of the mixed gas is between 70% and 97%, so there is no need to use a special expansion turbine able to handle high-concentration oxygen.

[0071]

(Mode of Embodiment 3)

An apparatus 102 for producing product nitrogen gas and product argon according to Mode of Embodiment 3 will be described with reference to fig. 3. Elements bearing the same reference symbols as those of the apparatus for producing product nitrogen gas and product argon according to Mode of Embodiment 1 or Mode of Embodiment 2 have the same function and will therefore not be described again.

A fourth rectification column 9 for rectifying the oxygen-enriched liquid vaporized in the second condenser may be disposed at the upper portion of the second condenser arranged in the third rectification column 6. The oxygen-enriched liquid vaporized in the second condenser is further separated by the fourth rectification column 9 into an oxygen-enriched liquid and a nitrogen-enriched gas. Here, the nitrogen-enriched gas is extracted from the column top portion of the fourth rectification column 9, in other words the column upper portion of the third rectification column 6, and is introduced into the second rectification column via a first oxygen-enriched liquid introduction pipe 121 on the gas-phase side. Meanwhile, the liquid which has been further enriched with oxygen in the fourth rectification column 9 is accumulated in the second condenser 7 and is introduced into the

second rectification column via a first oxygen-enriched liquid introduction pipe 122 on the liquid-phase side. The oxygen-enriched liquid separated into a gas phase and a liquid phase by the fourth rectification column 9 in this way is introduced into the second rectification column 5, whereby it is possible to increase the rectification efficiency in the second rectification column.

10 [0072]

According to the third mode of embodiment, the oxygen-enriched liquid accumulated in the column bottom portion of the first rectification column 2 is drawn from the first rectification column 2 by means of the oxygen-enriched liquid drawing pipe 21. The oxygen-enriched liquid is then introduced into the upper portion of the fourth rectification column 9 from a second oxygen-enriched liquid introduction pipe 133, and is introduced into the second condenser 7 via the fourth rectification column 9.

The oxygen-enriched liquid passing through the oxygen-enriched liquid drawing pipe may be introduced into the sub-cooler 4, but it need not be introduced therein.

25

[0073]

(Different Mode of Embodiment)

As a different mode of embodiment, the intermediate portion gas drawing pipe 15 in Mode of Embodiment 3 may also be configured to pass through the sub-cooler 4.

30 [0074]

(Mode of Embodiment 4)

An apparatus 103 for producing product nitrogen gas and product argon according to Mode of Embodiment 4 will be described with reference to fig. 4. Elements bearing the same reference symbols as those of the apparatus for producing product nitrogen gas and product argon

according to Modes of Embodiment 1-3 have the same function and will therefore not be described again.

5 In Mode of Embodiment 1 to Mode of Embodiment 3, the first condenser 3 is disposed above the first rectification column 2, and the second rectification column 5 is further disposed above the first condenser 3. However, the height of the rectification column becomes extremely high overall when the components are
10 stacked upwards in this way, which may make construction and installation difficult. According to Mode of Embodiment 4, a portion corresponding to the upper portion of the second rectification column (denoted by 541 in the drawing) is disposed to the side
15 of the first rectification column 2 and the first condenser 3.

[0075]

20 In Mode of Embodiment 4, the second rectification column comprises two sections, namely a section denoted by 542 in fig. 4 and a section denoted by 541. Gas is supplied from the column top portion of the second section 542, through a pipe 41, to the column bottom portion of the first section 541 of the second
25 rectification column. Meanwhile, liquid is supplied from the column bottom portion of the first section 541, through a pipe 42 and a reflux liquid pump 30, to the column top portion of the second section 542.

30 An intermediate portion gas drawing pipe 154 draws the intermediate portion gas from an intermediate portion of the upper portion 541 of the second rectification column, and merges with the condenser gas drawing pipe 14.

35

[0076]

The fourth rectification column 9 is also likewise split into two sections, as required, and the upper

portion of the fourth rectification column may be disposed to the side of the third rectification column 6 and the second condenser 7.

5 [0077]

(Exemplary Embodiment 1)

A simulation employing the nitrogen production apparatus 100 (shown in fig. 1) according to Mode of Embodiment 1 was used to verify the pressure (barA),
10 temperature (°C) and flow rate (kg/h) etc. at each portion, when air at 1295 kg/hr comprising 75.6 wt% nitrogen, with a temperature of 20°C and a pressure of 9.0 barA was used as a starting material.

15 [0078]

(Results)

The pressure of the raw air taken in from the outside was boosted from 1.013 barA to 9.0 barA by means of a raw air compressor (not depicted).

20

After this, raw air from which carbon dioxide and moisture had been removed in a removal section was introduced into the main heat exchanger 1. The temperature of the raw air at the time of introduction
25 into the main heat exchanger 1 was 20°C. The temperature of the raw air drawn from the main heat exchanger 1 was -160°C. The raw air was introduced into the first rectification column 2 where it was rectified. The operating pressure in the first rectification
30 column 2 was 8.8 barA. The number of theoretical plates in the first rectification column 2 was 50 plates.

10 wt% of the oxygen-enriched liquid accumulated in the column bottom portion of the first rectification column
35 2 was introduced at the position of the 50th theoretical plate in the second rectification column 5 at a temperature of -180°C via the first oxygen-enriched liquid introduction pipe 12. The portion of the oxygen-

enriched liquid accumulated in the column bottom portion of the first rectification column 2 which was not introduced into the second rectification column 5 was introduced into the second condenser in the third
5 rectification column 6 at a temperature of -180°C through the second oxygen-enriched liquid introduction pipe 13.

The nitrogen gas separated at the upper portion of the
10 first rectification column was condensed in the first condenser 3 to generate liquid nitrogen. 40 wt% of the resulting nitrogen was introduced into the upper portion of the second rectification column 5 at a temperature of -190°C through the nitrogen-containing
15 liquid introduction pipe 11. The introduction position was above the position of the 80th theoretical plate. Condenser gas containing 99 wt% oxygen gas from the upper portion of the first condenser disposed between the first rectification column 2 and the second
20 rectification column 5 was expelled from the condenser gas drawing pipe 14.

Intermediate portion gas was expelled from the intermediate portion of the second rectification column
25 5 through the intermediate portion gas drawing pipe 15. The composition of the intermediate portion gas was nitrogen 85 wt%, oxygen 13 wt% and argon 2 wt%. The attachment position of the intermediate portion gas drawing pipe 15 was the position of the 55th theoretical
30 plate.

The intermediate portion gas and the condenser gas were mixed to form a mixed gas which was introduced into the main heat exchanger 1 at a temperature of -170°C , and
35 the cold was released therefrom. The oxygen concentration of the mixed gas was 84%. After this, the mixed gas drawn from the main heat exchanger 1 was introduced into the expansion turbine 8 at -110°C and

expanded and cooled, then once again introduced into the main heat exchanger 1 at a temperature of -130°C . After this, heat exchange was performed with raw air in the interior of the main heat exchanger 1, the cold was released, and the gas was expelled from the main heat exchanger 1.

Product nitrogen gas (purity 99.99 wt%) at a temperature of -185°C was drawn from the column top portion of the second rectification column 5 through the product nitrogen gas drawing pipe 16. The temperature of the product nitrogen gas was raised to -170°C by means of heat exchange in the sub-cooler 4, after which cold was further released therefrom in the main heat exchanger 1, and product nitrogen gas at 15°C was formed. The purity of the product nitrogen gas was 99.99 wt%, the argon content was 10 ppm, and the oxygen content was 100 ppb.

Argon-containing gas (argon concentration 10 wt%) was introduced from the lower portion of the second rectification column 5 into the third rectification column 6 through the argon-containing gas introduction pipe 17, and the argon-containing gas was rectified. The operating pressure in the third rectification column 6 was 2.5 barA and the number of theoretical plates was 200 plates. The product argon drawing pipe 18 was disposed at the lower portion of the second condenser and product argon having a purity of 99.9 wt% was drawn therefrom.

The oxygen-enriched argon-containing liquid accumulated in the column bottom portion of the third rectification column 6 was returned to the second rectification column 5 through the argon-containing liquid drawing pipe 19. The argon-containing liquid contained 92 wt% oxygen and 8 wt% argon.

Vaporized oxygen-enriched liquid was expelled from the upper portion of the second condenser 7 disposed at the upper portion of the third rectification column 6, and this was merged in the first oxygen-enriched liquid introduction pipe 12 and introduced into the second rectification column 5.

[0079]

By virtue of the configuration above, it was possible to obtain product nitrogen gas (935 kg/hr) at a temperature of 20°C and a pressure of 2.2 barA, and product argon (14 kg/hr) at a temperature of -175°C and a pressure of 2.3 barA. The energy required to produce the product nitrogen gas and the product argon was 110 kW, and since the cold of the intermediate portion gas and the condenser gas could be efficiently utilized, it was considered possible to produce the product nitrogen gas and product argon gas in an energy-efficient manner. Furthermore, the production could be achieved using an expansion turbine in conventional use, rather than a special material able to withstand the use of oxygen gas. In addition, by providing the intermediate portion gas drawing pipe 15, it was possible to reduce the argon and oxygen concentrations in the product nitrogen gas, and it was possible to obtain high-purity product nitrogen gas.

[Key to Symbols]

[0080]

1. Main heat exchanger
2. First rectification column
3. First condenser
4. Sub-cooler
5. Second rectification column
6. Third rectification column
7. Second condenser
8. Expansion turbine
9. Fourth rectification column

- 11. Nitrogen-containing liquid introduction pipe
- 12. First oxygen-enriched liquid introduction pipe
- 13. Second oxygen-enriched liquid introduction pipe
- 14. Condenser gas drawing pipe
- 5 15. Intermediate portion gas drawing pipe
- 16. Product nitrogen gas drawing pipe
- 17. Argon-containing gas introduction pipe
- 18. Product argon drawing pipe
- 19. Argon-containing liquid drawing pipe
- 10 21. Oxygen-enriched liquid drawing pipe
- 22. Third oxygen-enriched liquid introduction pipe
- 23. Fourth rectification column column-top-portion gas introduction pipe
- 25. First merging point
- 15 100. Apparatus for producing product nitrogen gas and product argon

[Document Name] PATENT CLAIMS

[Claim 1]

A method for producing product nitrogen gas and product
5 argon, comprising:
a cooling step in which raw air from which
predetermined impurities have been removed is cooled;
a raw air introduction step in which the raw air cooled
in the cooling step is introduced into a first
10 rectification column;
a first oxygen-enriched liquid introduction step in
which an oxygen-enriched liquid drawn from a column
bottom portion of the first rectification column is
introduced into a second rectification column;
15 a second oxygen-enriched liquid introduction step in
which at least a portion of the oxygen-enriched liquid
drawn from the column bottom portion of the first
rectification column is introduced into a second
condenser disposed in a third rectification column;
20 a nitrogen-containing liquid introduction step in which
a nitrogen-containing liquid condensed in a first
condenser is introduced as a reflux liquid at an upper
portion of the second rectification column;
an expansion step in which at least a portion of a
25 mixed gas is expanded to generate cold, after being
made to release cold by means of heat exchange with raw
air in a main heat exchanger, the mixed gas comprising
an intermediate portion gas drawn from an intermediate
portion of the second rectification column and a
30 condenser gas drawn from the first condenser which is
configured to perform heat exchange between a gas
accumulated in a column top portion of the first
rectification column, and a liquid accumulated in a
column bottom portion of the second rectification
35 column, and is made to release further cold by means of
reintroduction into the main heat exchanger;
an argon-containing gas introduction step in which an
argon-containing gas drawn from a lower portion of the
second rectification column is introduced into the
40 third rectification column;

a product nitrogen gas drawing step in which product nitrogen gas is drawn from a column top portion of the second rectification column; and
a product argon drawing step in which product argon is
5 drawn from the intermediate portion of the third rectification column.

[Claim 2]

An apparatus for producing product nitrogen gas and
10 product argon, comprising:
a main heat exchanger for cooling raw air from which predetermined impurities have been removed;
a first rectification column into which the cooled raw air is introduced;
15 a second rectification column from which product nitrogen gas is drawn;
a third rectification column from which product argon is drawn;
a first condenser configured to perform heat exchange
20 between a gas accumulated in a column top portion of the first rectification column, and a liquid accumulated in a column bottom portion of the second rectification column;
a nitrogen-containing liquid introduction pipe for
25 introducing at least a portion of a nitrogen-containing liquid condensed in the first condenser into the second rectification column as a reflux liquid;
an argon-containing gas introduction pipe for
30 introducing an argon-containing gas into the third rectification column from a lower portion of the second rectification column;
an argon-containing liquid drawing pipe for introducing an argon-containing liquid into the second
35 rectification column from a column bottom portion of the third rectification column;
a condenser gas drawing pipe for drawing a condenser gas from a gas-phase portion of the first condenser;

an intermediate portion gas drawing pipe for drawing an intermediate portion gas from an intermediate portion of the second rectification column;
an expansion turbine for expanding and cooling a mixed
5 gas comprising the condenser gas and the intermediate portion gas, after said mixed gas has been made to pass through the main heat exchanger; a pipe for introducing into the main heat exchanger a gas expanded and cooled in the expansion turbine;
10 a product nitrogen gas drawing pipe for drawing the product nitrogen gas from the second rectification column; and
a product argon drawing pipe for drawing the product argon from an intermediate portion of the third
15 rectification column.

[Claim 3]

Apparatus for producing product nitrogen gas and product argon as claimed in claim 2, comprising:
20 an oxygen-enriched liquid drawing pipe for drawing, from a column bottom portion of the first rectification column, an oxygen-enriched liquid accumulated in the column bottom portion of the first rectification column;
25 a second oxygen-enriched liquid introduction pipe for introducing, into a second condenser disposed in the third rectification column, the oxygen-enriched liquid drawn from the oxygen-enriched liquid drawing pipe; and
a third oxygen-enriched liquid introduction pipe for
30 introducing the oxygen-enriched liquid drawn from the second condenser into the second rectification column.

[Claim 4]

The apparatus for producing product nitrogen gas and
35 product argon as claimed in claim 3, wherein the third oxygen-enriched liquid introduction pipe introduces the oxygen-enriched liquid in a gaseous state from a gas-phase portion of the second condenser into the second rectification column.

40

[Claim 5]

The apparatus for producing product nitrogen gas and product argon as claimed in claim 2 or 3, further comprising:

5 a fourth rectification column disposed at an upper portion of the second condenser; and
a fourth rectification column column-top-portion gas introduction pipe for introducing, into the second rectification column, a fourth rectification column column-top-portion gas extracted from a column top
10 portion of the fourth rectification column, wherein the third enriched-oxygen liquid introduction pipe introduces the oxygen-enriched liquid in a liquid state from a liquid-phase portion of the second condenser into the second rectification column, and
15 the oxygen-enriched liquid accumulated in the column bottom portion of the first rectification column is introduced into the second condenser via a gas-phase portion of the fourth rectification column.

20 [Claim 6]

The apparatus for producing product nitrogen gas and product argon as claimed in any one of claims 2 to 5, further comprising a first oxygen-enriched liquid introduction pipe for introducing, into the second
25 rectification column, at least a portion of the oxygen-enriched liquid accumulated in the column bottom portion of the first rectification column.

[Claim 7]

30 The apparatus for producing product nitrogen gas and product argon as claimed in any one of claims 2 to 6, wherein the mixed gas is a mixed gas comprising the condenser gas directly extracted from the gas-phase portion of the first condenser, and the intermediate
35 portion gas directly extracted from the intermediate portion of the second rectification column.

[Claim 8]

The apparatus for producing product nitrogen gas and product argon as claimed in any one of claims 2 to 6, wherein the mixed gas is a mixed gas in which the condenser gas directly extracted from the gas-phase
5 portion of the first condenser, and the intermediate portion gas directly extracted from the intermediate portion of the second rectification column are mixed, after which the mixture is made to pass through the main heat exchanger.

10

[Claim 9]

The apparatus for producing product nitrogen gas and product argon as claimed in any one of claims 2 to 8, wherein at least either one of the nitrogen-containing
15 liquid pipe and the oxygen-enriched liquid drawing pipe, and the product nitrogen gas drawing pipe pass through a sub-cooler.

[Claim 10]

20 The apparatus for producing product nitrogen gas and product argon as claimed in claim 9, wherein the intermediate portion gas drawing pipe, after being introduced into the sub-cooler, is connected to the condenser gas drawing pipe at a first merging point,
25 the first merging point being at a stage after the sub-cooler and before the expansion turbine.

[Claim 11]

The apparatus for producing product nitrogen gas and
30 product argon as claimed in any one of claims 2 to 10, wherein the intermediate portion of the second rectification column is below an attachment position of the nitrogen-containing liquid introduction pipe on the second rectification column side, and is above an
35 attachment position of the first oxygen-enriched liquid introduction pipe on the second rectification column side.

[Claim 12]

The apparatus for producing product nitrogen gas and product argon as claimed in any one of claims 2 to 11, wherein the ratio of the drawing flow rate of the
5 intermediate portion gas drawn from the intermediate portion gas drawing pipe to the drawing flow rate of the condenser gas drawn from the condenser gas drawing pipe is between 0.03 and 2.