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(71) Applicant: **L'AIR LIQUIDE, SOCIETE ANONYME  
POUR L'ETUDE ET  
L'EXPLOITATION DES PROCEDES GEORGES  
CLAUDE  
75007 Paris (FR)**

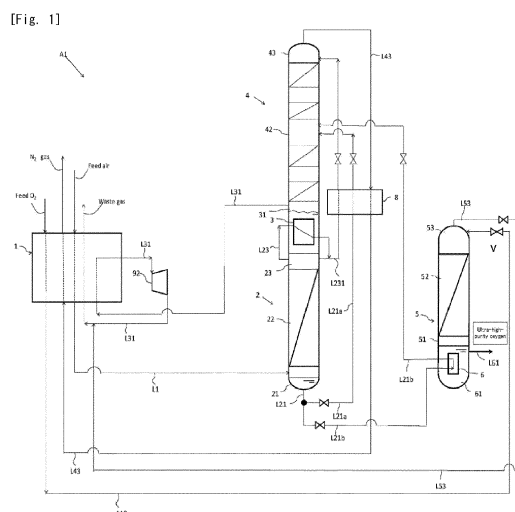
(72) Inventor: **HIROSE, Kenji**  
**HYOGO, 651-0087 (JP)**

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(74) Representative: **Air Liquide**  
**L'Air Liquide S.A.**  
**Direction de la Propriété Intellectuelle**  
**75, Quai d'Orsay**  
**75321 Paris Cedex 07 (FR)**

(54) **ULTRA-HIGH-PURITY OXYGEN PRODUCTION METHOD AND ULTRA-HIGH-PURITY OXYGEN PRODUCTION APPARATUS**

(57) An ultra-high-purity oxygen production method comprises a step in which feed oxygen comprising low-boiling-point components as impurities is introduced from a warm end of a main heat exchanger (1) and cooled, then introduced into an oxygen rectification column (5), and product ultra-high-purity oxygen from which the low-boiling-point components have been removed is drawn as a gas or a liquid from a lower portion of the oxygen rectification column.



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## Description

**[0001]** The present invention relates to an ultra-high-purity oxygen production method and an ultra-high-purity oxygen production apparatus. In particular, the present invention relates to the production of ultra-high-purity oxygen in which the impurity concentration is controlled to no greater than the ppb level.

## Background Art

**[0002]** There is a particular demand from the semiconductor industry for high-purity oxygen in which high-boiling-point components such as methane and low-boiling-point components such as argon, which are present as impurities in the oxygen, are controlled to no greater than the ppb level.

**[0003]** A method employing a catalyst in conjunction with an adsorbent, and cryogenic separation in which oxygen is liquefied and separated by means of a rectification operation are known as methods for removing the impurities, but adsorption processes and removal by molecular sieve are especially difficult for argon impurities because argon is chemically inert and its molecules are very close in size to oxygen molecules, thus cryogenic separation is suitable.

**[0004]** Known methods for obtaining ultra-high-purity oxygen include a method in which liquefied oxygen supplied from an air separation unit (JP 3929799) or oxygen gas (JP 2021-55890 A) is rectified, or a method in which an oxygen-containing liquid from which the high-boiling-point components have been removed is drawn from a rectification column for rectifying air, and argon is removed by an oxygen rectification column.

**[0005]** US 5049173 A describes a method for producing ultra-high-purity oxygen in a double-column rectification system, and WO 2014/173496 A2 describes a method for producing ultra-high-purity oxygen in a single-column nitrogen rectification system.

**[0006]** However, by-product oxygen from generating hydrogen by electrolysis of water is sometimes used as a feed material for the production of ultra-high-purity oxygen. Unlike the oxygen obtained from an air separation unit which uses atmospheric components as the feed material, the oxygen derived from water electrolysis does not contain high-boiling-point components derived from the atmosphere such as methane, but it does contain a certain amount of low-boiling-point components dissolved in the water. Low-boiling-point impurities in the water may be reduced by means of bubbling, etc. which utilizes nitrogen gas or oxygen gas, but it would be desirable to control the process by means of cryogenic separation from the point of view of stably removing impurities to a high level.

**[0007]** In the prior art, it would be reasonable to apply the technology disclosed in JP 2021-55890 A

**[0008]** for cryogenic separation of oxygen gas, but there is a need to develop less costly technology because

of the high cost of a nitrogen heating medium cycle utilizing multiple heat exchangers and compressors.

**[0009]** CN114017993A describes a process for liquefying oxygen in the main heat exchanger of an air separation unit.

**[0010]** WO2023274574 relates to a process in which oxygen from an electrolyser is sent to an air separation unit without being mixed with any fluid from that unit. Figure 2 shows an oxygen purification column, fed with a stream from an air separation column and a stream from an electrolyser.

**[0011]** The present disclosure provides an ultra-high-purity oxygen production method and an ultra-high-purity oxygen production apparatus that enable ultra-high-purity oxygen to be obtained at a low cost by removing low-boiling-point components from by-product oxygen obtained for example from water electrolysis.

**[0012]** According to an object of the invention, there is provided an ultra-high-purity oxygen production method utilizing an air separation unit comprising a main heat exchanger, a nitrogen rectification column, a nitrogen condenser, an oxygen rectification column, and an oxygen vaporizer, wherein the method comprises:

- a step in which feed oxygen comprising low-boiling-point components as impurities is introduced from a warm end of the main heat exchanger, cooled and at least partially liquefied, then introduced into the oxygen rectification column, and ultra-high-purity oxygen from which the low-boiling-point components have been removed is drawn as a gas or a liquid from a lower portion of the oxygen rectification column or from the oxygen vaporizer; and
- a step in which one or more of: a portion of feed air cooled in the main heat exchanger, a portion of the feed oxygen cooled in the main heat exchanger, and a liquid or gas drawn from a medium-pressure rectification column constituting the nitrogen rectification column is utilized as a heating medium in the oxygen vaporizer, liquefied oxygen supplied from a bottom portion of the oxygen rectification column is vaporized, and a vapour stream thereof is supplied to the bottom portion of the oxygen rectification column.

**[0013]** According to other optional features, the method further comprises:

- a step in which liquefied nitrogen or an oxygen-containing liquid supplied from the medium-pressure rectification column, or liquid nitrogen or liquefied air supplied from outside the air separation unit is utilized as a refrigerant in an oxygen condenser provided above or in a top portion of the oxygen rectification column, and a low-boiling-point component-containing oxygen stream supplied from the oxygen rectification column is liquefied and supplied to a top portion of the oxygen rectification column as a reflux

liquid.

- a step in which at least a portion of the feed oxygen cooled in the heat exchanger is expanded to provide refrigeration.
- a portion of the feed oxygen drawn from partway through the main heat exchanger is expanded by an expansion turbine and cooled, after which it is once again supplied to the main heat exchanger.
- the feed oxygen is expanded in a valve upstream of the oxygen rectification column.

**[0014]** According to another object of the invention, there is provided an ultra-high-purity oxygen production apparatus comprising: a main heat exchanger into which feed air and feed oxygen are introduced;

- a first rectification column having a bottom portion into which the feed air, that has undergone heat exchange in the main heat exchanger, is introduced;
- at least one nitrogen condenser for condensing a nitrogen-rich gas drawn from a column top of the first rectification column;
- a second rectification column having a column top into which is introduced a nitrogen-rich gas condensed in the nitrogen condenser and/or a nitrogen-rich liquid drawn from the column top of the first rectification column, after said nitrogen-rich liquid has been cooled in a sub-cooler;
- an oxygen rectification column having a column top or a purification portion into which the feed oxygen that has undergone heat exchange in the main heat exchanger is introduced;
- an oxygen vaporizer which is arranged below a bottom portion of the oxygen rectification column or within the oxygen rectification column and vaporizes liquefied oxygen while using, as a heating medium, one or more of: a portion of feed air cooled in the main heat exchanger, a portion of the feed oxygen cooled in the main heat exchanger, and a liquid or gas drawn from a medium-pressure rectification column constituting the nitrogen rectification column; and
- a sub-cooler for performing heat exchange of: an oxygen-enriched liquid drawn from the bottom portion of the first rectification column, a purified gas condensed in the nitrogen condenser and/or a purified gas drawn from the column top of the first rectification column, and a nitrogen-rich gas drawn from the column top of the second rectification column.

**[0015]** According to other optional features, the apparatus comprises:

- -an expansion turbine into which a gas drawn from a gas phase in the nitrogen condenser is introduced, after said gas has been passed through a part of the main heat exchanger.
- an oxygen condenser for condensing a low-boiling-

point component-containing oxygen gas drawn from the column top of the oxygen rectification column.

- a feed oxygen pipeline for introducing the feed oxygen, via the main heat exchanger, into the column top or a rectification portion of the oxygen rectification column;
- a branch feed oxygen pipeline which branches the feed oxygen from partway through the main heat exchanger in the feed oxygen pipeline, and merges into a pipeline before connection to the expansion turbine; and
- an ultra-high-purity oxygen extraction pipeline for extracting ultra-high-purity oxygen from a vaporized liquid portion of the oxygen vaporizer.

**[0016]** According to another object of the invention, there is provided an ultra-high-purity oxygen production apparatus comprising: a main heat exchanger into which feed air and feed oxygen are introduced;

- a nitrogen rectification column having a bottom portion into which the feed air, that has undergone heat exchange in the main heat exchanger, is introduced;
- a first nitrogen condenser for condensing a nitrogen-rich gas drawn from a column top of the nitrogen rectification column;
- a second nitrogen condenser for condensing the nitrogen-rich gas drawn from the column top of the nitrogen rectification column;
- an expansion turbine into which a gas drawn from a gas phase in the first nitrogen condenser is introduced, after said gas has been passed through a part of the main heat exchanger;
- a compressor for compressing a gas drawn from a gas phase in the second nitrogen condenser;
- an oxygen rectification column having a column top or a purification portion into which the feed oxygen that has undergone heat exchange in the main heat exchanger is introduced; and
- an oxygen vaporizer which is arranged below the bottom portion of the oxygen rectification column and vaporizes liquefied oxygen while using, as a heating medium, one or more of: a portion of feed air cooled in the main heat exchanger, a portion of the feed oxygen cooled in the main heat exchanger, and a liquid or gas drawn from the nitrogen rectification column.

**[0017]** According to optional features, the apparatus may comprise:

expansion means - for expanding at least part of the feed oxygen following cooling in the main heat exchanger to provide refrigeration for the apparatus.

**[0018]** The oxygen rectification column may be connected so as to be fed only by the feed oxygen.

**[0019]** The oxygen rectification column is connected so as to be fed only by feed oxygen coming from an electrolyser.

**[0020]** The apparatus may comprises an integrated apparatus including an electrolyser, means for sending water to the electrolyser, means for removing hydrogen from the electrolyser, means for removing an oxygen rich stream from the electrolyser, an ultra high purity oxygen production apparatus as described above and means for sending the oxygen rich stream from the electrolyser as the feed oxygen.

**[0021]** The ultra-high-purity oxygen production method according to the present disclosure may be applied in an air separation unit comprising: a main heat exchanger, a nitrogen rectification column (first (medium-pressure) rectification column), a nitrogen condenser, an oxygen rectification column, and an oxygen vaporizer.

**[0022]** The ultra-high-purity oxygen production method comprises a step in which feed oxygen comprising low-boiling-point components (e.g., nitrogen and argon) as impurities is introduced from a warm end of the main heat exchanger (1) and cooled, then introduced into the oxygen rectification column (5), and product ultra-high-purity oxygen from which the low-boiling-point components have been removed is drawn as a gas or a liquid from a lower portion of the oxygen rectification column (5) or from the oxygen vaporizer (6).

**[0023]** The method may also comprise a step in which the feed oxygen which has been at least partially liquefied in the main heat exchanger is introduced into the oxygen rectification column.

**[0024]** The method may also comprise a step in which one or more of: a portion of feed air cooled in the main heat exchanger, a portion of the feed oxygen cooled in the main heat exchanger, and a liquid or gas drawn from the medium-pressure rectification column is utilized as a heating medium in the oxygen vaporizer, liquefied oxygen supplied from a bottom portion of the oxygen rectification column is vaporized, and a vapour stream thereof is supplied to the bottom portion of the oxygen rectification column.

**[0025]** An oxygen condenser may be provided above or in a top portion of the oxygen rectification column.

**[0026]** Examples of liquid or gas drawn from the medium-pressure rectification column (2) that may be cited include oxygen-containing liquid, oxygen-containing gas, liquefied nitrogen, and nitrogen gas, etc.

**[0027]** The method may also comprise a step in which liquefied nitrogen or an oxygen-containing liquid supplied from the medium-pressure rectification column, or liquid nitrogen or liquefied air supplied from outside the air separation unit is utilized as a refrigerant in the oxygen condenser, and a low-boiling-point component-containing oxygen stream supplied from the oxygen rectification column is liquefied and supplied to a top portion of the oxygen rectification column as a reflux liquid.

**[0028]** The method may also comprise a step in which a portion of the feed oxygen drawn from partway through the main heat exchanger is expanded by an expansion turbine (92) and cooled, after which it is once again supplied to the main heat exchanger (1), so as to maintain

a heat balance in the main heat exchanger.

**[0029]** "Ultra-high-purity oxygen" means an oxygen concentration of 99.9999% or greater. "Feed oxygen" may be by-product oxygen (high purity oxygen, oxygen concentration of around 99.99%) generated by means of water electrolysis.

**[0030]** An ultra-high-purity oxygen production apparatus according to the present disclosure may comprise:

- a main heat exchanger into which feed air and feed oxygen are introduced;
- a first (medium-pressure) rectification column having a bottom portion into which the feed air, that has undergone heat exchange in the main heat exchanger, is introduced;
- at least one nitrogen condenser for condensing a nitrogen-rich gas drawn from a column top of the first rectification column;
- a second (low-pressure) rectification column having a column top into which is introduced a nitrogen-rich gas condensed in the nitrogen condenser and/or a nitrogen-rich gas drawn from the column top of the first rectification column, after said nitrogen-rich gas has been cooled in a sub-cooler;
- an expansion turbine into which a gas drawn from a gas phase in the nitrogen condenser (3) is introduced, after said gas has been passed through a part of the main heat exchanger;
- an oxygen rectification column having a column top or a purification portion into which the feed oxygen that has undergone heat exchange in the main heat exchanger is introduced; and
- an oxygen vaporizer which is arranged below a bottom portion of the oxygen rectification column (5) and vaporizes liquefied oxygen while using, as a heating medium, one or more of: a portion of feed air cooled in the main heat exchanger, a portion of the feed oxygen cooled in the main heat exchanger, and a liquid or gas drawn from a medium-pressure rectification column constituting the nitrogen rectification column (e.g., while using an oxygen-rich liquid drawn from the bottom portion of the first rectification column as the heating medium); and
- a sub-cooler for performing heat exchange of: an oxygen-rich liquid drawn from the bottom portion of the first rectification column, a purified gas condensed in the nitrogen condenser and/or a purified gas drawn from the column top of the first rectification column, and a nitrogen-rich gas drawn from the column top of the second rectification column.

**[0031]** The ultra-high-purity oxygen production apparatus may comprise:

- an oxygen condenser for condensing a low-boiling-point component-containing oxygen gas drawn from the column top of the oxygen rectification column.
- The ultra-high-purity oxygen production apparatus

may comprise:

- a feed air pipeline for introducing, via the main heat exchanger, the feed air into a gas phase in the bottom portion of the first rectification column (2), or into a lower portion of a purification portion);
- a first oxygen-rich liquid pipeline for introducing, into an intermediate stage of a rectification portion of the second rectification column, via the sub-cooler, an oxygen-rich liquid drawn from the bottom portion of the first rectification column;
- a condensing pipeline which delivers, to the nitrogen condenser, a nitrogen-rich gas drawn from the column top of the first rectification column, and which merges with a pipeline leading out from the column top;
- a first circulation gas pipeline for introducing, into the column top of the second rectification column (4), via the sub-cooler, the nitrogen-rich gas drawn from the column top of the first rectification column;
- a first waste gas pipeline for causing the gas, which is drawn from the gas phase in the nitrogen condenser, to pass through a part of the main heat exchanger, the gas then being used in the expansion turbine, and once again passed through the main heat exchanger;
- a product nitrogen gas pipeline for causing the nitrogen-rich gas drawn from the column top of the second rectification column to pass through the main heat exchanger, via the sub-cooler;
- a feed oxygen pipeline for introducing the feed oxygen, via the main heat exchanger, into the column top or the rectification portion of the oxygen rectification column;
- a branch feed oxygen pipeline which branches the feed oxygen from partway through the main heat exchanger in the feed oxygen pipeline, and merges into the first waste gas pipeline before connection to the expansion turbine;
- a second waste gas pipeline which causes a low-boiling-point component-containing oxygen gas, which is drawn from the column top of the oxygen rectification column, to merge into the waste gas pipeline or to pass through the main heat exchanger;
- a second oxygen-rich liquid pipeline for introducing the oxygen-rich liquid drawn from the bottom portion of the first rectification column into the oxygen vaporizer, from where it is introduced into the intermediate stage of the rectification portion of the second rectification column, or for introducing said oxygen-rich liquid into a cold heat liquid portion of the oxygen condenser for condensing the low-boiling-point component-containing oxygen gas drawn from the top portion of the oxygen rectification column;
- a second circulation gas pipeline for introducing, into the intermediate stage of the rectification portion of the second rectification column, an oxygen-rich liquid drawn from the cold heat liquid portion of the oxygen condenser;

- a third circulation gas pipeline for introducing, into the intermediate stage of the rectification portion (of the second rectification column, a gas drawn from a top portion of the oxygen condenser; and
- an ultra-high-purity oxygen extraction pipeline for extracting ultra-high-purity oxygen (liquid) from a vaporized liquid portion of the oxygen vaporizer.

**[0032]** An ultra-high-purity oxygen production apparatus according to other disclosure may comprise:

- a main heat exchanger into which feed air and feed oxygen are introduced;
- a nitrogen rectification column having a bottom portion into which the feed air, that has undergone heat exchange in the main heat exchanger, is introduced;
- a first nitrogen condenser for condensing a nitrogen-rich gas drawn from a column top of the nitrogen rectification column;
- a second nitrogen condenser for condensing the nitrogen-rich gas drawn from the column top of the nitrogen rectification column;
- an expansion turbine into which a gas drawn from a gas phase in the first nitrogen condenser is introduced, after said gas has been passed through a part of the main heat exchanger;
- a compressor which is connected to the expansion turbine and compresses a gas drawn from a gas phase in the second nitrogen condenser;
- an oxygen rectification column having a column top or a purification portion into which the feed oxygen that has undergone heat exchange in the main heat exchanger is introduced; and
- an oxygen vaporizer which is arranged below a bottom portion of the oxygen rectification column (5) and vaporizes liquefied oxygen while using, as a heating medium, an oxygen-rich liquid drawn from the bottom portion of the nitrogen rectification column.

**[0033]** The ultra-high-purity oxygen production apparatus may comprise:

- a feed air pipeline for introducing, via the main heat exchanger, the feed air into a gas phase in the bottom portion of the nitrogen rectification column, or into a lower portion of a rectification portion;
- a first oxygen-rich liquid pipeline for introducing, into a cold heat liquid portion (not depicted) of the second nitrogen condenser, the oxygen-rich liquid drawn from the bottom portion of the nitrogen rectification column;
- a first condensing pipeline for delivering a nitrogen-rich gas drawn from the column top of the nitrogen rectification column to the first nitrogen condenser and returning same to the column top;
- a second condensing pipeline for delivering the nitrogen-rich gas drawn from the column top of the

- nitrogen rectification column to the second nitrogen condenser and returning same to the column top;
- a product nitrogen gas pipeline for causing the nitrogen-rich gas drawn from the column top of the nitrogen rectification column to pass through the main heat exchanger;
  - an oxygen-containing liquid pipeline for introducing an oxygen-containing liquid drawn from the rectification portion of the nitrogen rectification column into the column top or rectification portion of the oxygen rectification column;
  - a first waste gas pipeline for causing the gas, which is drawn from the gas phase (31) in the column top of the first nitrogen condenser, to pass through a part of the main heat exchanger, the gas then being used in the expansion turbine, and once again passed through the main heat exchanger;
  - a recycled gas pipeline for causing a gas drawn from a gas phase in the column top of the second nitrogen condenser to be compressed in the compressor, then passed through a part of the main heat exchanger, from where it is introduced into a lower portion of the rectification portion of the nitrogen rectification column;
  - a feed oxygen pipeline for introducing the feed oxygen, via the main heat exchanger, into the column top or the rectification portion of the oxygen rectification column;
  - a branch feed oxygen pipeline which branches the feed oxygen from partway through the main heat exchanger in the feed oxygen pipeline (L10), and merges into the first waste gas pipeline before connection to the expansion turbine;
  - a second waste gas pipeline which causes a low-boiling-point component-containing oxygen gas, which is drawn from the column top of the oxygen rectification column, to merge into the waste gas pipeline or to pass through the main heat exchanger;
  - a second oxygen-rich liquid pipeline for introducing the oxygen-rich liquid drawn from the bottom portion of the nitrogen rectification column into the oxygen vaporizer, from where it is introduced into the cold heat liquid portion (not depicted) of the second nitrogen condenser; and
  - an ultra-high-purity oxygen extraction pipeline for extracting ultra-high-purity oxygen (liquid) from a vaporized liquid portion of the oxygen vaporizer.

**[0034]** The oxygen vaporizer of the ultra-high-purity oxygen production apparatus may utilize, as a heating medium, one or more of: a portion of the feed air cooled in the main heat exchanger, a portion of the feed oxygen cooled in the main heat exchanger, and an oxygen-containing liquid or liquefied nitrogen drawn from the medium-pressure rectification column.

**[0035]** The ultra-high-purity oxygen production apparatus may comprise:

- various measurement instruments such as flow rate measurement instruments, pressure measurement instruments, temperature measurement instruments, and liquid level measurement instruments;
- various valves such as control valves and gate valves; and
- pipes for connecting the components.

(1) By-product oxygen obtained by water electrolysis can be used to efficiently generate ultra-high-purity oxygen using fewer items of equipment than in the prior art, by combining an oxygen rectification column with an air separation unit or a nitrogen generating apparatus for producing nitrogen gas.

(2) In particular, the process for liquefying feed oxygen in the main heat exchanger is only possible because a sufficiently low temperature to liquefy oxygen can be ensured at the cold end of the main heat exchanger of the air separation unit, unlike in an oxygen gas purification method such as can be seen in the prior art.

(3) A considerable cost reduction can be envisaged in terms of equipment investment costs per apparatus, as compared with a conventional method requiring a cycle nitrogen compressor and a dedicated main heat exchanger, while at the same time the power required for the cycle nitrogen compressor can also be reduced.

(4) The present invention is of use in a semiconductor production process employing a water electrolysis apparatus.

#### Brief Description of the Drawings

#### **[0036]**

[Fig. 1] shows an ultra-high-purity oxygen production apparatus according to embodiment 1.

[Fig. 2] shows an ultra-high-purity oxygen production apparatus according to embodiment 2.

[Fig. 3] shows an ultra-high-purity oxygen production apparatus according to embodiment 3.

[Fig. 4] shows an ultra-high-purity oxygen production apparatus according to embodiment 4.

[Fig. 5] shows an ultra-high-purity oxygen production apparatus according to embodiment 5.

#### Embodiments of the Invention

**[0037]** Several embodiments of the present disclosure will be described below. The embodiments described below are given as an example of the present disclosure. The present disclosure is in no way limited by the following embodiments, and also includes a number of variant modes which are implemented within a scope that does not alter the essential point of the present disclosure. It should be noted that not all the constituents described

below are necessarily essential to the present disclosure. Upstream and downstream are based on a flow direction of a gas stream.

#### Embodiment 1

**[0038]** An ultra-high-purity oxygen production apparatus A1 according to embodiment 1 will be described with the aid of fig. 1.

**[0039]** The ultra-high-purity oxygen production apparatus A1 constitutes an air separation unit comprising: a main heat exchanger 1, a medium-pressure rectification column 2, a nitrogen condenser 3, a low-pressure rectification column 4, an expansion turbine 92, an oxygen rectification column 5, an oxygen vaporizer 6, and a sub-cooler 8.

**[0040]** Feed air and feed oxygen are introduced into the main heat exchanger 1 from a warm end thereof and drawn from a cold end thereof, while product nitrogen gas and waste gas are introduced from the cold end thereof and drawn from the warm end thereof. Predetermined impurities and moisture are removed from the feed air. The feed oxygen is by-product oxygen from water electrolysis, and contains low-boiling-point components (e.g., nitrogen and argon) as impurities. The oxygen concentration of the feed oxygen is around 99.99%.

**[0041]** The feed oxygen has previously been dried to remove water and may also be purified to removed other impurities, such as residual hydrogen. The feed oxygen is at a pressure between 15 and 30 bars abs within the heat exchanger 1.

**[0042]** In order to remove the low-boiling-point components in the oxygen by cryogenic separation, the oxygen is preferably liquefied first of all, then undergoing heat and substance exchange with a vapour stream containing oxygen inside a rectification column so that the low-boiling-point components are removed while oxygen is concentrated in the liquid phase, and, for this purpose, at least a portion of the feed oxygen is liquefied in the main heat exchanger 1 in this embodiment, after which the feed oxygen is fed to the oxygen rectification column 5.

**[0043]** The medium-pressure rectification column 2 comprises: a bottom portion 21 into which the feed air cooled in the main heat exchanger 1 is introduced, a rectification portion 22, and a column top 23. A feed air pipeline L1 is a pipeline for introducing the feed air, via the main heat exchanger 1, into a gas phase in the bottom portion 21 of the medium-pressure rectification column 2, or into a lower portion of a purification portion 22. A first oxygen rich liquid pipeline L21a is a pipeline for introducing, into an intermediate stage of a rectification portion 42 of the low-pressure rectification column 4, via the sub-cooler 8, an oxygen-rich liquid drawn from the bottom portion 21 of the medium-pressure rectification column 2. The first oxygen-rich liquid pipeline L21a and a second oxygen-rich liquid pipeline L21b may branch from a main pipeline L21 for the oxygen-rich liquid. A condensing

pipeline L23 is a pipeline which delivers, to the nitrogen condenser 3, a nitrogen-rich gas drawn from the column top 23 of the medium-pressure rectification column 2, and which merges with a first circulation gas pipeline L231 leading out from the column top 23. The first circulation gas pipeline L231 is a pipeline for introducing, into a column top 43 of the low-pressure rectification column 4, via the sub-cooler 8, a nitrogen-rich gas drawn from the column top 23 of the medium-pressure rectification column 2.

**[0044]** The nitrogen condenser 3 condenses a nitrogen-rich gas drawn from the column top 23 of the medium-pressure rectification column 2. A first waste gas pipeline L31 is a pipeline for causing the gas, which is drawn from the gas phase the nitrogen condenser 3, to pass through a part of the main heat exchanger 1, the gas then being used in the expansion turbine 92, and once again passed through the main heat exchanger 1.

**[0045]** The low-pressure rectification column 4 has the column top 43 and the rectification portion 42 into which is introduced a nitrogen-rich gas condensed in the nitrogen condenser 3 and/or a nitrogen-rich gas drawn from the column top 23 of the medium-pressure rectification column 2, after said nitrogen-rich gas has been cooled in the sub-cooler 8. A product nitrogen gas pipeline L43 is a pipeline for causing the nitrogen-rich gas drawn from the column top 43 of the low-pressure rectification column 4 to pass through the main heat exchanger 1, via the sub-cooler 8.

**[0046]** A gas drawn from a gas phase the nitrogen condenser 3 is introduced into the expansion turbine 92 after said gas has been passed through a part of the main heat exchanger 1. After being used in the expansion turbine 92, the gas is once again delivered to the main heat exchanger 1 from where it is drawn out as a waste gas.

**[0047]** The oxygen rectification column 5 has a column top 53 or a purification portion 52 into which the feed oxygen that has undergone heat exchange and liquefaction in the main heat exchanger 1 is introduced, following expansion in valve V. A feed oxygen pipeline L10 is a pipeline for introducing the feed oxygen, via the main heat exchanger 1, into the column top 53 or the rectification portion 52 of the oxygen rectification column 5. A second waste gas pipeline L53 is a pipeline for causing low-boiling-point component-containing oxygen gas, which is drawn from the column top 53 of the oxygen rectification column 5, to merge into the waste gas pipeline L31 downstream from the expansion turbine 92 and upstream from the main heat exchanger 1.

**[0048]** The oxygen vaporizer 6 is arranged below a bottom portion 51 of the oxygen rectification column 5 and vaporizes liquefied oxygen while using, as a heating medium, an oxygen-rich liquid drawn from the bottom portion 21 of the medium-pressure rectification column 2. The second oxygen-rich liquid pipeline L21b is a pipeline for introducing the oxygen-rich liquid drawn from the bottom portion 21 of the medium-pressure rectification column 2 into the oxygen vaporizers, from where it is introduced



into an intermediate stage of the rectification portion 42 of the low-pressure rectification column 4. An ultra-high-purity oxygen extraction pipeline L61 is a pipeline for extracting ultra-high-purity oxygen (liquid) from a vaporized liquid portion 61 of the oxygen vaporizer 6.

**[0049]** Thus the column 4 is fed by oxygen from the electrolyser and from the column 2

**[0050]** The oxygen vaporizer 6 is arranged below the oxygen rectification column 5 in order to supply a vapour stream to the oxygen rectification column 5. The oxygen vaporizer 6 vaporizes liquefied oxygen supplied from the bottom portion 51 of the oxygen rectification column 5 and supplies the vapour stream thereof to the bottom portion 51 of the oxygen rectification column 5. A portion of the feed oxygen is utilized as a heating medium. As another embodiment, a portion of the feed air supplied from the main heat exchanger 1, or a portion of an oxygen-containing liquid or liquefied nitrogen supplied from the medium-pressure rectification column 2 may be utilized.

**[0051]** The gas which is used as the heating medium may be liquefied and used as a reflux liquid in the low-pressure rectification column 4, or as a refrigerant in the main heat exchanger 1 or the sub-cooler 8. The liquid which is used as the heating medium is sub-cooled, and therefore vaporization loss during decompression is reduced.

**[0052]** The sub-cooler 8 performs heat exchange of: an oxygen-rich liquid drawn from the bottom portion 21 of the medium-pressure rectification column 2, a purified gas condensed in the nitrogen condenser 3 and/or a purified gas drawn from the column top 23 of the medium-pressure rectification column 2, and a nitrogen-rich gas drawn from the column top 43 of the low-pressure rectification column 4.

#### Embodiment 2

**[0053]** An ultra-high-purity oxygen production apparatus A2 according to embodiment 2 will be described with the aid of fig. 2.

**[0054]** The description of the ultra-high-purity oxygen production apparatus A2 will focus on features which are different from those of the ultra-high-purity oxygen production apparatus A1 of embodiment 1, and features which are the same will not be described, or will be described in simple terms. Reference symbols which are the same denote the same functions. The ultra-high-purity oxygen production apparatus A2 comprises an oxygen condenser 7 for condensing a low-boiling-point component-containing oxygen gas drawn from the column top 53 of the oxygen rectification column 5.

**[0055]** The second oxygen-rich liquid pipeline L21b is a pipeline for introducing the oxygen-rich liquid drawn from the bottom portion 21 of the medium-pressure rectification column 2 into the oxygen vaporizer 6, where heat is released from the oxygen-rich liquid and it is then introduced into a cold heat liquid portion 71 of the oxygen

condenser 7. A second circulation gas pipeline L71 is a pipeline for introducing, into the intermediate stage of the rectification portion 42 of the low-pressure rectification column 4, an oxygen-rich liquid drawn from the cold heat liquid portion 71 of the oxygen condenser 7. A third circulation gas pipeline L73 is a pipeline for introducing, into the intermediate stage of the rectification portion 42 of the low-pressure rectification column 4, a gas drawn from a column top 73 of the oxygen condenser 7. The oxygen condenser 7 is arranged above the oxygen rectification column 5 in order to improve a recovery rate of ultra-high-purity oxygen. This makes it possible to increase the amount of ultra-high-purity oxygen that can be recovered from the feed oxygen which is supplied, while maintaining the purity of the ultra-high-purity oxygen. An oxygen-containing liquid or liquefied nitrogen supplied from the medium-pressure rectification column 2 or the low-pressure rectification column 4, or liquefied feed air condensed in the oxygen vaporizer 6 may be utilized as a refrigerant in the oxygen condenser 7. Furthermore, liquefied nitrogen or liquefied air may also be supplied from the outside.

#### Embodiment 3

**[0056]** An ultra-high-purity oxygen production apparatus A3 according to embodiment 3 will be described with the aid of fig. 3.

**[0057]** The description of the ultra-high-purity oxygen production apparatus A3 will focus on features which are different from those of the ultra-high-purity oxygen production apparatus A2 of embodiment 2, and features which are the same will not be described, or will be described in simple terms. Reference symbols which are the same denote the same functions. The ultra-high-purity oxygen production apparatus A3 comprises a branch feed oxygen pipeline L11. The branch feed oxygen pipeline L11 branches the feed oxygen from partway through the main heat exchanger 1 in a feed oxygen pipeline L10, and merges into the first waste gas pipeline L31 before connection to the expansion turbine 92.

**[0058]** A portion of the feed high-pressure oxygen is drawn from partway through the main heat exchanger 1, and expanded by the expansion turbine 92 and cooled, after which it is once again supplied to the main heat exchanger 1, so as to maintain a heat balance in the main heat exchanger 1. This enables the cold heat required for liquefying the feed oxygen to be supplied to the main heat exchanger 1. If there is any surplus feed oxygen, the cold heat thereof may be utilized to contribute to maintaining a cold heat balance in the air separation unit or the nitrogen generating apparatus.

#### Embodiment 4

**[0059]** An ultra-high-purity oxygen production apparatus B1 according to embodiment 4 will be described with the aid of fig. 4.

**[0060]** The ultra-high-purity oxygen production appa-

ratus B1 comprises: a main heat exchanger 1, a nitrogen rectification column 2, a first nitrogen condenser 3, a second nitrogen condenser 30, an expansion turbine 92, a compressor 91, an oxygen rectification column 5, and an oxygen vaporizer 6. The difference with embodiments 1-3 lies in the single-column nitrogen rectification column, with two nitrogen condensers and a compressor for recycling gas being provided. The features which are different will mainly be described.

**[0061]** The nitrogen rectification column 2 comprises: a bottom portion 21 into which the feed air cooled in the main heat exchanger 1 is introduced, a rectification portion 22, and a column top 23.

**[0062]** The first nitrogen condenser 3 condenses a nitrogen-rich gas drawn from the column top 23 of the nitrogen rectification column 2. The second nitrogen condenser 30 condenses the nitrogen-rich gas drawn from the column top 23 of the nitrogen rectification column 2. A gas drawn from a gas phase in the first nitrogen condenser 3 is introduced into the expansion turbine 92 after said gas has been passed through a part of the main heat exchanger 1. The compressor 91 is connected to the expansion turbine 92 and compresses a gas drawn from a gas phase in the second nitrogen condenser 30. The oxygen vaporizer 6 is arranged below a bottom portion 51 of the oxygen rectification column 5 and vaporizes liquefied oxygen while using, as a heating medium, an oxygen-rich liquid drawn from the bottom portion 21 of the nitrogen rectification column 2.

**[0063]** A feed air pipeline L1 is a pipeline for introducing the feed air, via the main heat exchanger 1, into a gas phase in the bottom portion 21 of the nitrogen rectification column 2, or into a lower portion of a purification portion 22. A first oxygen-rich liquid pipeline L21a is a pipeline for introducing, into a cold heat liquid portion (not depicted) of the second nitrogen condenser 30, the oxygen-rich liquid drawn from the bottom portion 21 of the nitrogen rectification column 2. A first condensing pipeline L231 delivers a nitrogen-rich gas drawn from the column top 23 of the nitrogen rectification column 2 to the first nitrogen condenser 3 and returns same to the column top 23. A second condensing pipeline L232 delivers the nitrogen-rich gas drawn from the column top 23 of the nitrogen rectification column 2 to the second nitrogen condenser 30 and returns same to the column top 23. A product nitrogen gas pipeline L23 causes the nitrogen-rich gas drawn from the column top 23 of the nitrogen rectification column 2 to pass through the main heat exchanger 1, from where it is drawn as product nitrogen gas.

**[0064]** A first waste gas pipeline L31 is a pipeline for causing the gas, which is drawn from the gas phase 31 in the column top of the first nitrogen condenser 3, to pass through a part of the main heat exchanger 1, the gas then being used in the expansion turbine 92, and once again passed through the main heat exchanger 1. A recycled gas pipeline L301 is a pipeline for causing a gas drawn from a gas phase 301 in the column top of the second nitrogen condenser 30 to be compressed in the

compressor 91, then passed through a part of the main heat exchanger 1, from where it is introduced into a lower portion of the rectification portion 22 of the nitrogen rectification column 2.

**[0065]** A feed oxygen pipeline L10 is a pipeline for introducing the feed oxygen, via the main heat exchanger 1, into the column top 53 of the oxygen rectification column 5. A second waste gas pipeline L53 is a pipeline for causing low-boiling-point component-containing oxygen gas, which is drawn from the column top 53 of the oxygen rectification column 5, to merge into the waste gas pipeline L31. A second oxygen-rich liquid pipeline L21b is a pipeline for introducing the oxygen-rich liquid drawn from the bottom portion 21 of the nitrogen rectification column 2 into the oxygen vaporizer 6, from where it is introduced into the cold heat liquid portion (not depicted) of the second nitrogen condenser 30. An ultra-high-purity oxygen extraction pipeline L61 is a pipeline for extracting ultra-high-purity oxygen (liquid) from a vaporized liquid portion 61 of the oxygen vaporizer 6.

#### Embodiment 5

**[0066]** An ultra-high-purity oxygen production apparatus B2 according to embodiment 5 will be described with the aid of fig. 5. Embodiment 5 has the same basic configuration as embodiment 4. The difference lies in the feed oxygen pipeline L10 and the oxygen-containing liquid pipeline L22.

**[0067]** The feed oxygen pipeline L10 is a pipeline for introducing the feed oxygen, via the main heat exchanger 1, into an intermediate stage of the rectification portion 52 of the oxygen rectification column 5. The oxygen-containing liquid pipeline L22 is a pipeline for introducing, into the column top 53 of the oxygen rectification column 5, an oxygen-containing liquid drawn from an intermediate stage (a position above the feed air introduction pipeline L1) of the rectification portion 22 of the nitrogen rectification column 2.

**[0068]** That is to say, the feed oxygen is introduced into the intermediate stage of the oxygen rectification column 5, and the oxygen-containing liquid from the intermediate stage of the nitrogen rectification column 2 is supplied to the column top 53 of the oxygen rectification column 5. The oxygen-containing liquid is drawn from a stage of the nitrogen rectification column above the stage where the feed air is supplied, so that the oxygen-containing liquid does not contain any high-boiling point impurities derived from the atmosphere.

**[0069]** By virtue of this configuration, oxygen originating from the nitrogen rectification column can be purified into high-purity oxygen while at the same time a liquid for condensing the feed oxygen can be supplied to the oxygen rectification column, making it possible to produce high-purity oxygen while the operating rate of a water electrolysis apparatus and a nitrogen generating apparatus is optimized for high-purity oxygen demand.

**[0070]** For example, when there is low demand for hy-

drogen but high demand for high-purity oxygen, a short-fall in high-purity oxygen can be met by purifying the oxygen-containing liquid from the nitrogen rectification column in the oxygen rectification column, while the oxygen originating from the water electrolysis apparatus can be purified into high-purity oxygen. In this way, it is possible to optimize power consumption without the need for the water electrolysis apparatus, which consumes a large amount of power, to operate correspondingly with high-purity oxygen demand.

**[0071]** Thus the column 5 is fed simultaneously by oxygen from the electrolyser and from the column 2.

#### Examples

**[0072]** In the ultra-high-purity oxygen production apparatus A1 (air separation unit (nitrogen generating apparatus)) of embodiment 1, feed air at a flow rate of 1000 Nm<sup>3</sup>/h, a temperature of 20°C, and a pressure of 7.7 bar was introduced into the main heat exchanger 1 and cooled, then the feed air was introduced into the bottom portion 21 of the medium-pressure rectification column 2. The feed air was rectified in the column of the medium-pressure rectification column 2 operated at 7.5 bar, and liquid nitrogen was drawn from the column top 23 at 408 Nm<sup>3</sup>/h, with an oxygen-containing liquid (oxygen-rich liquid) being drawn from the column bottom portion 21 at 592 Nm<sup>3</sup>/h. The liquid nitrogen (nitrogen gas condensed in the condenser 3) drawn out through the pipe L231 and the oxygen-containing liquid (oxygen-rich liquid) drawn out through the pipe L21a were each cooled in the sub-cooler 8, after which the liquid nitrogen (nitrogen gas condensed in the condenser 3) was supplied to the top portion 43 of the low-pressure rectification column 4 operated at 2.5 barA. The oxygen-containing liquid (oxygen-rich liquid) was supplied to the intermediate portion of the low-pressure rectification column 4. The liquid nitrogen and the oxygen-containing liquid were rectified while undergoing heat and substance exchange with the vapour stream supplied from the nitrogen condenser 3, and nitrogen gas was drawn from the top portion 43 of the low-pressure rectification column 4 at 730 Nm<sup>3</sup>/h, and a waste gas was drawn from the bottom portion 31 at 270 Nm<sup>3</sup>/h.

**[0073]** Nitrogen gas drawn out through the pipe L43 was warmed in the sub-cooler 8 and then further warmed in the main heat exchanger 1, being drawn from the warm end of the main heat exchanger 1 at a temperature of 17.5°C and a pressure of 2.3 barA. The waste gas drawn out through the pipe L31 was warmed to -120°C in the main heat exchanger 1 then expanded by the expansion turbine 92 and cooled, after which the waste gas was once again supplied to the main heat exchanger 1, and drawn from the warm end of the main heat exchanger 1 at a temperature of 17.5°C, and a pressure of 1.15 barA.

**[0074]** The feed oxygen containing 1 ppm of argon as impurity was introduced into the main heat exchanger 1 at a flow rate of 30 Nm<sup>3</sup>/h, a temperature of 20°C, and a

pressure of 10 barA, cooled to -153.5°C and liquefied. The cooled liquefied oxygen was decompressed and then supplied to the top portion 53 of the oxygen rectification column 5 (NTP = 60) operated at 1.5 barA, where the cooled liquefied oxygen was rectified while undergoing heat and substance exchange with the vapour stream supplied from the oxygen vaporizer 6. The oxygen-containing liquid (oxygen-rich liquid) was supplied at 310 Nm<sup>3</sup>/h from the bottom portion 21 of the medium-pressure rectification column 2 as a heating medium for the oxygen vaporizers, and cooled, then supplied to the intermediate portion of the rectification portion 42 of the low-pressure rectification column 4. Ultra-high-purity oxygen liquid in which the content of low-boiling-point components (argon impurity) had been reduced to 10 ppb was obtained at 7.3 Nm<sup>3</sup>/h from the vaporized liquid portion 61 of the oxygen vaporizer 6 or the column bottom portion of the oxygen rectification column 5.

Other embodiments

#### [0075]

(1) Although not explicitly stated, pressure regulators and flow rate controllers, etc. may be installed in each pipeline in order to regulate pressure and regulate flow rate.

(2) Although not explicitly stated, control valves and gate valves, etc. may be installed in each line. (3)

Although not explicitly stated, pressure regulators and temperature measurement devices, etc. may be installed in each column in order to regulate pressure and regulate temperature.

Key to Symbols

#### [0076]

- 1 Heat exchanger
- 2 Medium-pressure rectification column
- 3 Nitrogen condenser
- 4 Low-pressure rectification column
- 5 Oxygen rectification column
- 6 Oxygen vaporizer
- 7 Oxygen condenser
- 8 Sub-cooler
- 91 Compressor
- 92 Expansion turbine

#### Claims

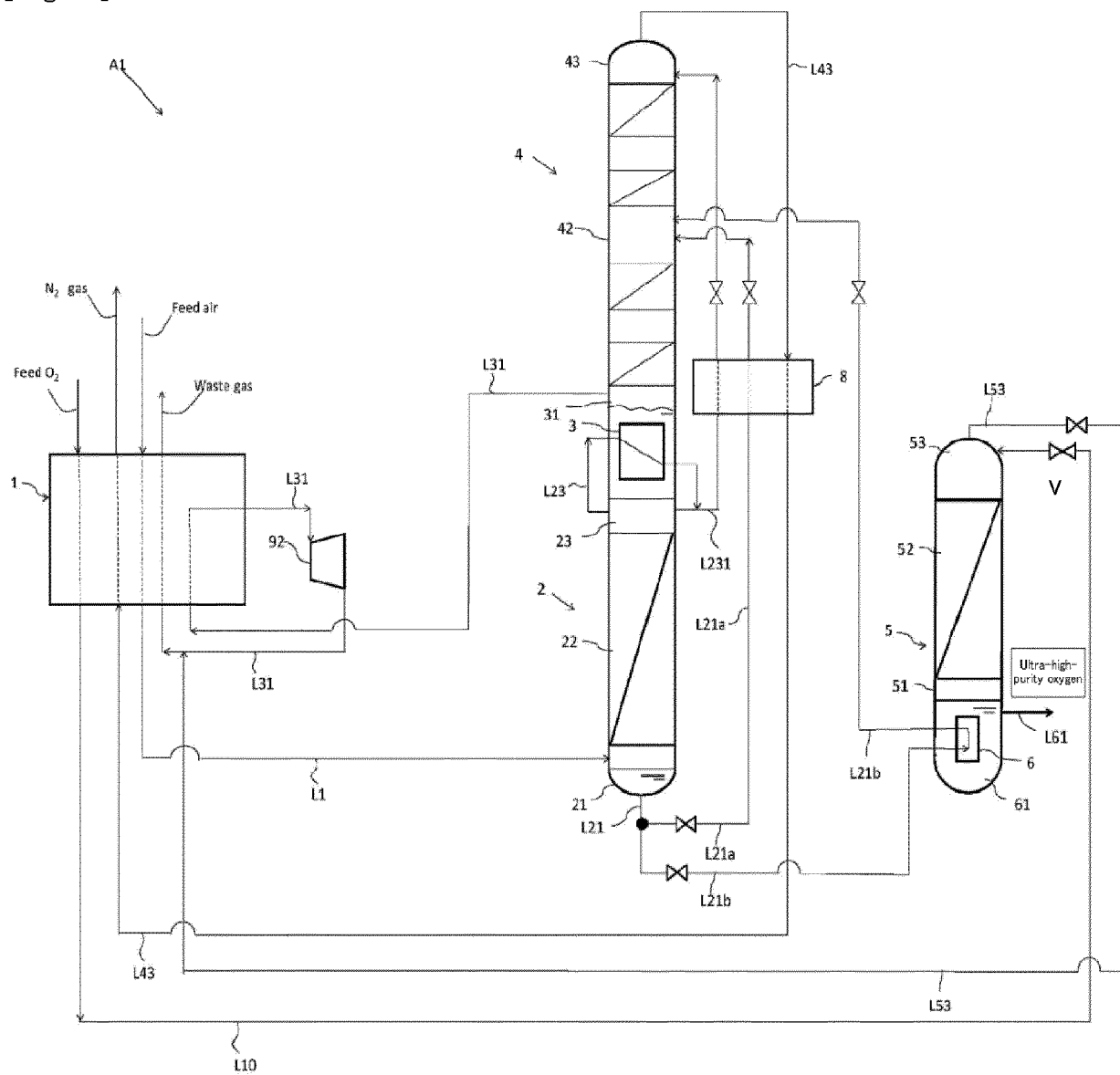
1. Ultra-high-purity oxygen production method utilizing an air separation unit comprising a main heat exchanger (1), a nitrogen rectification column (2), a nitrogen condenser (3), an oxygen rectification column (5), and an oxygen vaporizer (6), wherein the method comprises:

- a step in which feed oxygen comprising low-boiling-point components as impurities is introduced from a warm end of the main heat exchanger, cooled and at least partially liquefied (L10), then introduced into the oxygen rectification column, and ultra-high-purity oxygen (L61) from which the low-boiling-point components have been removed is drawn as a gas or a liquid from a lower portion of the oxygen rectification column or from the oxygen vaporizer; and
  - a step in which one or more of: a portion of feed air cooled in the main heat exchanger, a portion of the feed oxygen cooled in the main heat exchanger, and a liquid (L21b) or gas drawn from a medium-pressure rectification column constituting the nitrogen rectification column is utilized as a heating medium in the oxygen vaporizer, liquefied oxygen supplied from a bottom portion of the oxygen rectification column is vaporized, and a vapour stream thereof is supplied to the bottom portion of the oxygen rectification column.
2. Method according to Claim 1, further comprising a step in which liquefied nitrogen or an oxygen-containing liquid supplied from the medium-pressure rectification column, or liquid nitrogen or liquefied air supplied from outside the air separation unit is utilized as a refrigerant in an oxygen condenser (7) provided above or in a top portion of the oxygen rectification column (5), and a low-boiling-point component-containing oxygen stream supplied from the oxygen rectification column is liquefied and supplied to a top portion of the oxygen rectification column as a reflux liquid.
  3. Method according to Claim 1 or 2, further comprising a step in which at least a portion (L10, L11) of the feed oxygen cooled in the heat exchanger (1) is expanded to provide refrigeration.
  4. Method according to Claim 3, wherein a portion (L11) of the feed oxygen drawn from partway through the main heat exchanger (1) is expanded by an expansion turbine (92) and cooled, after which it is once again supplied to the main heat exchanger.
  5. Method according to Claim 3 wherein the feed oxygen (L10) is expanded in a valve (V) upstream of the oxygen rectification column (5).
  6. Ultra-high-purity oxygen production apparatus comprising: a main heat exchanger (1) into which feed air and feed oxygen are introduced;
    - a first rectification column (2) having a bottom portion into which the feed air, that has undergone heat exchange in the main heat exchanger, is introduced;
    - at least one nitrogen condenser (3) for condensing a nitrogen-rich gas drawn from a column top of the first rectification column;
    - a second rectification column (4) having a column top into which is introduced a nitrogen-rich gas condensed in the nitrogen condenser and/or a nitrogen-rich liquid drawn from the column top of the first rectification column, after said nitrogen-rich liquid has been cooled in a sub-cooler;
    - an oxygen rectification column (5) having a column top or a purification portion into which the feed oxygen that has undergone heat exchange in the main heat exchanger is introduced;
    - an oxygen vaporizer (6) which is arranged below a bottom portion of the oxygen rectification column or within the oxygen rectification column and vaporizes liquefied oxygen while using, as a heating medium, one or more of: a portion of feed air cooled in the main heat exchanger, a portion of the feed oxygen cooled in the main heat exchanger, and a liquid or gas drawn from a medium-pressure rectification column constituting the nitrogen rectification column; and
    - a sub-cooler (8) for performing heat exchange of: an oxygen-enriched liquid drawn from the bottom portion of the first rectification column, a purified gas condensed in the nitrogen condenser and/or a purified gas drawn from the column top of the first rectification column, and a nitrogen-rich gas drawn from the column top of the second rectification column.
  7. Ultra-high-purity oxygen production apparatus according to Claim 6, comprising an expansion turbine (9, 92) into which a gas drawn from a gas phase in the nitrogen condenser is introduced, after said gas has been passed through a part of the main heat exchanger.
  8. Ultra-high-purity oxygen production apparatus according to Claim 6 or 7, comprising an oxygen condenser (7) for condensing a low-boiling-point component-containing oxygen gas drawn from the column top of the oxygen rectification column.
  9. Ultra-high-purity oxygen production apparatus according to Claim 6, 7 or 8, comprising:
    - a feed oxygen pipeline for introducing the feed oxygen, via the main heat exchanger (1), into the column top or a rectification portion of the oxygen rectification column (5);
    - a branch feed oxygen pipeline (L11) which branches the feed oxygen from partway through the main heat exchanger in the feed oxygen pipeline, and merges into a pipeline before connection to the expansion turbine; and

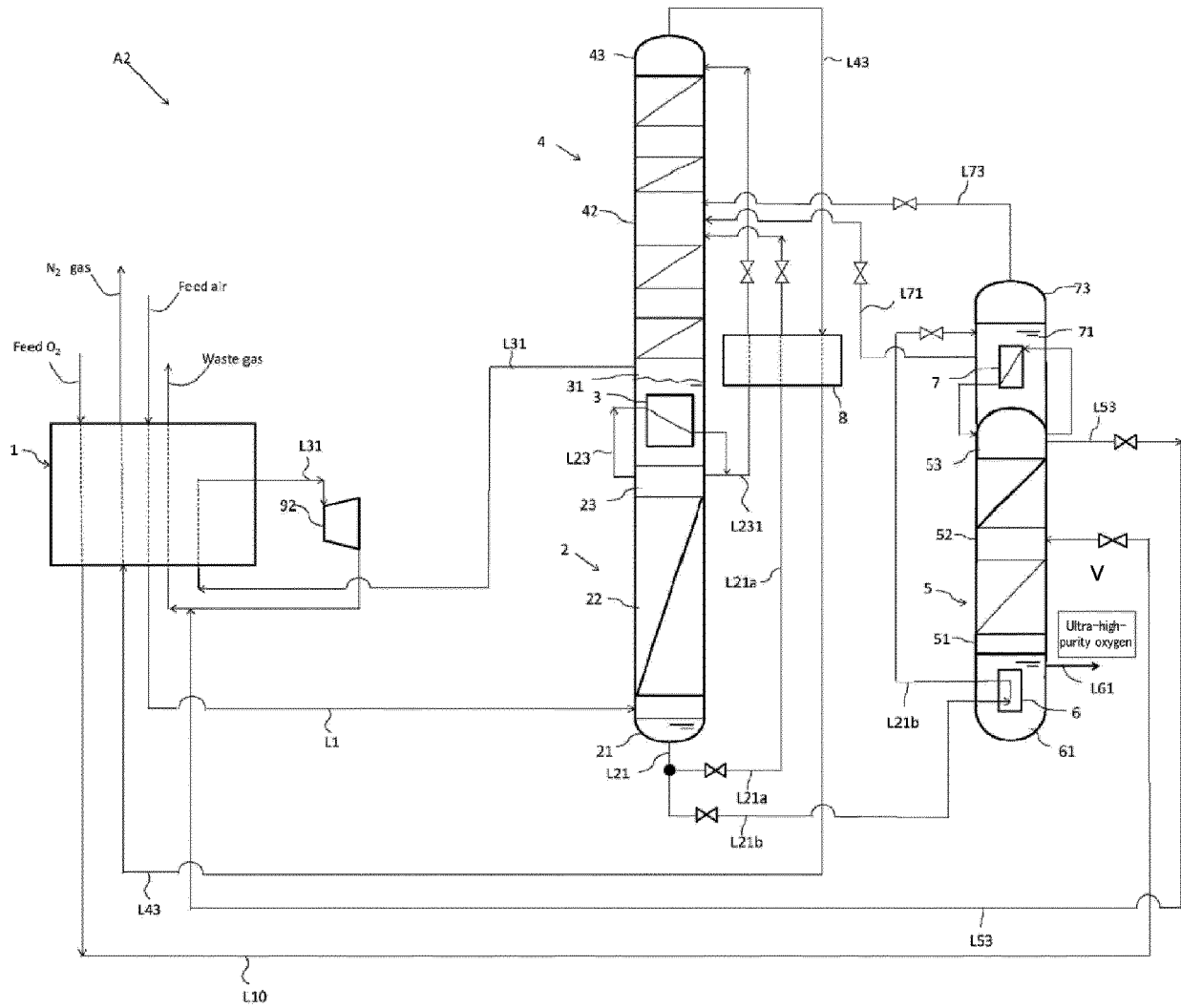
- an ultra-high-purity oxygen extraction pipeline for extracting ultra-high-purity oxygen from a vaporized liquid portion of the oxygen vaporizer.
10. Ultra-high-purity oxygen production apparatus comprising: a main heat exchanger (1) into which feed air and feed oxygen are introduced; 5
- a nitrogen rectification column (2) having a bottom portion into which the feed air, that has undergone heat exchange in the main heat exchanger, is introduced; 10
  - a first nitrogen condenser (3) for condensing a nitrogen-rich gas drawn from a column top of the nitrogen rectification column; 15
  - a second nitrogen condenser (30) for condensing the nitrogen-rich gas drawn from the column top of the nitrogen rectification column; 20
  - an expansion turbine (91) into which a gas drawn from a gas phase in the first nitrogen condenser is introduced, after said gas has been passed through a part of the main heat exchanger; 25
  - a compressor (92) for compressing a gas drawn from a gas phase in the second nitrogen condenser; 30
  - an oxygen rectification column (5) having a column top or a purification portion into which the feed oxygen that has undergone heat exchange in the main heat exchanger is introduced; and 35
  - an oxygen vaporizer (6) which is arranged below the bottom portion of the oxygen rectification column and vaporizes liquefied oxygen while using, as a heating medium, one or more of: a portion of feed air cooled in the main heat exchanger, a portion of the feed oxygen cooled in the main heat exchanger, and a liquid or gas drawn from the nitrogen rectification column.
11. Apparatus according to any of Claims 6 to 10 comprising expansion means (V, 92) for expanding at least part of the feed oxygen following cooling in the main heat exchanger to provide refrigeration for the apparatus. 40
12. Apparatus according to any of Claims 6 to 10 wherein the oxygen rectification column is connected so as to be fed only by the feed oxygen. 45
13. Apparatus according to any of Claims 6 to 10 wherein the oxygen rectification column is connected so as to be fed only by feed oxygen coming from an electrolyser. 50
14. Integrated apparatus including an electrolyser, means for sending water to the electrolyser, means for removing hydrogen from the electrolyser, means for removing an oxygen rich stream from the elec-

trolyser, an ultra high purity oxygen production apparatus according to any of Claims 6 to 13 and means for sending the oxygen rich stream from the electrolyser as the feed oxygen.

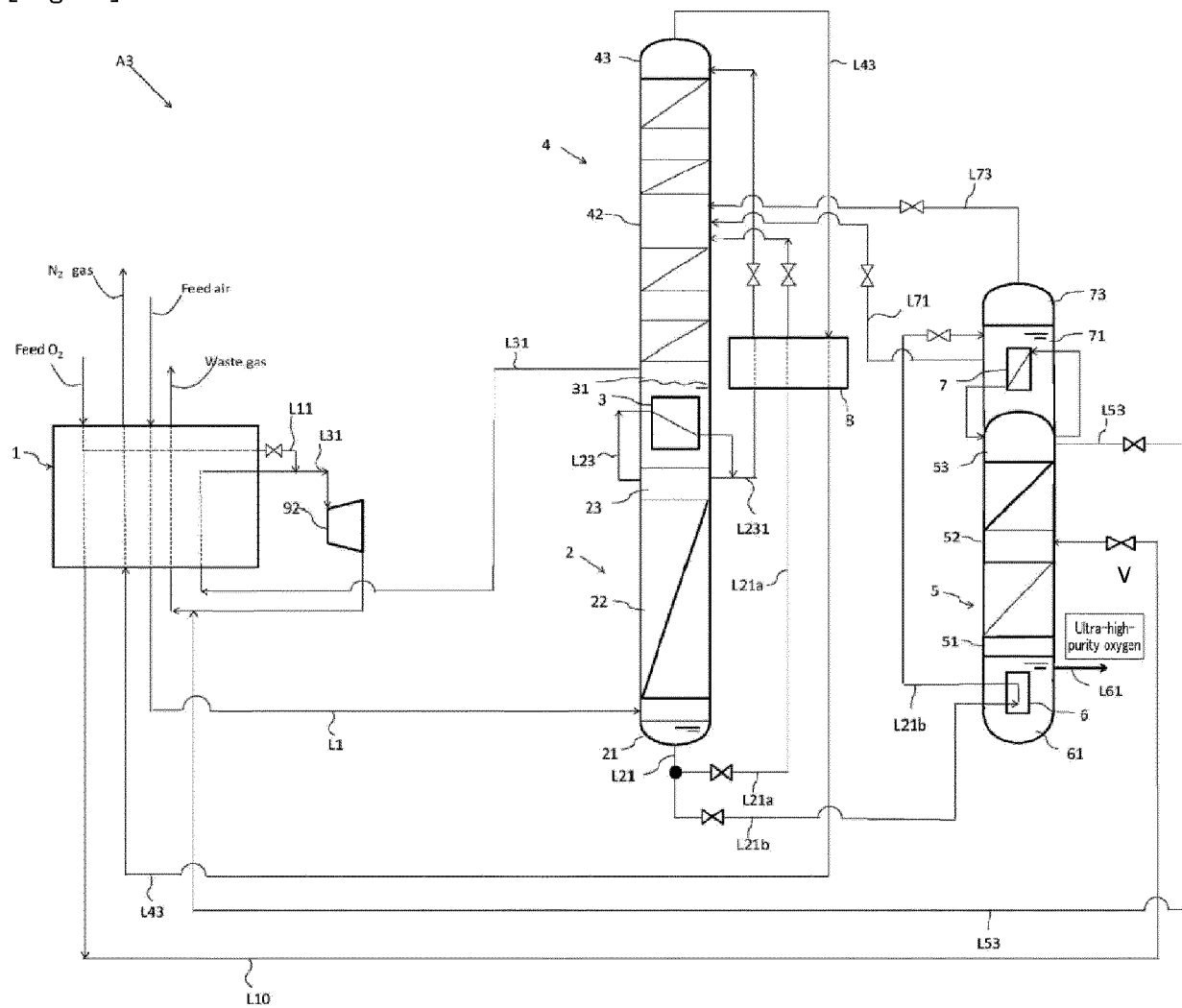
[Fig. 1]



[Fig. 2]

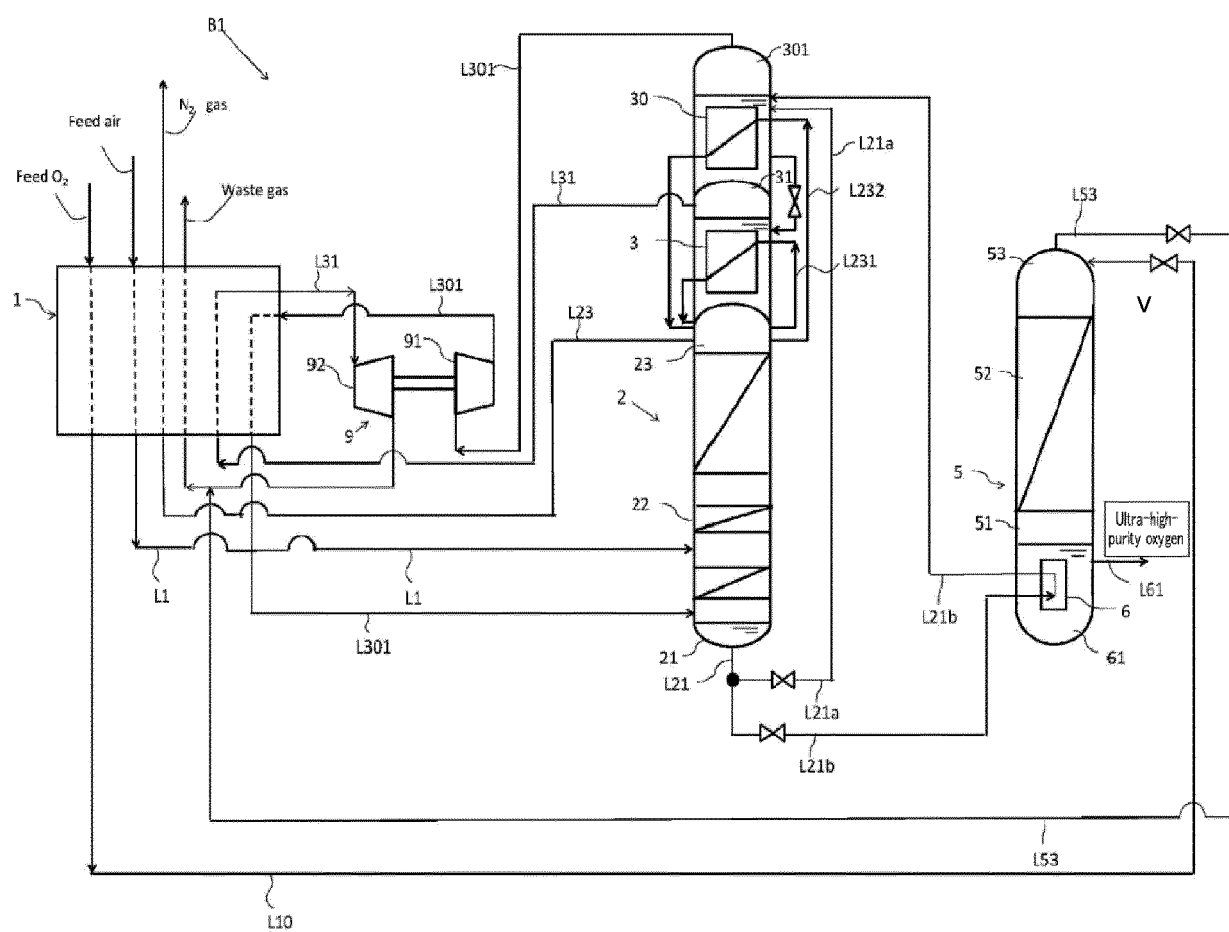


[Fig. 3]

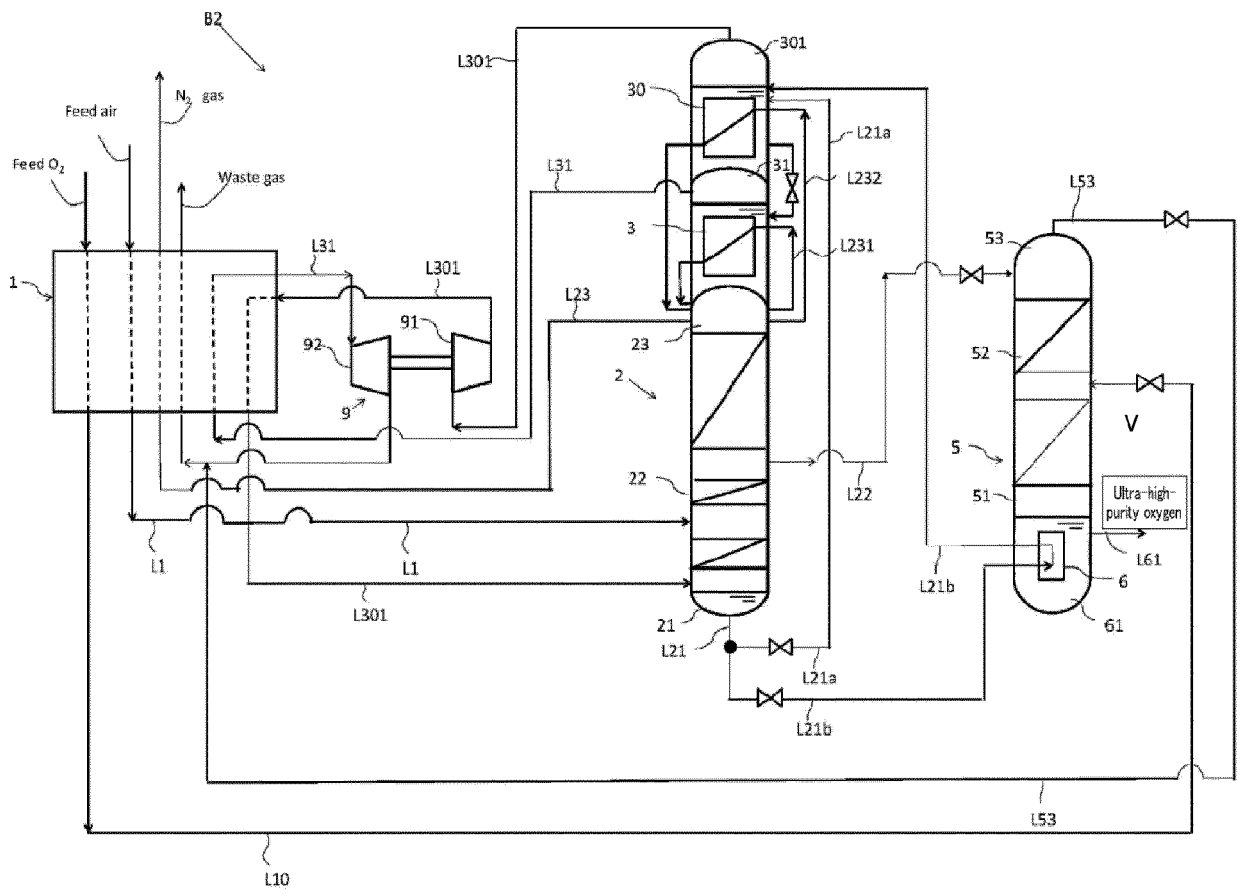




[Fig. 4]



[Fig. 5]



**REFERENCES CITED IN THE DESCRIPTION**

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**Patent documents cited in the description**

- JP 3929799 B **[0004]**
- JP 2021055890 A **[0004]** **[0007]**
- US 5049173 A **[0005]**
- WO 2014173496 A2 **[0005]**
- CN 114017993 A **[0009]**
- WO 2023274574 A **[0010]**