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Process for removing hydrogen by cryogenic distillation in the production of high purity nitrogen.

A process for removing hydrogen by cryogenic distillation in the production of high purity nitrogen, which comprises :

a) feeding a compressed, cleaned and dried feed mixture (12) comprising oxygen and nitrogen, which has been cooled to about the dew point thereof, to a first distillation column 13, whereby said nitrogen 16 is extracted at the top of said distillation column as a liquid, and a liquid stream rich in oxygen 17 is extracted at the bottom of said first distillation column ;

b) extracting a minor fraction of the gas 30 at the top of the first distillation column, the minor fraction containing the lighter impurities ;

c) expanding said liquid nitrogen at the top of the first distillation column into a second distillation column 14 at an intermediate level, said second distillation column being operated at a pressure sufficiently lower than the pressure of said first distillation column to provide a sufficient temperature difference in a condenser-reboiler located between the first and second distillation columns ;

d) vaporizing the liquid stream rich in oxygen in the overhead condenser 4 of the second distillation column 14 against the condensing vapor at the top of the second distillation column to form a condensate at the top of the second distillation column, and returning said condensate to the top of the second distillation column as reflux ;

e) extracting a minor fraction of the gas 31 at the top of the second distillation column containing substantially all lighter impurities ; and

f) vaporizing the liquid at the bottom of the second distillation column 14 by heat exchange with the condensing gas at the top of the first distillation column 13, and recovering as product a fraction of the liquid 24, a fraction of the vaporized liquid 29 or both, the product containing substantially no lighter air impurities.

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention relates to a process for removing hydrogen by cryogenic distillation in the production of high purity nitrogen.

Description of the Background

In producing nitrogen by cryogenic distillation of atmospheric air or any mixture comprising oxygen and nitrogen, hydrogen contained in atmospheric air is concentrated into the nitrogen product. Hydrogen contamination is undesirable particularly in electronic applications where very pure nitrogen is required.

At present, contaminate hydrogen in nitrogen is removed by passing compressed atmospheric feed air through a catalytic bed at a temperature of about 250 to 500°F, whereby the hydrogen reacts with oxygen to form water and carbon dioxide which are then removed in a subsequent step either by adsorption or by reversing exchangers. Removal of hydrogen by this method is undesirable, however, due to the expense of the catalyst and the possible poisoning of the catalyst by other impurities present in air, such as, for example, sulfur-containing compounds. Furthermore, the catalytic reactor and the equipment associated therewith are very expensive and represent a significant portion of the total required expense.

SUMMARY OF THE INVENTION

Accordingly, it is an object of the present invention to provide a process for removing hydrogen by cryogenic distillation in the production of high purity nitrogen.

It is also an object of the present invention to provide a process for removing hydrogen by cryogenic distillation in the production of high purity nitrogen which avoids the use of a catalytic reactor.

It is further an object of this application to provide a process for removing light impurities from a mixture mainly containing a light product, one or more heavier components and a trace of one or more lighter impurities.

The above object and others which will become more apparent in view of the following disclosure are provided, in part, by a process for removing hydrogen by cryogenic distillation in the production of high purity nitrogen, which entails:

- a) feeding a compressed, cleaned and dried feed mixture comprising oxygen and nitrogen, which has been cooled to about the dew point thereof, to the bottom of a first distillation column, whereby said nitrogen is extracted at the top of said distillation column as a liquid, and a liquid stream rich in oxygen collects at the bottom of said first

distillation column;

b) extracting a minor fraction of the gas at the top of the first distillation column, the minor fraction containing the lighter impurities;

c) expanding the liquid nitrogen at the top of the first distillation column into a second distillation column at an intermediate level, the second distillation column being operated at a pressure which is sufficiently lower than the pressure of the first distillation column, to provide a sufficient temperature difference in a condenser-reboiler located between the first and second distillation columns;

d) expanding the liquid stream rich in oxygen in the overhead condenser of the second distillation column to form a condensate at the top of the second distillation column, and returning the condensate to the top of the second distillation column as reflux,

e) extracting a minor fraction of the gas at the top of the second distillation column containing substantially all remaining lighter impurities components, and

f) vaporizing the liquid at the bottom of the second distillation column by heat exchange with the condensing gas at the top of the first distillation column, and recovering as product a fraction of the liquid, a fraction of the vaporized liquid or both, the product containing substantially no lighter impurities.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 represents a flow sheet for hydrogen removal by cryogenic distillation in the production of high purity nitrogen, where liquid product is extracted at the bottom of the second column as well as the gaseous product.

Figure 2 represents a flow sheet for hydrogen removal by cryogenic distillation in the production of high purity nitrogen, where nitrogen recovery is enhanced with a nitrogen cycle. Oxygen-rich liquid in the bottom of the first column is vaporized by indirect heat exchange with a compressed nitrogen cycle which is condensed and expanded at the top of the first column to increase its reflux and reboil.

Figure 3 represents a flow sheet for hydrogen removal by cryogenic distillation in the production of high purity nitrogen, where the refrigeration requirement is achieved by expanding a fraction of compressed air in a turbine before being fed to the first distillation column. The nitrogen recovery is enhanced by condensing an air stream in the bottom reboiler of the first distillation column.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

In accordance with the present invention, a method is provided for removing hydrogen by cryogenic distillation in the production of high purity nitrogen without using a catalytic reactor containing a catalyst. In essence, the present invention provides a double column process wherein a first distillation column is used to extract a first fraction of nitrogen product in liquid form. A very small amount of hydrogen is present in this liquid due to the high relative volatility of hydrogen as compared to nitrogen. Then, this liquid nitrogen is fed to a second distillation column, where it is further purified to yield a high purity nitrogen product at the bottom of the distillation column.

In more detail, the present invention provides a process for removing hydrogen by cryogenic distillation in the production of high purity nitrogen, which entails several steps.

First, compressed air or a feed mixture comprising oxygen and nitrogen which is substantially free of water and carbon dioxide and which has been cooled to about the dew point thereof is fed to the bottom of a first distillation column which is operated at a pressure such that heavy air components are separated from nitrogen. Thereby, nitrogen is produced at the top of the distillation column as a liquid, and a liquid stream rich in oxygen is produced at the bottom of the distillation column.

In accordance with the present invention, it is preferred that the first distillation column be operated at a pressure of about 4 to 12 bar in order to effectively separate the heavy air components, such as oxygen and argon, from nitrogen. This air normally contains up to about 20 vpm of hydrogen. As already noted, the first distillation column produces at the top a liquid product rich in nitrogen.

As used in the present specification, the term "heavy air components" refers to all components of air which have a lower volatility than nitrogen, i.e. its vapor pressure is lower than the vapor pressure of nitrogen at the same temperature. Similarly, the term "light air components" as used in the present specification is intended to include all components of air which have a higher volatility than nitrogen, i.e. its vapor pressure is greater than the vapor pressure of nitrogen at the same temperature. For example, oxygen and argon are examples of heavy air components, and hydrogen and helium are examples of light air components.

Then, the liquid nitrogen is expanded at the top of the first distillation column into a second distillation column at an intermediate level, and the second distillation column is operated at a pressure sufficiently lower than the pressure of the first distillation column to provide a sufficient temperature difference in the condenser-reboiler located between the two col-

umns.

Thereafter, the liquid stream rich in oxygen is vaporized in the overhead condenser of the second distillation column to form a condensate of a major fraction of the gas at the top of the second distillation column, and then the condensate is returned to the top of the second column as reflux.

Finally, a minor fraction of the gas at the top of the second distillation column is extracted, which contains substantially all the light air components, and the liquid at the bottom of the second distillation column is vaporized by heat exchange with the condensing gas at the top of the first distillation column, and a fraction of this vaporized liquid containing substantially no light air components is recovered as product.

Generally, the second distillation column may be operated at any pressure lower than the pressure of the first distillation column to provide a sufficient temperature difference in the condenser-reboiler separating the two columns. It is preferred, however, that the second distillation column be at a pressure at least about 0.4 bars lower than the pressure of the first distillation column. It is even more preferred, however, if the second distillation column is at a pressure about 0.6 bar lower than the pressure of the first distillation column.

Moreover, as used in the present specification, the phrase containing "substantially all light air components" means that at least 99.99% of all light air components are contained therein. Also, as used in the present specification, the phrase containing "substantially no light air components" means that no more than 0.01% of all light air components are contained therein.

The process of the present invention may be practiced with a number of variations, some of which will now be discussed.

First, in addition to the general description provided above, liquid product may also be extracted at the bottom of the second distillation column as well as the gaseous product. This is represented in Figures 2 and 3.

Alternatively, a subcooler can be added to subcool the bottom liquid of the first distillation column against the outgoing gaseous product and the residual stream rich in oxygen. This is represented in Figure 2.

Furthermore, the refrigeration requirement can be achieved by expanding the stream rich in oxygen, or expanding the gaseous nitrogen product or by adding liquid to the process in a liquid assist or by expanding a fraction of the feed air.

The present invention may be used in conjunction with other processes whenever the removal of a light product from a mixture containing heavier components is required.

The present invention may also be used advan-

tageously in conjunction with any process where light products are to be removed from mixtures of heavier components. As a specific example, the present process may be used to remove carbon monoxide from mixtures also containing heavier hydrocarbons, such as methane (CH₄). Generally, the process is applicable to several hydrocarbon mixtures containing lighter impurities. The present invention may be used with any of these to effect removal of the light component.

Beyond petrochemical processes, there are many other types of reaction mixtures which contain a lighter component and one or more heavy components. In some of the processes, the light component in the reaction mixture may be an unreacted raw material. In other processes, the lighter component in the reaction mixture may be one of the reaction products. The present process may be used to advantage with any of these processes to remove light components.

Generally, it is preferred, however, to use the present process in conjunction with processes producing a light product from a mixture containing mainly the light product, one or more heavier components and with traces of a lighter impurity. It is more preferred if the lighter impurity is present in the mixture only in an amount of up to about 1% by volume, most preferably only up to about 0.5% by volume.

Thus, in accordance with another aspect of the present invention, a process is provided for producing or removing a light or more volatile product from a mixture mainly containing the light or more volatile product and heavier or less volatile components and traces of lighter impurities which are lighter or have a higher volatility than the light product.

In more detail, the present invention provides a process for removing a light product from a mixture mainly comprising a light product, one or more heavier components and a trace of one or more lighter impurities by cryogenic distillation, which comprises:

- a) feeding said mixture mainly comprising the light product, the one or more heavier components and the trace of one or more lighter impurities to a first distillation column such that the one or more heavier components are separated from the light product containing the lighter impurities, whereby the light product extracted at the top of the distillation column as a liquid and a liquid stream rich in the one or more heavier components is extracted at the bottom of the first distillation column, and wherein the lighter impurities accumulate at the top of the first column, wherein a portion of the lighter impurities are soluble in the light product liquid, and a portion of the lighter impurities remain in a non-condensable vapor fraction stream, the non-condensable vapor fraction stream being removed from the column along with the lighter impurities contained therein;
- b) expanding the light product containing some

lighter impurities of the first distillation column into a second distillation column at an intermediate level, to produce a light product stream and a minor gaseous fraction containing a greater portion of the remaining lighter impurities;

c) expanding the liquid stream rich in the one or more heavier components extracted from the bottom of the first column into the overhead condenser of the second distillation column, where it is vaporized against the condensing gas stream at the top of the second distillation column, this condensate being returned to the top of the second distillation column as reflux;

d) extracting said minor fraction of the gas at the top of the second distillation column; and

e) vaporizing the liquid at the bottom of the second distillation column by heat exchange with the condensing gas at the top of the first distillation column, and recovering as product a fraction of this vaporized liquid containing substantially no light product.

Notably, with regard to step a) recited above, the lighter impurities are accumulated at the top of the first column. Some of the lighter impurities are soluble in the light-product liquid, and some of the lighter impurities remain in a vapor fraction called the non-condensable stream. This stream is removed from the column along with the lighter impurities contained therein.

As used herein, in step b) above, for example, the term "the greater portion of" refers to any portion greater than about 50% by volume. However, it is preferred that the greater portion be in excess of about 80%, even more preferably in excess of about 99% by volume.

Thus, the present invention provides an efficient means for separating a light product, one or more heavier components and one or more lighter impurities. In accordance with this aspect of the present invention, the feed mixture mainly contains the light product and one or more heavier components. Generally, the feed mixture contains from 50 to 99.99% by volume of light product and one or more heavier components. It is preferred, however, if the feed mixture contains from 75 to 99.99% by volume of light product and one or more heavier components.

As noted above, generally, the one or more lighter impurities are present in an amount of up to about 1% by volume, preferably not more than about 0.5% by volume.

Generally, as used herein the term "light product" means the mixture component having the higher volatility. The term "heavier components" means the mixture component or components having the lowest volatility. The term "lighter impurities" means the impurity component or components having an intermediate volatility and which are present in amounts of only up to about 1% by volume.

In accordance with the present invention the "trace" of lighter impurities is intended to mean a minor amount of generally less than 1% by volume. Also, the term "non-condensable" is intended to mean non-condensable under conditions prevailing outlet for the top condenser of both columns.

In order to more fully describe the present invention, reference will now be made to Figures 1-3.

In Figure 1, a cooled, compressed, cleaned and dried feed stream containing light product, one or more heavier components and a trace of one or more lighter impurities, such as atmospheric air, is fed via conduit (10) to heat exchange means (11), and then to the high pressure column (13) via conduit (12). A nitrogen-rich liquid is fed from the high pressure column (13) to the low pressure column (14) via conduit (16) for feed. Also, liquid nitrogen (LIN) may be removed from the column as liquid product from the bottom of the low pressure column (14).

Non-condensable material is withdrawn from the column at condenser-reboiler (15), and from the overhead condenser (80) of the low pressure column.

Waste gas is removed from the overhead condenser (80) via conduits (21) and (22), optionally through subcooler (18), to heat exchange means (11), where it exits the process via conduit (28). This waste originates from a bottom stream (17) withdrawn from the high pressure column, wherein after it is optionally passed through subcooler (18) and to the overhead condenser (80) via conduit (20).

In Figure 2, a cooled, compressed, cleaned and dried feed stream containing light product, one or more heavier components and a trace of one or more lighter impurities, such as atmospheric air, and which is close to the dew point is fed to an intermediate location of a high pressure column (13), wherein an oxygen-rich stream separates at the bottom and a nitrogen-rich stream at the top. A liquid nitrogen stream is extracted at the top of the high pressure column (13) and fed to the low pressure column (14) via conduit (16) at an intermediate location. A minor gaseous fraction or non-condensable containing some lighter impurities is removed at the top of the high pressure columns (13) via conduit (30).

Then, lighter impurities are removed via the non-condensable stream at the top of the low pressure column (14) with the bottom fraction being substantially free, i.e., less than about 0.5% by volume thereof, of lighter impurities.

Nitrogen product can be extracted from the bottom of the low pressure column as a liquid (LIN) via conduit (24). Gaseous nitrogen product is extracted from the column via conduit (19) and rewarmed in exchanger (11). A portion of this product is recovered via conduit (54) and the remaining portion is compressed in compressor (60). A fraction of this compressed stream may be recovered as product via conduit (57). The remaining fraction is sent to the high pressure

column reboiler (52) via conduit (51) where it condenses to provide the reboil for the high pressure column.

Then, the condensed recycle stream is fed via conduit (53) from the reboiler to the top of the high pressure column to provide extra reflux for the high pressure column.

An oxygen-rich stream is passed from the bottom of the high pressure column (13) via conduit (17) to the overhead condenser (80) of the low pressure column, whereby it vaporizes and passes to the exchanger (11), and is rewarmed. The rewarmed stream is then fed to an expander and then to an exchanger where it is used to provide required refrigeration and then exits as waste.

In Figure 3, fraction of a cooled, compressed, cleaned and dried feed stream is fed via conduit (73) to the bottom reboiler (52) of the distillation column (13) where it is liquified, the liquified feed stream is then fed to the high pressure column (13). Another fraction of the cooled, compressed, cleaned and dried feed stream is expanded via expander (71) into the high pressure column (13), wherein in the top section thereof pure nitrogen and lighter impurities are extracted and in the bottom section oxygen-rich liquid is extracted. Some lighter impurities may be removed via conduit (30).

Liquid nitrogen fraction is extracted at the top of the high pressure column and fed via conduit (16) to an intermediate stage of the low pressure column. The gaseous nitrogen fraction which forms at the top of the high pressure column is condensed in reboiler (15) to provide reboil for the low pressure column.

The low pressure column (14) further purifies liquid nitrogen feed and a liquid product may be recovered at the bottom of the low pressure column via conduit (24), which is free of lighter impurities. Conduit (19) affords recovery of gaseous nitrogen which is free of lighter impurities.

The remaining lighter impurities are removed via conduit (31) and exit at the top of the low pressure column.

Oxygen-rich liquid from the bottom of the high pressure column is transferred via conduit (17) to the top condenser (80) of the low pressure column, where it is vaporized and leaves the process via conduit (21), (22) and (28), optionally passing through subcooler (10), and then passing through exchanger (11)

Gaseous nitrogen exits the column via conduit (19), optionally passing through the subcooler (18), through conduit (23) and then passing through exchanger (11), wherein after it exits the process via conduit (29).

By vaporizing the oxygen-rich liquid, some of the gaseous nitrogen at the top of the low pressure column is condensed and returned as reflux in the low pressure column.

Thus, in accordance with the present invention

both processes and apparatus as described above are provided.

Having described the present invention, it will now be apparent to one skilled in the art that the many changes and modifications may be made to the above-described embodiments without departing from the spirit and the scope of the present invention.

Claims

1. A process for removing hydrogen by cryogenic distillation in the production of high purity nitrogen, which comprises:

a) feeding a compressed, cleaned and dried feed mixture comprising oxygen and nitrogen, which has been cooled to about the dew point thereof, to a first distillation column, whereby said nitrogen is extracted at the top of said distillation column as a liquid, and a liquid stream rich in oxygen is extracted at the bottom of said first distillation column;

b) extracting a minor fraction of the gas at the top of the first distillation column, the minor fraction containing part of the lighter impurities;

c) feeding said liquid nitrogen from the top of the first distillation column into a second distillation column at an intermediate level, said second distillation column being operated at a pressure sufficiently lower than the pressure of said first distillation column to provide a sufficient temperature difference in a condenser-reboiler located between the first and second distillation columns;

d) vaporizing the liquid stream rich in oxygen in the overhead condenser of the second distillation column against the condensing vapor at the top of the second distillation column to form a condensate at the top of the second distillation column, and returning said condensate to the top of the second distillation column as reflux;

e) extracting a minor fraction of the gas at the top of the second distillation column containing substantially all remaining lighter impurities; and

f) vaporizing the liquid at the bottom of the second distillation column by heat exchange with the condensing gas at the top of the first distillation column, and recovering as product a fraction of the liquid, a fraction of the vaporized liquid or both, the product containing substantially no lighter impurities.

2. The process of Claim 1, wherein said second distillation column is operated at a pressure at least 0.4 bar below the pressure of said first distillation

column.

3. The process according to one of Claims 1 or 2, wherein said first distillation column is operated at a pressure of about 4 to 12 bar.

4. The process according to one of Claims 1 to 3, wherein said second distillation column is operated at a pressure of about 0.6 bar below the pressure of said first distillation column.

5. A process for removing a light product from a mixture mainly comprising a light product, one or more heavier components and a trace of one or more lighter impurities which comprises:

a) feeding said mixture mainly comprising the light product, the one or more heavier components and the trace of one or more lighter impurities to a first distillation column such that the one or more heavier components are separated from the light product containing the lighter impurities, whereby the light product is extracted at the top of the distillation column as a liquid and a liquid stream rich in the one or more heavier components collect at the bottom of the first distillation column; and wherein the lighter impurities accumulate at the top of the first column, wherein a portion of the lighter impurities are soluble in the light-product liquid, and a portion of the lighter impurities remain in a non-condensable vapor fraction stream, said non-condensable vapor fraction stream being removed from the column along with the lighter impurities contained therein;

b) expanding the light product containing some lighter impurities of the first distillation column into a second distillation column at an intermediate level to produce a light product stream and a minor gaseous fraction containing a greater portion of the remaining lighter impurities;

c) expanding the liquid stream rich in the one or more heavier components extracted from the bottom of the first column into the overhead condenser of the second distillation column, where it is vaporized against the condensing gas stream at the top of the second distillation column, the condensate being returned to the top of the second distillation column as reflux;

d) extracting said minor fraction of the gas at the top of the second distillation column; and e) vaporizing the liquid at the bottom of the second distillation column by heat exchange with the condensing gas at the top of the first distillation column, and recovering as product a fraction of the liquid, a fraction of the vaporized

rized liquid or both, the product containing substantially no lighter impurities.

6. An apparatus for removing hydrogen by cryogenic distillation in the production of high-purity nitrogen, which comprises a double fractionating means having a high pressure fractionating means in fluid connection with a feed stream comprising oxygen and nitrogen which extracts a first fraction of nitrogen production liquid from, separate conduit means for feeding said liquid nitrogen product from said high pressure fractionating means to a low pressure fractionating means for further purification.
7. The apparatus of Claim 6, which further comprises a compressed nitrogen cycle in fluid connection with a bottom reboiler of the high pressure fractionating means, whereby oxygen-rich liquid in the bottom of the first column in contact with said reboiler is vaporized by indirect heat exchange, said compressed nitrogen cycle stream being condensed and expanded at the top of the first column to increase its reflux and reboil.
8. The apparatus according to one of Claims 6 or 7, which further comprises a turbine in fluid connection with said first distillation column, whereby required refrigeration is achieved by expanding a fraction of compressed air in said turbine before being fed to the first distillation column.
9. The apparatus of Claim 8, which further comprises a bottom reboiler of the high pressure fractionating means whereby the compressed feed stream is condensed against a vaporizing oxygen-rich liquid by indirect heat exchange, said condensed feed stream then being fed to the high pressure fractionating means.

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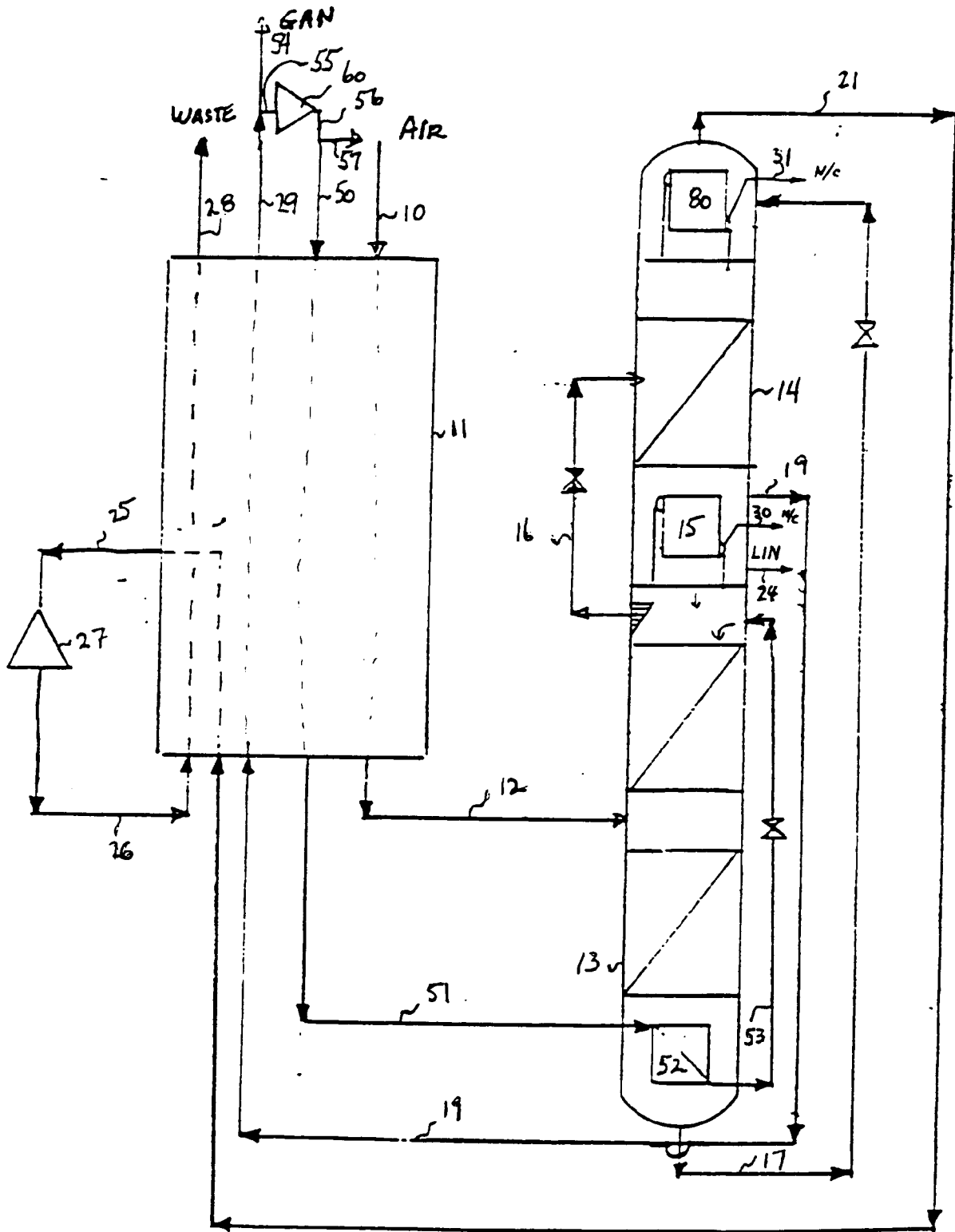


FIGURE 2

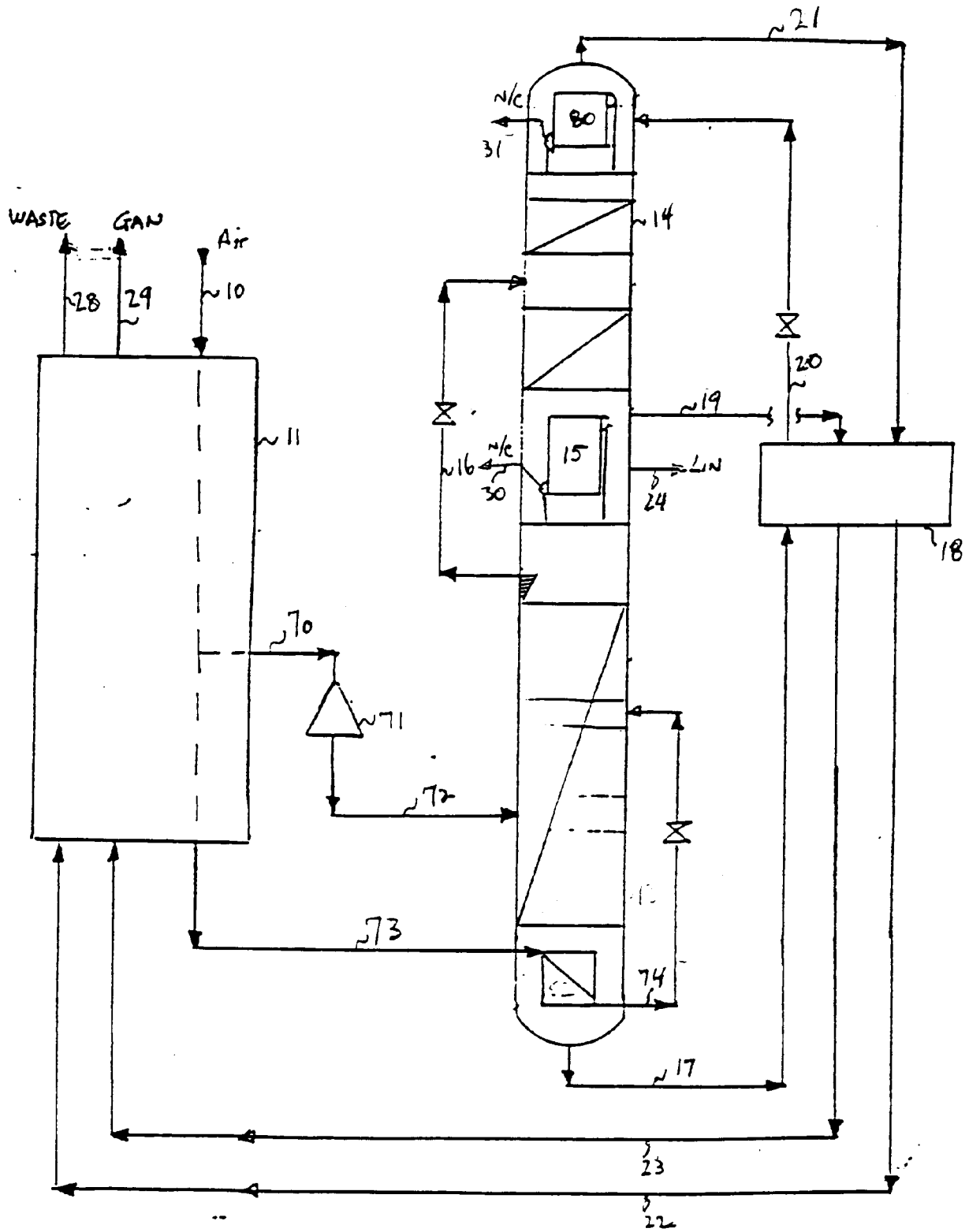


FIGURE 3



European Patent
Office

EUROPEAN SEARCH REPORT

Application Number

EP 92 40 2799

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
P,X	US-A-5 123 947 (AIR PRODUCTS AND CHEMICALS) 23 June 1992 * abstract * * figure 1 * * column 2, line 37 - column 3, line 10 * * column 3, line 42 - column 5, line 34 * ---	1-6	F25J3/04
P,X	EP-A-0 485 612 (K.K.KOBE SEIKO SHO) 12 December 1991 * abstract * * page 3, line 29 - page 5, line 9 * * figure 1 * & WO-A-91 19 142 ---	1,2,4-6	
A	US-A-4 902 321 (UNION CARBIDE CORPORATION) * abstract * * figure 1 * * column 2, line 40 - column 3, line 38 * -----	1,5	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			F25J
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 17 DECEMBER 1992	Examiner SIEM T.D.
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</p>			

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