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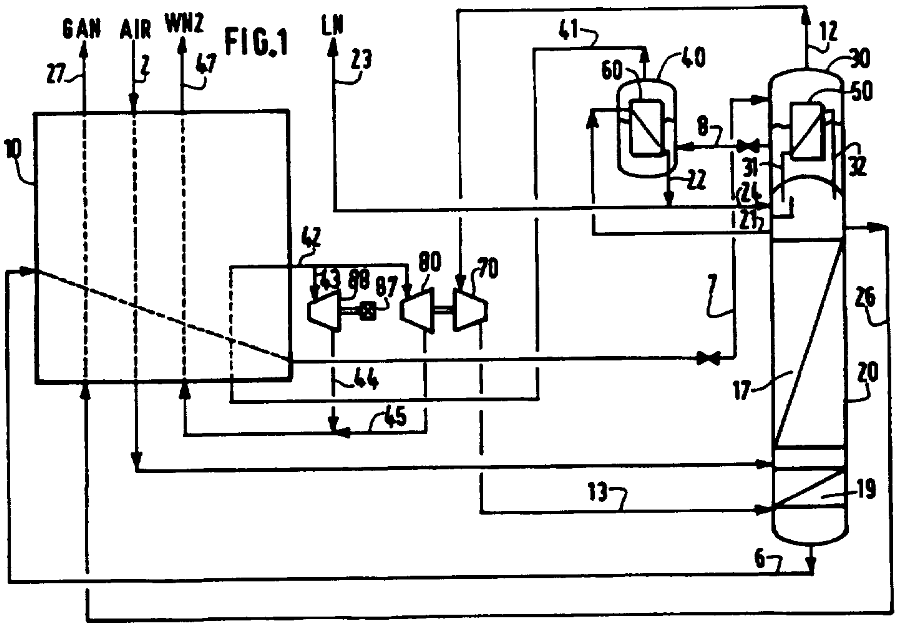

(57) Abstract

A process and apparatus are disclosed for highly efficient generation of nitrogen product in a single column arrangement. Oxigen-enriched liquid from a distillation column (20) is partially vaporized to form a liquid and a vapor phase. The liquid is vaporized in a second reboiler/condenser (60) and thereafter the vapor may be expanded to provide process refrigeration. The vapor portion having higher nitrogen content is compressed and returned to the distillation column separately from the air feed for higher overall nitrogen recovery.

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High efficiency nitrogen generator

The present invention is directed to a highly efficient process and apparatus for the production of nitrogen from air by cryogenic distillation.

Numerous processes for the generation of nitrogen from air are known in the art. Where the primary product is nitrogen, single column processes for the separation of air at cryogenic conditions utilizing an oxygen-enriched stream for expansion and refrigeration for the process are well known.

A basic process and apparatus for the generation of nitrogen using waste oxygen-enriched stream expansion is described in US-A- 4,883,519. In this document, a process and an apparatus according to the pre-characterizing portions of the independent claims are described.

In US-A-4,883,519, the nitrogen-enriched vapor for the first condenser is compressed with the feed air upstream of the heat exchanger. For this, a complex and non-standard compressor is required.

Since the nitrogen-enriched vapor is compressed upstream of the main exchanger, the valves, piping, purification systems, coolers, columns and heat exchanger all have to have dimensions calculated from the size of the total compressed stream comprising the main air feed and the nitrogen-enriched vapor.

By the present invention, it is intended to reduce the capital costs of the apparatus and the energy costs of the process. In addition, it is possible to reduce the size of the air purification system with respect to that used in the prior art.

It is also known from EP-A-0.607.979 that the refrigeration requirements for a single column nitrogen generator may be supplied by expanding part of the feed air.

According to the invention, there is provided a process for the production of highly pure nitrogen product from air by cryogenic separation, comprising the steps of :

(a) feeding a compressed, dry, cleaned, and cooled feed air stream to a distillation column at a first level of the column ;

(b) separating said feed air in said distillation column to form a nitrogen-enriched vapor at the top of the column, and an oxygen-enriched liquid at the bottom of the column ;

(c) condensing in a first condenser a portion of said nitrogen-enriched vapor by indirect heat exchange with at least a portion of said oxygen-enriched liquid which at least partially vaporizes to form an oxygen-rich liquid and a second nitrogen-enriched vapor ;

(d) vaporizing at least a portion of said oxygen-rich liquid using a second condenser by indirect heat exchange with at least a portion of said nitrogen-enriched vapor to produce a waste stream and a nitrogen-enriched condensate ;

(e) recycling at least a portion of said second nitrogen-enriched vapor to a recycle compressor to form a compressed recycle stream,

characterized in that it comprises (f) feeding at least part of said compressed recycle stream to a second level of said column separated from said first level by at least one theoretical stage.

Preferentially, the said recycle compressor is a cold compressor and the oxygen enriched vapor delivered to the cold compressor is at a temperature less than -50°C.

Advantageously, the recycled portion of said nitrogen-enriched vapor is compressed in a compressor other than the main compressor.

According to the invention, there is also provided an apparatus for the production of nitrogen product by cryogenic distillation comprising :

(a) a heat exchanger to cool a feed air stream against products of feed air distillation ;

(b) a distillation column for separating said feed air into a substantially nitrogen vapor and an oxygen-enriched liquid and means for sending said feed air to a first level of said column ;

(c) a first condenser capable of vaporizing said oxygen-enriched liquid to form an oxygen-rich condensate and a nitrogen-enriched recycle stream by indirect heat exchange with a portion of said substantially nitrogen vapor ;

(d) means for withdrawing said oxygen-rich liquid and delivering said oxygen-rich liquid to a second condenser ;

(e) means for withdrawing said nitrogen-enriched recycle stream and delivering such nitrogen-enriched recycle stream to a recycle compressor ;

(f) indirect heat exchange means in said second condenser to provide for vaporization of said oxygen-rich liquid ;

(g)means to withdraw said waste stream and delivering said waste stream to said heat exchanger ;

(h) compressor means to compress said nitrogen-enriched recycle stream ; and

(i) means for sending said compressed recycle stream to a second level of said column ;

(j) means to deliver a portion of said nitrogen-enriched vapor to said heat exchanger for warming against other process streams,

characterized in that said first and second levels are separated by at least one theoretical stage.

Refrigeration for the process may be provided by expanding either a fraction of the feed air or a fraction of an oxygen-enriched stream produced by the distillation column.

Figure 1 is a schematic view of one embodiment of the present invention depicting major process streams and apparatus components.

Figure 2 is a schematic view of another embodiment of the present invention comprising a dissipative brake assembly and depicting major process streams and apparatus components.

Referring to Figure 1 wherein the preferred embodiment of the present invention is depicted, a feed air stream 2 is cooled in main heat exchanger 10 and delivered to the distillation column 20 in feed line 4. Before delivery to the distillation column, the feed air stream is dried and purified using well known techniques which may comprise, for example, adsorbers, filters, additional heat exchangers, or the like. In the single distillation column 20, oxygen is stripped in distillation section 17 and a nitrogen-enriched vapor is formed above the distillation section. At the bottom of the distillation column 20, an oxygen-enriched liquid stream 6 is withdrawn and subcooled against other process streams in main heat exchanger 10. Thereafter, the oxygen-enriched liquid stream is expanded and delivered to condenser section 30 via line 7. The first condenser section 30 comprises a first reboiler/condenser 50 wherein a first portion of the nitrogen-rich vapor from the distillation column is delivered via line 31, condensed by indirect heat exchange with the oxygen-enriched liquid stream and the nitrogen condensate returned to the distillation column as reflux in line 32. If desired, a portion of the nitrogen condensate may be withdrawn as a liquid nitrogen product.

The vaporization of a portion of the oxygen-enriched liquid stream in condenser section 30 produces a liquid phase and a nitrogen-enriched vapor phase in the shell of condenser section 30. In accordance with the present invention, each of such phases having different composition are further processed to provide highly efficient recovery of nitrogen product. The liquid formed in first condenser section 30 is withdrawn, at least a portion expanded and delivered via stream 8 to a second condenser section 40 which includes reboiler/condenser 60. In accordance with the present invention, at least a portion of the oxygen-rich liquid from the first condenser shell is vaporized in second condenser 40 by indirect heat exchange with at least a portion of the nitrogen-enriched vapor from the distillation column. Such second portion of nitrogen-enriched vapor is delivered to reboiler/condenser 60 via line 21 and produces a condensed nitrogen-enriched liquid in the condenser 40 which is withdrawn from condenser 40 via line 22, and at least a portion returned as reflux to the distillation column via line 24. Optionally, a liquid nitrogen product may be withdrawn from the second condenser via line 23. If desired, the liquid nitrogen produced may comprise either nitrogen condensate from the first condenser, second condenser, or a combination from both sources.

In accordance with the present invention, vaporized oxygen-enriched stream 41 is warmed against other process streams to form warmed oxygen-enriched stream 42. At least of portion of warmed oxygen enriched stream 42 is expanded in expansion device 80 to form expanded waste stream 45 which is further warmed against process streams in the main heat exchanger and thereafter taken from the process as waste stream 47.

The vapor formed in first condenser section 30 is withdrawn in line 12 and delivered to compressor 70 and following compression thereafter delivered in line 13 to the distillation column. In accordance with the present invention, the vapor stream 12 withdrawn from condenser 30 has a higher oxygen content than feed air, and it is preferable that the stream be recycled following compression to a point at least one theoretical stage below the feed point of main feed air in line 4. Typically, said recycle stream comprises between 25 and 29 mole percent oxygen and said waste stream comprises greater than 46 mole percent oxygen. Preferably, a distillation section 19 is disposed between the main air feed point and the point in the distillation column where recycle oxygen enriched stream 13 is returned.

In the preferred embodiment of our invention, expansion device 80 is mechanically coupled to compressor 70 such that at least some of the energy of expansion is directly used for to compression, and compressor 70 is preferably a cold compressor which is mechanically integrated with expansion device 80. In this case, an energy absorption device 89 is used to dissipate energy of expansion of a portion of stream 42 in device expansion 88, for thermal balance in the process. The devices 80 and 88 can be combined as a single device coupled to compressor 70 as shown in Figure 2. In this configuration a brake device 81 can be attached to the shaft of the coupled system to dissipate a portion of the energy, to keep the overall process in balance.

Gaseous nitrogen product is withdrawn from the top of distillation column 20 and delivered to the main heat exchanger in line 26 to be warmed and available as gaseous nitrogen product in line 27.

Among other factors, one advantage of the process and apparatus of the present invention is that a higher pressure may be maintained in condenser section 30, since a liquid stream is withdrawn enabling the vaporized stream to contain less oxygen. Further, if condenser 30 is operated at higher pressure, the work required by compressor 70 is lessened, and therefore higher recycle flow can be achieved at the same power input for compressor 70. In the processes of the present invention, higher recycle flow together with an increased nitrogen concentration translates to a higher overall recovery of nitrogen. Other advantages will become apparent to those skilled in the art once having the benefit of the herein provided description of the present invention, and the examples provided below.

<u>EXAMPLE</u>

The invented process has been simulated for a nitrogen generator having a nitrogen product flow of 100,000 SCFH at 124 psia and 1ppm oxygen purity.

A dry and clean atmospheric air stream (substantially free of nitrogen and CO_2) of 173,549 SCFH at 132 psia and 60°F (stream 2) is cooled in exchanger 10 to a temperature of -268°F before entering an intermediate stage of the distillation column 17 via stream 4.

A oxygen rich liquid flow of 132,519 SCFH containing 39.77 mol percent oxygen was withdrawn from the bottom of column 17 via stream 6, subcooled in exchanger 10 to -277.6°F, expanded across a valve and fed to the main vaporizer shell 30 via stream 7. A gaseous oxygen rich recycle stream 12 having a flow of 58,971 SCFH and 27.7 mol percent oxygen exits the main vaporizer 30 at 74.9 psia and -279.4°F. Stream 12 was then compressed in recycle booster 70 to 129.8 psia and fed to the bottom of the column 17. The balance of the oxygen rich liquid which was fed to the main vaporizer 30 was withdrawn via stream 8 and vaporized in the auxiliary vaporizer 40 at 57.75 psia and -279.4°F. This gaseous oxygen rich waste stream 41 was warmed in the main exchanger 10 to -238°F, expanded in turbines 80 and 88, then reentered the main exchanger 10 where it was warmed to 55°F. The waste stream 47 has a flow of 73,548 SCFH and contained 49.5 mol percent oxygen.

A gaseous nitrogen stream with a flow of 100,000 SCFH at 126.4 psia and -276.6°F was withdrawn from the top of distillation column 17 via stream 26, warmed in exchanger 10 and delivered as product at 124 psia and 55°F by stream 27.

To illustrate the advantages of the present invention, the process given by figure 4 of US-A-4,966,002 was simulated to compare the air feed requirement to the present process. Similar production requirements, heat leaks, exchanger temperature pinches, column operating pressures, etc. were used in carrying out the simulation.

The simulation results showed air feed to the cold box is reduced by 4.55% when compared to the process of Figure 4 of US-A-4,966,002.

Similarly the process of the present invention was compared with that of US-A-4.883.519 giving the following results :

	US-A-4,883,519	Example
Oxygen content of waste nitrogen (stream 47) (%)	40.7	49.5
Recycle stream pressure (psia)	68	74.9
Recycle stream flow (% feed air)	17.25	34
Feed air flow (% total feed in column)	85.3	74.6
Relative power consumption	100	90

Thus, the power consumption of the process of the present invention is considerably lower than that of US-A-4,883,519.

CLAIMS

1. A process for the production of highly pure nitrogen product from air by cryogenic separation, comprising the steps of :

(a) feeding a compressed, dry, cleaned, and cooled feed air stream to a distillation column (20) at a first level ;

(b) separating said feed air in said distillation column to form a nitrogen-enriched vapor at the top of the column, and an oxygen-enriched liquid at the bottom of the column ;

(c) condensing in a first condenser (50) a portion of said nitrogen-enriched vapor by indirect heat exchange with at least a portion of said oxygen-enriched liquid which at least partially vaporizes to form an oxygen-rich liquid and a second nitrogen-enriched vapor ;

(d) vaporizing at least a portion of said oxygen-rich liquid in a second condenser (60) by indirect heat exchange with at least a portion of said nitrogen-enriched vapor to produce a waste stream and a nitrogen-enriched condensate ;

(e) recycling at least a portion of said second nitrogen-enriched vapor to a recycle compressor (70) to form a compressed recycle stream,

characterized in that it comprises (f) feeding at least part of said compressed recycle stream to a second level of said column separated from said first level by at least one theoretical stage.

2. A process as claimed in claim 1, wherein at least a portion of said nitrogen-enriched condensate is removed as liquid nitrogen product.

3. A process as claimed in claim 1, wherein all of said nitrogen-enriched condensate from said second condenser is returned as reflux to said distillation column.

4. A process as claimed in any preceding claim wherein at least a portion of said nitrogen-enriched vapor condensed in said first condenser (50) is removed as liquid nitrogen product.

5. A process as claimed in any preceding claim wherein said recycle stream comprises between 25 and 29 mole percent oxygen and said waste stream comprises greater than 46 mole percent oxygen.

6. A process as claimed in any preceding claim comprising expanding at least a portion of said waste stream or of said feed air in an expansion device (80) to provide refrigeration for said process.

7. A process as claimed in any preceding claim wherein said expansion device (80) is mechanically coupled to said recycle compressor (70).

8. A process as claimed in any preceding claim, wherein said compressor (70) is a cold compressor and said oxygen enriched vapor delivered to said cold compressor is at least at a temperature of less than -50 degrees Celsius.

9. A process as claimed in any preceding claim further comprising expanding a portion of said waste stream in a second expansion device (80) mechanically coupled to an energy-dissipating device (81).

10. A process as claimed in any preceding claim wherein substantially all of said oxygen-rich condensate is vaporized, warmed and expanded in said expansion device (80).

11. A process as claimed in any preceding claim wherein at least a portion of said feed air is stripped in a stripping zone (19) in said distillation column (20) to produce at least a portion of said oxygen-enriched liquid.

12. A process as claimed in any preceding claim wherein said portion of said nitrogen-enriched vapor is compressed in a compressor (70) other than the main air compressor.

13. An apparatus for the production of nitrogen product under cryogenic conditions comprising:

(a) a heat exchanger (10) to cool a feed air stream against products of feed air distillation ;

(b) a distillation column (20) for separating said feed air into a substantially nitrogen vapor and an oxygen-enriched liquid and means for sending said feed air to a first level of said column (20) ;

(c) a first condenser (50) capable of vaporizing said oxygen-enriched liquid to form an oxygen-rich condensate and a nitrogen-enriched recycle stream by indirect heat exchange with a portion of said substantially nitrogen vapor ;

(d) means (8) for withdrawing said oxygen-rich liquid and delivering said oxygen-rich liquid to a second condenser (60) ;

(e) means for withdrawing said nitrogen-enriched recycle stream and delivering such nitrogen-enriched recycle stream to a recycle compressor (70) ;

(f) indirect heat exchange means in said second condenser to provide for vaporization of said oxygen-rich liquid;

(g) means (41) to withdraw said waste stream and delivering said waste stream to said heat exchanger;

(h) compressor means (70) to compress said nitrogen-enriched recycle stream ;

(i) means for sending said compressed recycle stream to a second level of said column (20) ; and

(j) means to deliver a portion of said nitrogen-enriched vapor to said heat exchanger for warming against other process streams,

characterized in that said first and second levels are separated by at least one theoretical stage.

14. An apparatus as claimed in claim 13, further comprising :

(a) means to withdraw said waste stream from said heat exchanger and expand at least a portion of said waste stream in at least one expansion device (80) or means to expand at least part of the feed air in an expansion device.

15. An apparatus as claimed in claim 13 or 14, further comprising:

(a) stripping means (19) in said distillation column (20) below said first level ; and

(b) means for deliver compressed nitrogen-enriched recycle stream from said compressor means (70) to said distillation column below said stripping section.

16. An apparatus as claimed in any of Claims 12 to 15 wherein said compressor means (70) are located within a cold box used to insulate the column (20) and/or the heat exchanger (10).

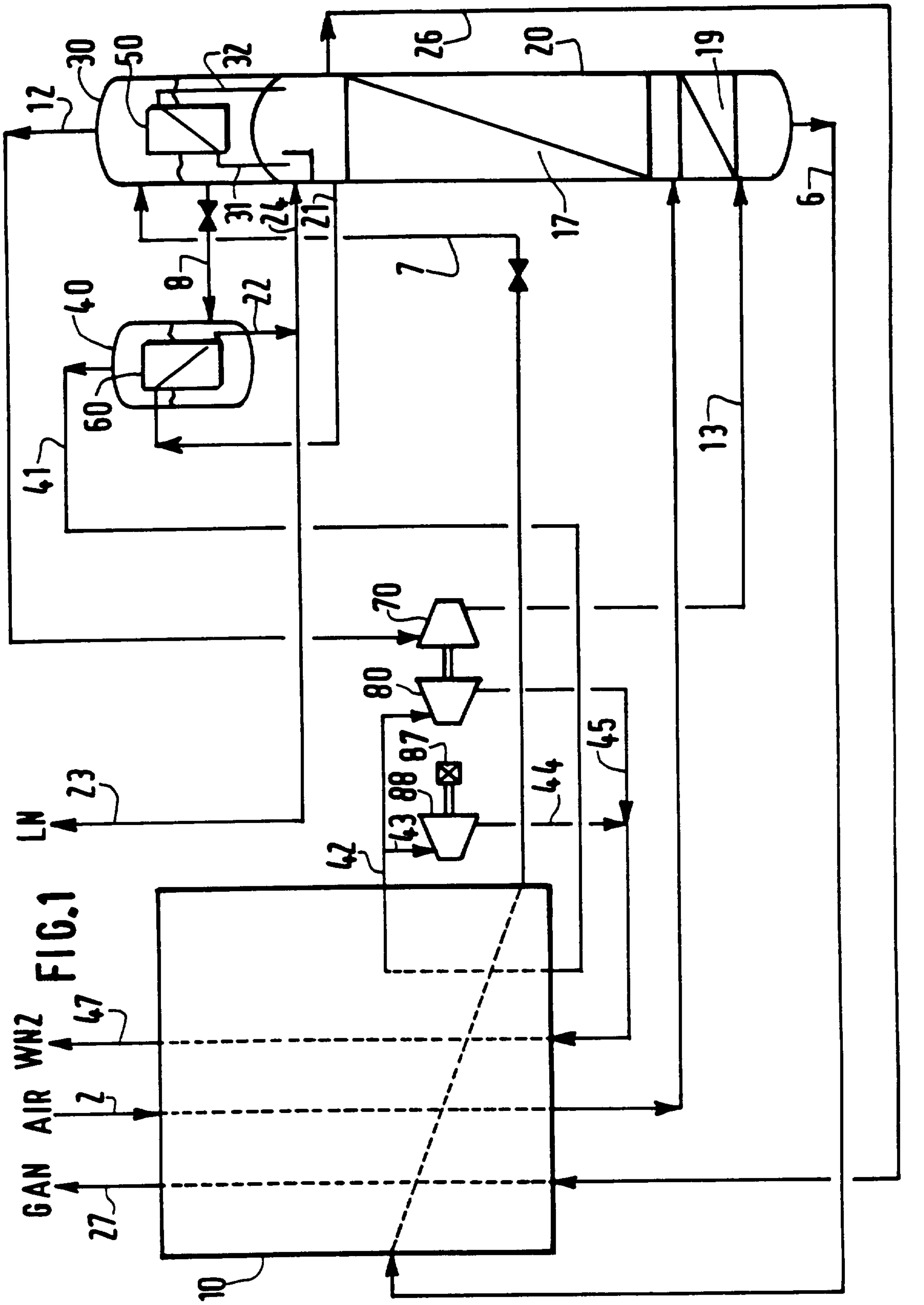

FIG.1

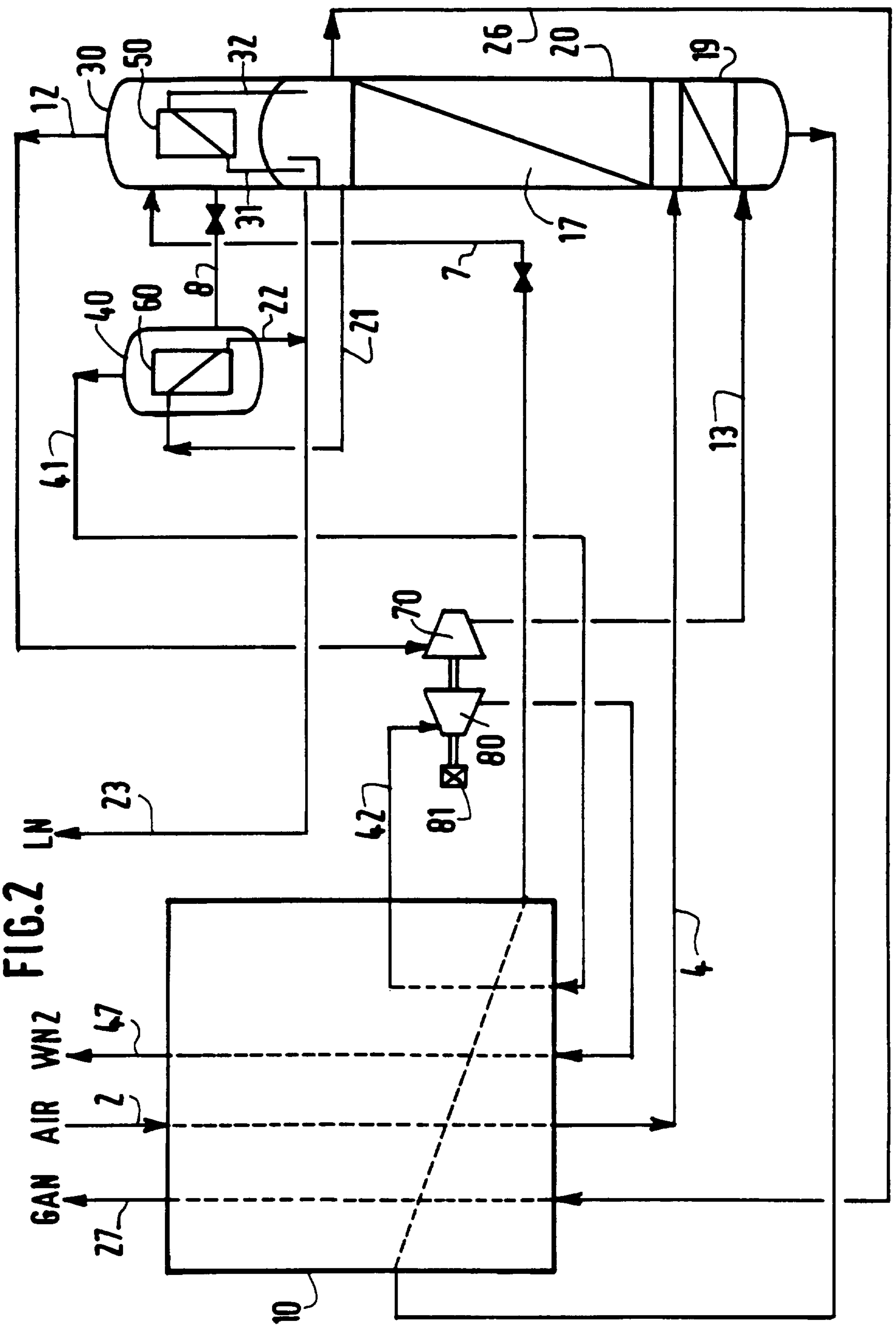
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FIG.2

INTERNATIONAL SEARCH REPORT

Inter nal Application No
PC1/IB 96/00323

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 F25J3/04

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 F25J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US,A,4 883 519 (AGRAWA ET AL.) 28 November 1989 cited in the application see claims; figure 2	1-5, 13-15
Y	US,A,4 867 773 (THOROGOOD ET AL.) 19 September 1989 see column 7, line 49-56; claims; figures 2,3	1-5, 13-15
A	US,A,4 996 002 (PARKER ET AL.) 26 February 1991 cited in the application	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
5 July 1996	10.07.96

Name and mailing address of the ISA	Authorized officer
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INTERNATIONAL SEARCH REPORT

.formation on patent family members

Interr nal Application No
PC1/IB 96/00323

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A-4883519	28-11-89	CA-A- 1280356	19-02-91
		EP-A- 0425738	08-05-91
US-A-4867773	19-09-89	CA-A- 1280686	26-02-91
US-A-4996002	26-02-91	CA-A- 1334959	28-03-95
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		JP-A- 1156409	20-06-89