AUSTRALIA 643513 10

PATENTS ACT 1990

PATENT REQUEST: STANDARD PATENT

We, THE BOC GROUP, INC., a corporation organised under the laws of the State of Delaware, United States of America, being the person identified below as the Applicant, request the grant of a patent to the person identified below as the Nominated Person, for an invention described in the accompanying standard complete specification.

Full application details follow.

Applicant: THE BOC GROUP, INC.

Address: 575 Mountain Avenue, Murray Hill, New Providence, New Jersey 07974, United States of America

Nominated Person: As above

Address: As above

Invention Title: "PROCESS FOR PRODUCING OXYGEN ENRICHED PRODUCT STREAM"

Name of actual inventor: Eiji Hirooka

BASIC CONVENTION APPLICATION DETAILS:
Application Number: 07/501,456
Country: United States of America
Country Code: US
Date of Application: 29th March, 1990

Address for service is: SHELSTON WATERS
55 Clarence Street
SYDNEY NSW 2000

Attorney Code: SW

DATED this 18th Day of December, 1992
THE BOC GROUP, INC.

by

Fellow Institute of Patent Attorneys of Australia
of SHELSTON WATERS

To: The Commissioner of Patents
WODEN ACT 2606

File: 15944
Fee: Previously paid

P/00/008
Section 29(1)
Regulation 3.1(2)

AUSTRALIA

PATENTS ACT 1990

NOTICE OF ENTITLEMENT

We, THE BOC GROUP, INC., a corporation organised under the laws of the State of Delaware, United States of America, of 575 Mountain Avenue, Murray Hill, New Providence, New Jersey 07974, United States of America, being the applicant in respect of Application No. 73719/91, state the following:-

1. The person nominated for the grant of the patent has entitlement from the actual inventor by assignment.

2. The person nominated for the grant of the patent has entitlement from the applicant of the basic application listed on the patent request form by assignment.

3. The basic application listed on the patent request form is the first application made in a Convention country in respect of the invention.

For and on behalf of
THE BOC GROUP, INC.

Donald L MacLean CRE ..... Dec 3, 1992

(Signature) (Date)

Name: Donald L. MacLean
Title: V.P. Gases & Vacuum Technology

File: 15944

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(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 643513

(54) Title
PROCESS FOR PRODUCING OXYGEN ENRICHED PRODUCT STREAM

International Patent Classification(s)
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THE BOC GROUP,INC.

(72) Inventor(s)
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(56) Prior Art Documents
AU 605677 30731/89 B01D 53/04
AU 604950 20298/88 B01D 53/04
AU 584785 69194/87 B01D 53/04

(57) Claim

1. A process for producing an oxygen enriched product stream from a feed gas containing at least oxygen and nitrogen utilizing two adsorption columns containing an adsorbent capable of selective adsorption of nitrogen and a product reservoir comprising:

(i) introducing gas into the outlet of a first adsorption column at a low pressure from the outlet of a second adsorption column at a higher pressure to substantially equalize the pressures in the columns while withdrawing gas from the inlet of the second column;

(ii) after equalization of step (i), introducing product gas contained within the product reservoir at a higher pressure into the outlet of the first column to backfill the first column while continuing to withdraw gas from the inlet of the second column;

(iii) introducing feed gas into the inlet of the first

.../2

column at a predetermined minimum pressure of between 0 and 7 psig and recovering the product of enriched oxygen gas from the outlet of the first column to introduce said enriched oxygen stream to the product reservoir, while introducing a portion of the oxygen enriched stream to the outlet of the second column to purge the second column and simultaneously withdrawing gas from the inlet of the second column to desorb and evacuate nitrogen rich gas from the second column;

(iv) introducing gas into the outlet of the second column from the outlet of the first column at an

9. An apparatus when used for carrying out the process of any one of Claims 1 to 8, said apparatus comprising two adsorption columns and a reservoir in fluid communication with said adsorption columns and adapted to receive enriched oxygen streams from said adsorption columns and to supply enriched oxygen stream to said adsorption columns, and means to repetitively cycle said two adsorption columns between receiving said feed stream of air and producing said enriched oxygen stream, and a vacuum pump means, said vacuum pump being connected to both of said adsorption columns and adapted to withdraw gas continuously from one or the other of said adsorption columns during said repetitive cycle.

COMMONWEALTH OF AUSTRALIA 643513

FORM 10

PATENTS ACT 1952

COMPLETE SPECIFICATION

FOR OFFICE USE:

Class Int.Class

Application Number:
Lodged:

Complete Specification Lodged:
Accepted:
Published:

Priority:

Related Art:

Name of Applicant: THE BOC GROUP, INC.

Address of Applicant: 575 Mountain Avenue, Murray Hill, New Providence, New Jersey 07974, United States of America

Actual Inventor: Eiji Hirooka

Address for Service: SHELSTON WATERS, 55 Clarence Street, Sydney

Complete Specification for the Invention entitled:

"PROCESS FOR PRODUCING OXYGEN ENRICHED PRODUCT STREAM"

The following statement is a full description of this invention, including the best method of performing it known to us:-

PROCESS FOR PRODUCING OXYGEN ENRICHED PRODUCT STREAM

BACKGROUND OF THE INVENTION

This invention relates to a process for obtaining an oxygen enriched gas from a mixed gas containing principally oxygen and nitrogen as gas components, such as air, by means of pressure swing adsorption (PSA).

Pressure swing adsorption systems and process have been widely used to produce oxygen enriched streams from mixed gases, including air, and a multitude of such systems and processes have been utilized.

It is advantageous, in such systems, to utilize a relatively short time cycle for carrying out the process inasmuch as shorter times obtain good utilization of the sieve material used to adsorb one of the components. The short cycle times generally employ a finer particle size of sieve material to reduce diffusive resistance. Typical examples of short cycle times are shown and described in U.S. Patents 4,194,891 and 4,194,892. Oxygen production increased with the process in the aforementioned patents however the yield was fairly low, e.g., 10-20% yields.

Conventional vacuum PSA processes do produce a higher oxygen yield (50-60%) however the production rate is somewhat low. The

normal production rate of conventional three bed PSA processes is not particularly high; as an example, typically, the bed size factor is about 2000-2600 kg of zeolite per metric ton of oxygen produced per day.

Optimally, one would obviously like to obtain the high production rate typified by faster cycle times and finer sieve particles along with the high yield typified by conventional three bed systems yet have a process that is inexpensive and relatively simple in operation.

SUMMARY OF THE INVENTION

According to a first aspect, the present invention consists in a process for producing an oxygen enriched product stream from a feed gas containing at least oxygen and nitrogen utilizing two adsorption columns containing an adsorbent capable of selective adsorption of nitrogen and a product reservoir comprising:

(i) introducing gas into the outlet of a first adsorption column at a low pressure from the outlet of a second adsorption column at a higher pressure to substantially equalize the pressures in the columns while withdrawing gas from the inlet of the second column;

(ii) after equalization of step (i), introducing product gas contained within the product reservoir at a higher pressure into the outlet of the first column to backfill

the first column while continuing to withdraw gas from the inlet of the second column;

(iii) introducing feed gas into the inlet of the first column at a predetermined minimum pressure of between 0 and 7 psig and recovering the product of enriched oxygen gas from the outlet of the first column to introduce said enriched oxygen stream to the product reservoir, while introducing a portion of the oxygen enriched stream to the outlet of the second column to purge the second column and simultaneously withdrawing gas from the inlet of the second column to desorb and evacuate nitrogen rich gas from the second column;

(iv) introducing gas into the outlet of the second column from the outlet of the first column at an initially high pressure to equalize the pressures in the columns while withdrawing gas from the inlet of the first column;

(v) after equalization of step (iv), introducing oxygen enriched product gas contained within the product reservoir at a higher pressure into the outlet of the second column while continuing to withdraw gas from the inlet of the first column;

(vi) introducing feed gas into the inlet of the second column at a predetermined minimum pressure of between 0 and 7 psig and recovering the product of enriched oxygen

gas from the outlet of the second column to introduce said enriched oxygen stream to the product reservoir while introducing a portion of the oxygen enriched stream to the outlet of the first column to purge the first column while simultaneously withdrawing gas from the inlet of the first column to desorb and evacuate nitrogen rich gas from the first column; and

(vii) cyclically repeating steps (i) to (vi) while withdrawing oxygen enriched product gas from the product reservoir.

According to a second aspect, the present invention consists in an apparatus when used for carrying out the process of the first apsect, said apparatus comprising two adsorption columns and a reservoir in fluid communication with said adsorption columns and adapted to receive enriched oxygen streams from said adsorption columns and to supply enriched oxygen stream to said adsorption columns, and means to repetitively cycle said two adsorption columns between receiving said feed stream of air and producing said enriched oxygen stream, and a vacuum pump means, said vacuum pump being connected to both of said adsorption columns and adapted to withdraw gas continuously from one or the other of said adsorption columns during said repetitive cycle.

The PSA process of the present invention achieves an enriched oxygen product that has a high oxygen yield as well as high production rate by solving the disadvantages of the prior art while minimizing cost and

maintaining simplicity of operation.

The system uses short cycle times to gain good usage of the sieve material but requires only two beds, thus greatly simplifying the prior three-bed process that heretofore were needed for high yields.

As will be shown, one of the further features is the power saving by continuous operation of a vacuum pump with a two column system by simultaneously carrying out a equalization step supplying an oxygen enriched stream from the product to the outlet end of one column and a desorption to evacuate nitrogen rich gas from the inlet end of the same column. By this means, the vacuum pump is run continuously throughout this step as well as the remaining steps. Since the vacuum pump is not running idle or unused during any step of the overall process, its use, and thus its power consumption, is optimized.

Thus, the two bed PSA process having high yield and production rate is achieved using relatively, fine particles of zeolite sieve material, such as 20-35 mesh size and even with larger particles such as 8-12 mesh size at short cycle times less than 30 seconds, preferably about 15-25 seconds. The range of pressure swing uses ----------

vacuum for desorption less than 300 torr, preferably to 200 torr and maximum product pressure less than 5 psig and preferably less than 3 psig.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic flow diagram of the present invention; and

FIG. 2 is a column cycle of the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENT

The process of the present invention will specifically be described with reference to the schematic flow diagram shown on FIG. 1 and the column cycle of FIG 2.

Taking FIG. 1 and FIG. 2 together, there is shown a process for producing an enriched oxygen gas stream continuously generated from a gas containing principally oxygen and nitrogen, such as air. Each of the two adsorption columns A and B contains an adsorbent capable of selectively adsorbing nitrogen.

The process is performed with relatively fine particles of zeolite of about 8-35 mesh, preferably about 12-20 mesh. Typical zeolite sieve material is available from various zeolite manufacturers in the form of beads or pellets. Control of each of the steps can be regulated by conventional means, e.g., a timer to control solenoid operated valves of standard commercial design.

In step 1, valves 1A and 2A are closed, thereby closing off the lower or inlet end of the first column A. At the top, or outlet end of column A, valves 4A and 5A are closed and valve 3A is open. With respect to the second column B, valves 9B and 10B are closed, thus gas from the outlet of column B is being introduced into the outlet of column A through valve 3A and the flow is controlled by valve 8B. At the same time in Step 1, at the inlet end of column B valve 6B is closed but valve 7B is open, thus gas is being withdrawn from the inlet end of column B by means of vacuum pump 16.

During this step, the pressure in column B which is initially higher than the pressure in column A is used to equalize the pressures in both columns, that is, column B may, at the beginning of step 1, be at a positive pressure of about 1010 torrs (4.84 psig) and is reduced to about 500 torrs (-5.03 psig) by the end of step 1 while column A commences step 1 at a pressure of about 260 torrs (-9.67 psig) and the pressure is raised to 470 torr (-5.61 psig). At the end of step 1, therefore, the column pressures are essentially equalized. Step 1 may take place extremely rapidly, and preferably from about 2 seconds to 6 seconds and more preferably in about 4 seconds.

In step 2, valve 3A is closed and valve 5A and valve 24 opened and oxygen enriched product gas from the reservoir 18 is introduced into the outlet of column A and is controlled by metering valve 26 to backfill column A and to raise the pressure further in column A. Typically, since the pressure in product reservoir is about 800 torr, the pressure within column A continues to increase from 470 torr (-5.61 psig) to about 660 torr (-1.93 psig). At the same time, gas is still being withdrawn from the inlet of column B through open valve 7B by vacuum pump 16 and the pressure within column B

continues to decrease. That pressure may decrease, in step 2, from 500 torr (-5.03 psig) to about 450 torr (-6.00 psig). Again, the timing of step 2 is extremely rapid, preferably being from about 1 to 5 seconds, and more preferably about 3 seconds.

In step 3, valve 1A is opened and air, or other feed gas containing principally oxygen and nitrogen under a predetermined feed pressure, is introduced to the inlet of column A. Inlet pressures may vary but the inlet pressure should have a predetermined minimum pressure of between 0 and 7 psig, preferably between about 3 to about 7 psig and more preferably about 5 psig. At the outlet of column A, valve 5A is closed and valve 4A is opened, thus the feed air passes through column A where nitrogen is adsorbed and an oxygen enriched product stream passes from the outlet end of column A, through valve 4A, check valve 11 and through line 20 to the product reservoir 18. During this step, the pressure in column A may increase from 660 torr (-1.93 psig) to about 1010 torr (4.84 psig) in producing oxygen enriched product. At the same----------

time, in step 3, gas continues to be withdrawn from the inlet end of column B to desorb or evacuate nitrogen enriched gas from column B by means of vacuum pump 16. Simultaneously, with the desorption of column B via its inlet, valve 10B is open and valve 24 open and oxygen enriched product stream is introduced into the outlet of column B to purge column B. The flow of the oxygen enriched product stream is controlled by metering valve 26. Thus column B is both purged by an oxygen enriched stream of gas introduced into its outlet and desorbed by withdrawing nitrogen rich gas from its inlet. The pressure in column B thus continues to decrease, typically from about 450 torr (-6.10 psig) to about 260 torr (-9.67 psig) as the withdrawal by means of vacuum pump 16 continues uninterrupted. Step 3 is also carried out quite rapidly, in a range of cycle time preferably of about 10 seconds to 25 seconds and more preferably about 18 seconds.

Continuing on to step 4, equalization again takes place, this time by introducing the now higher pressure gas of column A into column B. That is carried out by closing valve 4A at the outlet of column A and opening valve 3A. At the outlet of column B, valve 10B is closed and thus, gas passes from column A to column B for equalization of pressures controlled by metering valve 8B. At the same time, of course, the feed stream is cut off by closing valve 1A at the inlet to column A and valve 2A is opened so that gas can be withdrawn from the inlet end of column A through vacuum pump 16. The inlet of column B is closed completely by closing valve 7B.

In step 4, therefore, the pressures within column A and B are approximately equalized, the pressure in column A is reduced from about 1010 torr (4.84 psig) to about 500 torr (-5.03 psig) while the pressure in column B is increased from about 260 torr (-9.67 psig) to about 470 torr (-5.61). Step 4 is carried out preferably in a time of from about 2 to about 6 seconds, and more preferably in about 4 seconds.

In step 5, valve 3A is closed, thus closing entirely the outlet end of column A while gas continues to be withdrawn from the inlet end of column A drawing the pressure down, typically, from 500 torr

(-5.03 psig) to a further 450 torr (-6.00 psig). Valve 10B and 28 are opened and oxygen enriched product from product reservoir 18 enters column B to backfill that column controlled by metering valve 30 such that the pressure in column B is increased from, typically, about 470 torr (-5.61 psig) to about 660 torr (-1.93 psig). Again, as in step 2, the backfilling step takes place in from about 1 to 5 seconds, and preferably in about 3 seconds.

Finally, in step 6, valve 6B is opened, thus introducing the pressurized feed stream into the inlet of column B. Valve 9B is opened so that the oxygen enriched product stream from column B passes through check valve 12 and continues via line 20 to product reservoir 18.

During step 6, valves 24 and 5A are opened to allow oxygen enriched gas to enter the outlet of column A to purge column A, controlled by metering valve 26. Simultaneously, with the purging of column A, gas continues to be withdrawn from the inlet of column A by vacuum pump 16 to desorb or evacuate nitrogen rich gas. Typically, again, the pressure within column A decreases from about 450 torr (-6.00 psig) to about 260 torr (-9.67 psig) while the pressure in column B increases from about 660 torr (-1.93 psig) to about 1010 torr (4.84 psig). The timing of step 6 can be from about 10 to about 25 seconds and preferably in about 18 seconds.

At the completion of step 6, the entire sequence is repeated on a continual cyclic basis so that product is continuously taken from product reservoir 18 through valve 32 during each of the steps.

As can be seen, the vacuum pump 16 is also continuously utilized to withdraw gas alternatively, from one or the other of the two columns, thus it is efficiently utilized to minimize power use throughout the process.

EXAMPLES

Using the apparatus described, with the sequence of steps as outlined, an operation of this method was conducted to obtain an

oxygen enriched product stream. Two adsorption columns, A and B, each 2 inches in diameter and 15 inches in height were packed with Calcium X zeolite molecular sieve material in the form of beads commercially available from Laporte Co. for three runs and zeolite material in the form of pellets from Tosoh Company for one run.

The production rate, and yields were obtained in accordance with the following table:

Table 1

Molecular sieves material (Zeolite)	Laporte			Tosoh Zeolum SA
Size of MS	0.4-0.8 mm (beads)			1.5mm (pellets)
Range of pressure swing	3.5 psig to-200 torr			
Cycle times (second)	25	20	17	25
Oxygen purity (%)	93	93	93	93
Bulk density (kg/cm3)	681			620
Oxygen yield (%)	59	58	56	55
specific product (standard liter/hr of produced oxygen/liter of bed)	40	51	59	40
Bed size factor (kg zeolite per metric tons of oxygen per day)	535	420	375	487

From the above test, it can be seen that a high yield, high production rate at a high purity can be achieved by a two column system where vacuum is continuously applied to one or the other of the columns during each of the steps, thus the vacuum pump is efficiently used and power conserved. The cycles are extremely rapid to achieve good utilization of the sieve material, yet the construction and operation of the two bed system is obviously more advantageous than the more complex, more expensive three bed systems.

While a particular embodiment of the invention has been shown,

it should be understood that the invention is not limited thereto, since modifications may be made, and it is contemplated to cover such modifications as fall within the spirit and scope of the appended claims:

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:-

1. A process for producing an oxygen enriched product stream from a feed gas containing at least oxygen and nitrogen utilizing two adsorption columns containing an adsorbent capable of selective adsorption of nitrogen and a product reservoir comprising:

(i) introducing gas into the outlet of a first adsorption column at a low pressure from the outlet of a second adsorption column at a higher pressure to substantially equalize the pressures in the columns while withdrawing gas from the inlet of the second column;

(ii) after equalization of step (i), introducing product gas contained within the product reservoir at a higher pressure into the outlet of the first column to backfill the first column while continuing to withdraw gas from the inlet of the second column;

(iii) introducing feed gas into the inlet of the first column at a predetermined minimum pressure of between 0 and 7 psig and recovering the product of enriched oxygen gas from the outlet of the first column to introduce said enriched oxygen stream to the product reservoir, while introducing a portion of the oxygen enriched stream to the outlet of the second column to purge the second column and simultaneously withdrawing gas from the inlet of the second column to desorb and evacuate nitrogen rich gas from the second column;

(iv) introducing gas into the outlet of the second column from the outlet of the first column at an

initially high pressure to equalize the pressures in the columns while withdrawing gas from the inlet of the first column;

(v) after equalization of step (iv), introducing oxygen enriched product gas contained within the product reservoir at a higher pressure into the outlet of the second column while continuing to withdraw gas from the inlet of the first column;

(vi) introducing feed gas into the inlet of the second column at a predetermined minimum pressure of between 0 and 7 psig and recovering the product of enriched oxygen gas from the outlet of the second column to introduce said enriched oxygen stream to the product reservoir while introducing a portion of the oxygen enriched stream to the outlet of the first column to purge the first column while simultaneously withdrawing gas from the inlet of the first column to desorb and evacuate nitrogen rich gas from the first column; and

(vii) cyclically repeating steps (i) to (vi) while withdrawing oxygen enriched product gas from the product reservoir.

2. A process as defined in Claim 1 wherein said feed gas introduced in steps (iii) and (vi) is air at a pressure of from about 3 to about 7 psig and said adsorbant is a fine particle zeolite of about 8-35 mesh.

3. A process as defined in Claim 2 wherein said fine particle zeolite is in the form of beads or pellets having a size of between about 12 and about 20 mesh.

4. A process as defined in any one of Claims 1 to 3 wherein said steps (i) and (iv) take place in about 2 to 6 seconds; said steps (ii) and (v) take place in about 1 to 5 seconds; and said steps (iii) and (vi) take place in about 10 to 25 seconds.

5. A process as defined in Claim 4 wherein said steps (i) and (iv) take place in about 4 seconds; said steps (ii) and (v) take place in about 3 seconds; and said steps (iii) and (vi) take place in about 18 seconds.

6. A process as defined in any one of Claims 1 to 5 wherein said step (i) and (iv) include equalizing the pressures in the columns to a pressure of from about 470 to about 500 torr; said product gas in steps (ii) and (v) is introduced at a pressure of about 800 torr; and said feed gas is introduced in steps (iii) and (vi) at a pressure of from about 0 to 7 psig.

7. A process as defined in Claim 6 wherein said feed gas is introduced in steps (iii) and (vi) at a pressure of less than about 5 psig.

8. A process as defined in any one of claims 1 to 7 wherein said steps (i) through (vi) are carried out in less than about 30 seconds.

9. An apparatus when used for carrying out the process of any one of Claims 1 to 8, said apparatus comprising two adsorption columns and a reservoir in fluid communication with said adsorption columns and adapted to receive enriched oxygen streams from said adsorption columns and to supply enriched oxygen stream to said

adsorption columns, and means to repetitively cycle said two adsorption columns between receiving said feed stream of air and producing said enriched oxygen stream, and a vacuum pump means, said vacuum pump being connected to both of said adsorption columns and adapted to withdraw gas continuously from one or the other of said adsorption columns during said repetitive cycle.

10. Apparatus as defined in Claim 9 wherein said air feed stream has a pressure of between about 0 to 7 psig and said adsorption columns contain fine particle zeolite material of about 8-35 mesh.

11. Apparatus as defined in claim 10 wherein said fine particle zeolite material is in the form of beads or pellets and having a size of between 12 and 20 mesh.

12. A process for producing an oxygen enriched product stream from a feed gas containing at least oxygen and nitrogen substantially as herein described with reference to Figures 1 and 2 of the accompanying drawings.

13. An apparatus when used for carrying out the process of Claim 12, substantially as herein described with reference to Figures 1 and 2 of the accompanying drawings.

DATED this 7th day of September 1993

THE BOC GROUP, INC.

Attorney: LEON K. ALLEN

Fellow Institute of Patent Attorneys of Australia

of SHELSTON WATERS

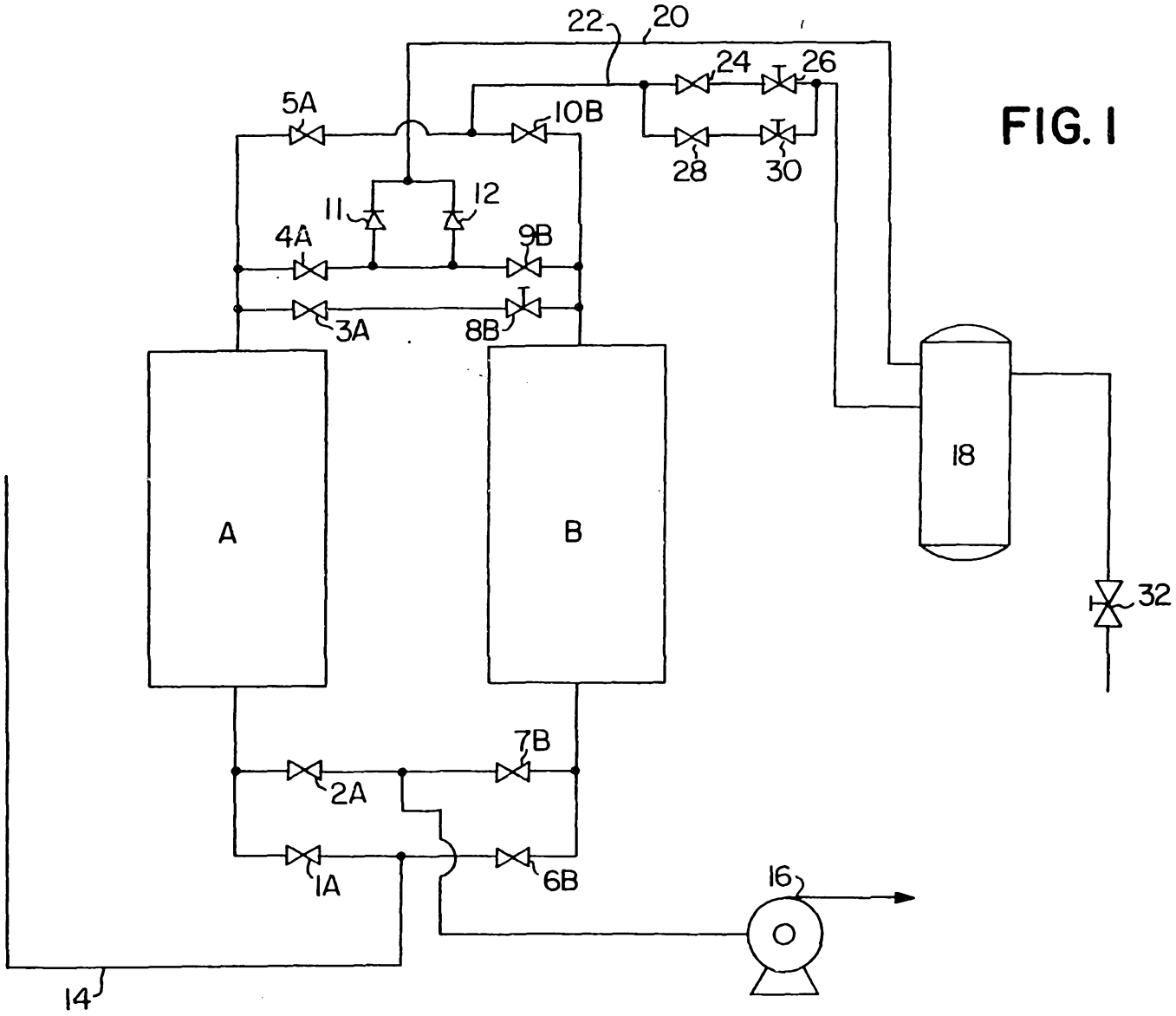

FIG. 1

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STEP 1

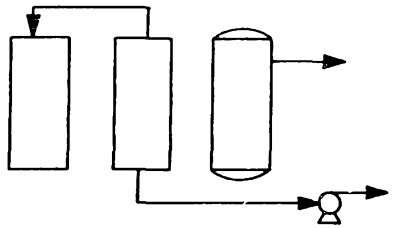

FIG. 2A

STEP 2

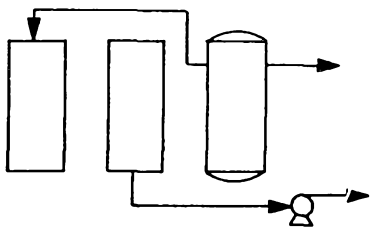

FIG. 2B

STEP 3

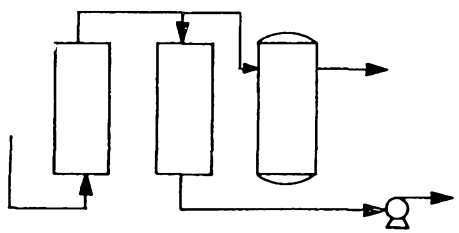

FIG. 2C

STEP 4

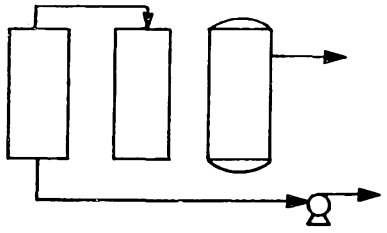

FIG. 2D

STEP 5

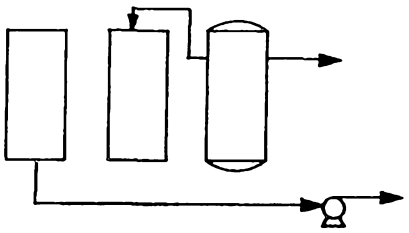

FIG. 2E

STEP 6

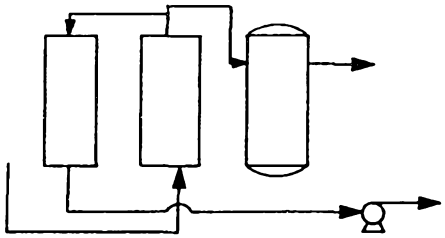

FIG. 2F