(12) UK Patent Application (19) GB (11) 2 321 861 (13) A

(43) Date of A Publication 12.08.1998

(21) Application No 9800017.7

(22) Date of Filing 05.01.1998

(30) Priority Data
(31) 796915 (32) 06.02.1997 (33) US

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(51) INT CL[6]
B01D 53/047

(52) UK CL (Edition P)
B1L LAB LAF L201 L308 L602A

(56) Documents Cited
WO 86/05119 A1 **US 4572723 A** **US 4534346 A**
US 4477264 A

(58) Field of Search
UK CL (Edition P) **B1L LAA LAB LAF LAH LAJ LAK LDJ LDK**
INT CL[6] **B01D 53/047 53/053**
Online: WPI, Claims, Japio

(54) Abstract Title
Air separation by PSA

(57) High purity nitrogen or oxygen in relatively small quantities is obtained using a single PSA adsorbing vessel wherein the nitrogen or oxygen is produced for about 4 minutes and only about 1 minute is needed to regenerate the molecular sieve material. A compressor is utilized to provide air under pressure and during regeneration of the molecular sieve material, the compressor continues to run and regenerates the sieve material by providing a vacuum in the adsorbing vessel. The alternate pressure and vacuum are controlled by a valve operated distributor.

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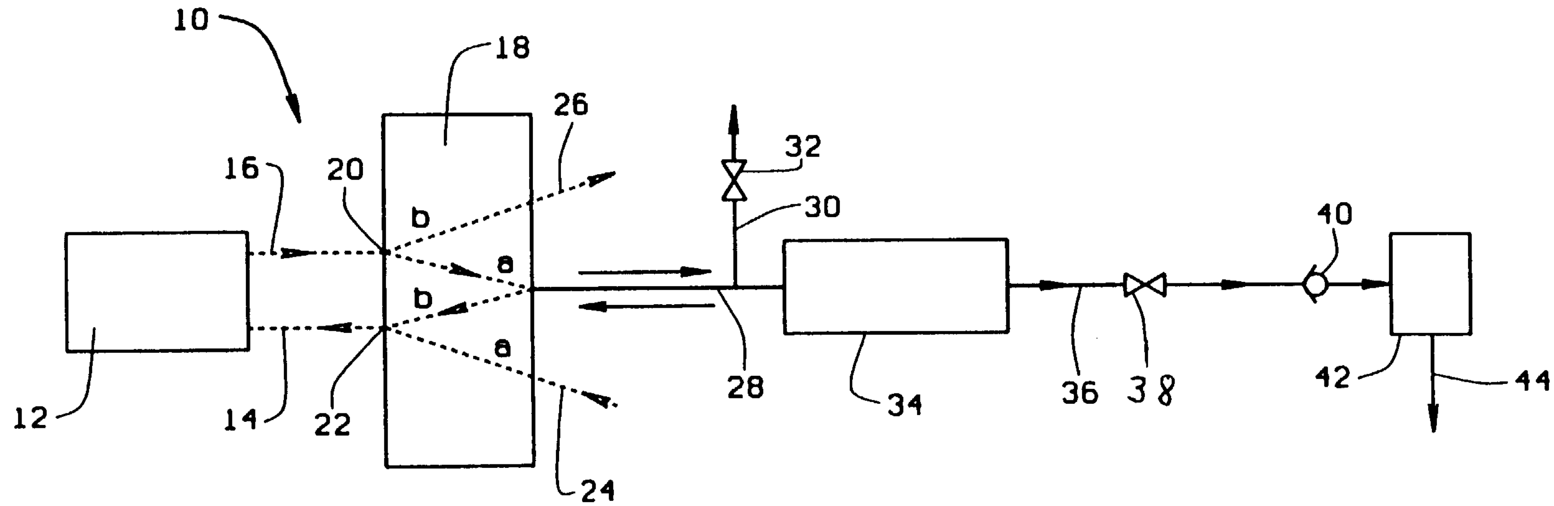

FIG. 1

BACKGROUND OF THE INVENTION

The present invention relates to preparation of high purity nitrogen or oxygen from air by use of pressure swing adsorption (PSA) technology. High purity nitrogen is recognized in the art as a term for nitrogen containing less than 5 % oxygen. The purity of the nitrogen gas stated herein is based on 100 % nitrogen less the oxygen content of the gas. Also high purity oxygen contains less than 5 % nitrogen and is based on 100 % oxygen less the nitrogen content of the gas.

It is known that air contains approximately 78% nitrogen and almost 21% oxygen, the remainder being trace amounts of argon, carbon dioxide, carbon monoxide and the like. The least expensive source of nitrogen or oxygen is air. Many processes and apparatuses have been developed to remove nitrogen from air to obtain substantially pure nitrogen.

These processes include separation of air utilizing membranes, molecular sieves, cryogenics and the like. One of the more economical processes is pressure swing adsorption using molecular sieves. PSA systems utilize carbon molecular sieve material under pressure in an adsorbing vessel to selectively adsorb oxygen from the pressurized air leaving substantially pure nitrogen. The molecular sieve material tends to adsorb substantially all of the gases in air except nitrogen leaving the nitrogen in the remainder of the adsorbing vessel resulting in production of high purity nitrogen. After the carbon molecular sieve material becomes substantially saturated with oxygen and other gases, the molecular sieve material is regenerated by removing the gases including oxygen from the sieve material. In order to remove the oxygen, it is necessary to purge the sieve material whether by reducing the pressure to atmosphere or flushing it with a gas which will remove the oxygen or by use of vacuum.

Similarly, when it is desirable to obtain high purity oxygen, a PSA system can be used, but the molecular sieve material is zeolite or the like.

A typical PSA system will have two adsorbing vessels or towers so that when the sieve material is being regenerated, in one tower, the other tower is producing nitrogen, hence the term "Pressure Swing Adsorption". The requirement for two towers and all of the necessary mechanisms to first activate one and then to shut down the first one while simultaneously starting the second one, necessitates complex mechanisms which are costly to purchase, utilize and maintain.

U.S. Patent 4,572,723 provides a process for obtaining nitrogen from air by use of one adsorbing vessel. In this process, only one tower is provided so that when the carbon molecular sieve becomes saturated with oxygen, it is necessary to interrupt the process and allow the oxygen to be removed from the sieve material by reducing the pressure in the adsorbing vessel to atmospheric pressure. The patent discloses a nitrogen producing cycle of about 500 seconds and then a regeneration cycle of about 500 seconds. Regeneration of the sieve material is achieved by venting the residual gases from the adsorbing vessel to the atmosphere. In order to utilize a system of this type, it is necessary to have a storage reservoir for the nitrogen under pressure. In this manner there is a supply of nitrogen for continuous use.

Most generally in small nitrogen systems, a compressor is used to provide the necessary pressure in the adsorbing vessel and to transfer the nitrogen to the storage reservoir. When utilizing only one tower, the compressor is turned off and then restarted for each cycle. Having to shut off the compressor, for several minutes, every few minutes, results in inefficient use of the compressor. A larger, more expensive compressor is therefore required for the prior art processes, e.g., in U.S. Patent 4,572,723.

The present invention provides small volumes of high purity nitrogen or oxygen from a single adsorbing vessel where the purified gas production time per hour is at least 25 % higher than that of the prior art, resulting in higher productivity for the same amount of molecular sieve material. Further, there is no need to turn off the compressor, thus the compressor runs continuously and consequently more efficiently.

BRIEF SUMMARY OF THE INVENTION

The present invention provides an apparatus and a process for the production of high purity nitrogen or oxygen from air in an amount less than about 6 Nm^3/hr. (Normal cubic meters are measured at 0° C at atmospheric pressure). The apparatus is comprised of:

(a) a compressor;

(b) a valve operated distributor suitable for feeding pressurized air to an adsorbing vessel and/or removing gas by vacuum from the adsorbing vessel;

(c) the adsorbing vessel containing molecular sieve material suitable for adsorption of selected gases from pressurized air received from the distributor to provide a pressurized purified gas stream;

(d) means for controlling the flow of the pressurized purified gas stream; and

(e) means for storing the purified pressurized gas under pressure.

Preferably, a compressor is utilized which is capable of producing up to about 20,000 l/hr of compressed air at a pressure up to about 100 psig (about 7 bar g), or vacuum of up to about 25 inches of Hg (about 63 cm Hg) without interruption. The compressor and vacuum cycles function alternately while the compressor runs continually.

This latter function is performed by utilization of a valve operated distributor. The valve operated distributor regulates the flow pattern of the air depending on whether the adsorption cycle is being utilized or whether the regeneration cycle is being used. The adsorption cycle, in a typical example, takes about 4 minutes whereas the regeneration cycle takes about 1 minute. The distributor reverses the flow of air in the adsorbing vessel placing the vessel under vacuum during regeneration of the molecular sieve material. Frequently, it is desirable to provide dessicant material in the adsorbing vessel. The dessicant is regenerated during the vacuum cycle.

The process of the present invention for the production of high purity nitrogen gas or high purity oxygen gas from air in an amount less than about 6 Nm^3/hr, the resulting purified gas containing about 3 % or less of residual gas, comprises:

(a) placing air under a predetermined pressure;

(b) transporting the pressurized air by means of a valve operated distributor to an adsorbing vessel containing molecular sieve material to separate the air into a purified gas stream under pressure and residual gas entrapped in the molecular sieve material;

(c) removing the purified gas stream under pressure from the adsorbing vessel through a flow control means to a means for storing the purified gas under pressure;

(d) substantially removing the residual gas entrapped in the molecular sieve material by venting the pressurized gas in the adsorbing vessel to the atmosphere, while simultaneously reversing the air flow through the distributor to create a vacuum in the adsorbing vessel;

(e) venting the removed residual gas from the process, and

(f) repeating steps (a) through (e).

BRIEF DESCRIPTION OF THE SEVERAL VIEWS OF THE DRAWINGS

FIG. 1 is a schematic illustration of one embodiment of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

The following detailed description illustrates the invention by way of example and not by way of limitation. This description will clearly enable one skilled in the art to make and use the invention, and describes several embodiments, adaptations, variations, alternatives and uses of the invention, including what is presently believed to be the best mode of carrying out the invention.

FIG. 1 is a schematic of one embodiment 10 of the present invention. A compressor 12 draws air through an inlet 24 to a line 14 into the compressor 12 to provide the necessary pressurized air to a line 16. The pressurized air enters a valve operated distributor 18 through an inlet 20. At the inlet 20 there is a three-way valve having one inlet and two outlets, thus the distributor 18 sends pressurized air either through the distributor channel "a" and hence to a line 28, or through a distributor channel "b" which allows the air to vent to the atmosphere through a line 26. At the outlet 22 from the distributor there is a three way valve, thus when the distributor is in the "a" mode, air is drawn through a line 24, but when the distributor is in the "b" mode, residual gas is drawn through the line 28 to the outlet 22. When the distributor is in the "a" mode, the pressurized air in the line 28 enters an adsorbing vessel 34. The adsorbing vessel 34 contains molecular sieve material and functions to adsorb residual gases present in minor portions and thus yields a high purity gas under pressure. The gas under pressure leaves the adsorbing vessel through a line 36 wherein the flow of the purified gas is regulated by a control valve 38. The purified gas under pressure flows through a check valve 40 to a storage container 42 and is dispensed through a line 44 to the area of intended use.

When the molecular sieve material of the adsorbing vessel 34 becomes saturated with residual gas, the flow of the purified gas through control valve 38 stops and a valve 32 opens to allow the pressure in the adsorbing vessel 34 to be released through a line 30. Generally this valve 32 is open only for a few seconds while simultaneously a switching takes place in the distributor 18. The switching allows channel "b" to draw a vacuum through the line 28 placing the adsorbing vessel 34 under vacuum and the residual gas being removed from the adsorbing vessel goes through the compressor and is vented to the atmosphere through the channel "b" outlet to the line 26. When the molecular sieve is regenerated, generally in one minute or less, the distributor 18 returns to the "a" mode and purified gas is again being produced in the adsorbing vessel 34.

The process of the present invention pressurizes air in the compressor 12 up to about 7 bar g or less, and passes the pressurized air to the distributor 18 which in turn sends the air to the adsorbing vessel 34. The adsorbing vessel 34 separates the air into a purified gas stream and residual gas entrapped in the molecular sieve material. The pressurized purified gas stream is sent to storage 42 whereupon it is dispensed as needed. When the molecular sieve material in the adsorbing vessel 34 becomes saturated with residual gas, the residual gas is removed by means of vacuum and vented to the atmosphere thereby regenerating the molecular sieve material. The vacuum in the adsorbing vessel 34 is created by switching the mode in the valve operated distributor 18 from channel "a" to channel "b". In order to make the transition from pressure to vacuum in the adsorbing vessel 34, a release valve 32 is opened for about 5 seconds while the channel switching takes place in the distributor 18.

Example 1

Air is compressed to reach the pressure shown in Table 1 below. The pressurized air is held under compression in an adsorbing vessel for the time indicated. The adsorbing vessel contains carbon molecular sieve material. When the sieve material becomes saturated with residual gas, mostly oxygen, the adsorbing vessel is placed under vacuum for the time shown. The flow rate of nitrogen and the purity of the nitrogen are also provided in Table 1.

Table 1

Pressure Bar	Compression Time	Vacuum Time	Volume N_2	Purity %
4.5	1 min 45 sec	1 min	150 l/hr	99.9
4.0	3 min	1 min	240 l/h	99.4
4.0	2 min	40 sec	200 l/h	99.7
4.0	4 min	40 sec	400 l/h	97.0
4.0	4 min	40 sec	320 l/h	98.0
4.0	3 min	40 sec	280 l/h	99.0
4.5	1 min 45 sec	1 min	50 l/h	99.99

A comparison of the prior art patent U. S. 4,572,723 and the present invention is set forth in Table 2 below.

Table 2

	U. S. 4,572,723	Present invention
Pressure, bar g	7.0	4.5
Carbon Molecular Sieve, Vol., liters	16.0	4.5
Nitrogen flow, l/min	0.70	0.83
Nitrogen purity, %	99.99	99.99

Capacity of the carbon molecular sieve material is linear to the amount of material used. The capacity of the carbon molecular sieve material increases with the increasing pressure as well, and is at least 50 % higher at 7.0 bar g versus 4.5 bar g. The above data reveal that the present invention when using less than half the amount of molecular sieve material and a much lower pressure than the prior art, still produces as much or more nitrogen at a level of high purity as the prior art. These improvements are significant The present invention permits the customer who requires only a small quantity of nitrogen of high purity to have the advantages of the PSA technology.

From the above, it is readily seen that the process and apparatus of the present invention is several times more efficient than the prior art and hence provides nitrogen at less cost per unit.

In view of the above, it will be seen that the several objects and advantages of the present invention have been achieved and other advantageous results have been obtained.

As various changes could be made in the above process and apparatus without departing from the scope of the invention, it is intended that all matter contained in the above description or shown in the accompanying drawing shall be interpreted as illustrative and not in a limiting sense.

CLAIMS:

1. An apparatus for the production of high purity nitrogen or higher purity oxygen from air in an amount less than about 6 Nm^3/hr, the apparatus comprising:

(a) a compressor to provide pressurized air or vacuum;

(b) a valve operated distributor suitable for receiving and feeding the pressurized air to an adsorbing vessel or removing gas by vacuum from the adsorbing vessel;

(c) the adsorbing vessel containing molecular sieve material suitable for adsorption of selected gases from pressurized air received from the distributor to provide a pressurized purified nitrogen or oxygen gas stream;

(d) means for controlling the flow of the pressurized purified gas stream; and

(e) means for storing the purified nitrogen or oxygen gas under pressure.

2. The apparatus of Claim 1 wherein nitrogen is the purified gas and the molecular sieve material is carbon molecular sieve material.

3. The apparatus of Claim 1 wherein oxygen is the purified gas and the molecular sieve material is zeolite molecular sieve material.

4. A process for the production of high purity nitrogen gas or high purity oxygen gas from air in an amount less than about 6 Nm^3/hr, the resulting purified gas containing about 3 % or less of residual gas, which process comprises:

(a) placing air under a predetermined pressure;

(b) transporting the pressurized air by means of a valve operated distributor to an adsorbing vessel containing molecular sieve material to separate the air into a purified gas stream under pressure and residual gas entrapped in the molecular sieve material;

(c) removing the purified gas stream under pressure from the adsorbing vessel through a flow control means to a means for storing the purified gas under pressure;

(d) substantially removing the residual gas entrapped in the molecular sieve material by venting the pressurized gas in the adsorbing vessel to the atmosphere, while simultaneously reversing the air flow through the valve operated distributor where the compressor creates a vacuum in the adsorbing vessel;

(e) venting the residual gas from the process, and

(f) repeating steps (a) through (e).

5. The process of Claim 4 wherein the purified gas is nitrogen, the residual gas is substantially oxygen and the molecular sieve material is carbon molecular sieve material.

6. The process of Claim 5 wherein the purified nitrogen gas contains less than 1 % oxygen.

7. The process of Claim 6 wherein the purified nitrogen gas contains less than 0.5 % oxygen.

8. The process of Claim 4 wherein the purified gas is oxygen, the residual gas is substantially nitrogen and argon, and the molecular sieve material is zeolite molecular sieve material.

9. The process of Claim 8 wherein the purified oxygen gas contains less than 1 % nitrogen.

10. The process of Claim 9 wherein the purified oxygen gas contains less than 0.5 % nitrogen.

Application No: GB 9800017.7
Claims searched: All
Examiner: Michael R. Wendt
Date of search: 11 March 1998

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.P): B1L (LAA, LAB, LAF, LAH, LAJ, LAK, LDJ, LDK)

Int Cl (Ed.6): B01D 53/047, 53/053

Other: WPI, Claims, Japio

Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
X	WO 86/05119 A1	(GUILD ASSOC.) e.g see Figure 1. Page 5 lines 16 etc. Abstract.	1 - 5
A	US 4572723	(BERGWERKSVER.) - referred to in application - see whole document.	1, 2, 4 & 5
X	US 4534346	(GUILD ASSOC.) e.g. see Figure 1. Column 2 lines 51 etc.	1, 2, 4 & 5
X	US 4477264	(AIR P. & C.) e.g. See figure. Column 3 lines 7 etc.	1, 3, 4, 8

X Document indicating lack of novelty or inventive step	A Document indicating technological background and/or state of the art.
Y Document indicating lack of inventive step if combined with one or more other documents of same category.	P Document published on or after the declared priority date but before the filing date of this invention.
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