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| APPLICATION NO. | ISSUE DATE | PATENT NO. | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|---|---|---|---|---|
| 15/931,716 | 07/19/2022 | 11389614 | 67853-012 PUS4 | 4948 |

26096 7590 06/29/2022

CARLSON, GASKEY & OLDS, P.C.
400 WEST MAPLE ROAD
SUITE 350
BIRMINGHAM, MI 48009

# ISSUE NOTIFICATION

The projected patent number and issue date are specified above.

**Determination of Patent Term Adjustment under 35 U.S.C. 154 (b)**
(application filed on or after May 29, 2000)

The Patent Term Adjustment is 314 day(s). Any patent to issue from the above-identified application will include an indication of the adjustment on the front page.

If a Continued Prosecution Application (CPA) was filed in the above-identified application, the filing date that determines Patent Term Adjustment is the filing date of the most recent CPA.

Applicant will be able to obtain more detailed information by accessing the Patent Application Information Retrieval (PAIR) WEB site (http://pair.uspto.gov).

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Application Assistance Unit (AAU) of the Office of Patents Stakeholder Experience (OPSE), Stakeholder Support Division (SSD) at (571)-272-4200.

INVENTOR(s) (Please see PAIR WEB site http://pair.uspto.gov for additional inventors):

Theodore W. Jagger, White Bear Lake, MN;
Nicholas P. Van Brunt, White Bear Lake, MN;
John A. Kivisto, Oak Grove, MN;
Perry B. Lonnes, White Bear Lake, MN;

APPLICANT(s) (Please see PAIR WEB site http://pair.uspto.gov for additional applicants):

Vbox, Incorporated, White Bear Lake, MN;

The United States represents the largest, most dynamic marketplace in the world and is an unparalleled location for business investment, innovation, and commercialization of new technologies. The USA offers tremendous resources and advantages for those who invest and manufacture goods here. Through SelectUSA, our nation works to encourage and facilitate business investment. To learn more about why the USA is the best country in the world to develop technology, manufacture products, and grow your business, visit SelectUSA.gov.

## PART B - FEE(S) TRANSMITTAL

Complete and send this form, together with applicable fee(s), by mail or fax, or via EFS-Web.

By mail, send to: Mail Stop ISSUE FEE
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

By fax, send to: (571)-273-2885

INSTRUCTIONS: This form should be used for transmitting the ISSUE FEE and PUBLICATION FEE (if required). Blocks 1 through 5 should be completed where appropriate. All further correspondence including the Patent, advance orders and notification of maintenance fees will be mailed to the current correspondence address as indicated unless corrected below or directed otherwise in Block 1, by (a) specifying a new correspondence address; and/or (b) indicating a separate "FEE ADDRESS" for maintenance fee notifications.

CURRENT CORRESPONDENCE ADDRESS (Note: Use Block 1 for any change of address)

26096 7590 06/08/2022
CARLSON, GASKEY & OLDS, P.C.
400 WEST MAPLE ROAD
SUITE 350
BIRMINGHAM, MI 48009

Note: A certificate of mailing can only be used for domestic mailings of the Fee(s) Transmittal. This certificate cannot be used for any other accompanying papers. Each additional paper, such as an assignment or formal drawing, must have its own certificate of mailing or transmission.

**Certificate of Mailing or Transmission**

I hereby certify that this Fee(s) Transmittal is being deposited with the United States Postal Service with sufficient postage for first class mail in an envelope addressed to the Mail Stop ISSUE FEE address above, or being transmitted to the USPTO via EFS-Web or by facsimile to (571) 273-2885, on the date below.

| | |
|---|---|
| Molly R. Anderson | (Typed or printed name) |
| /Molly R. Anderson/ | (Signature) |
| 06/09/2022 | (Date) |

| APPLICATION NO. | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|---|---|---|---|---|
| 15/931,716 | 05/14/2020 | Theodore W. Jagger | 67853-012 PUS4 | 4948 |

TITLE OF INVENTION: REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR

| APPLN. TYPE | ENTITY STATUS | ISSUE FEE DUE | PUBLICATION FEE DUE | PREV. PAID ISSUE FEE | TOTAL FEE(S) DUE | DATE DUE |
|---|---|---|---|---|---|---|
| nonprovisional | SMALL | $600 | $0.00 | $0.00 | $600 | 09/08/2022 |

| EXAMINER | ART UNIT | CLASS-SUBCLASS |
|---|---|---|
| JONES, CHRISTOPHER P | 1776 | 096-121000 |

1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363).

☐ Change of correspondence address (or Change of Correspondence Address form PTO/AIA/122 or PTO/SB/122) attached.

☐ "Fee Address" indication (or "Fee Address" Indication form PTO/AIA/47 or PTO/SB/47; Rev 03-02 or more recent) attached. **Use of a Customer Number is required.**

2. For printing on the patent front page, list
(1) The names of up to 3 registered patent attorneys or agents OR, alternatively,
(2) The name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.

1 Carlson, Gaskey & Olds, P.C.
2
3

3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document must have been previously recorded, or filed for recordation, as set forth in 37 CFR 3.11 and 37 CFR 3.81(a). Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE: Vbox, Incorporated

(B) RESIDENCE: (CITY and STATE OR COUNTRY): White Bear Lake, MN

Please check the appropriate assignee category or categories (will not be printed on the patent) : ☐ Individual ☒ Corporation or other private group entity ☐ Government

4a. Fees submitted: ☒ Issue Fee ☐ Publication Fee (if required) ☐ Advance Order - # of Copies _____

4b. Method of Payment: *(Please first reapply any previously paid fee shown above)*

☒ Electronic Payment via EFS-Web ☐ Enclosed check ☐ Non-electronic payment by credit card (Attach form PTO-2038)

☒ The Director is hereby authorized to charge the required fee(s), any deficiency, or credit any overpayment to Deposit Account No. 501482

5. **Change in Entity Status** (from status indicated above)

☐ Applicant certifying micro entity status. See 37 CFR 1.29

☐ Applicant asserting small entity status. See 37 CFR 1.27

☐ Applicant changing to regular undiscounted fee status.

NOTE: Absent a valid certification of Micro Entity Status (see forms PTO/SB/15A and 15B), issue fee payment in the micro entity amount will not be accepted at the risk of application abandonment.
NOTE: If the application was previously under micro entity status, checking this box will be taken to be a notification of loss of entitlement to micro entity status.
NOTE: Checking this box will be taken to be a notification of loss of entitlement to small or micro entity status, as applicable.

NOTE: This form must be signed in accordance with 37 CFR 1.31 and 1.33. See 37 CFR 1.4 for signature requirements and certifications.

Authorized Signature /Timothy J. Murphy/ Date 06/09/2022

Typed or printed name Timothy J. Murphy Registration No. 64613



PTOL-85 Part B (08-18) Approved for use through 01/31/2020 OMB 0651-0033 U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

GCE - Exhibit 1071, Page 2
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

# Electronic Patent Application Fee Transmittal

| | |
|---|---|
| **Application Number:** | 15931716 |
| **Filing Date:** | 14-May-2020 |
| **Title of Invention:** | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR |
| **First Named Inventor/Applicant Name:** | Theodore W. Jagger |
| **Filer:** | Timothy J. Murphy/Molly Anderson |
| **Attorney Docket Number:** | 67853-012 PUS4 |

Filed as Small Entity

**Filing Fees for Utility under 35 USC 111(a)**

| Description | Fee Code | Quantity | Amount | Sub-Total in USD($) |
|---|---|---|---|---|
| **Basic Filing:** | | | | |
| **Pages:** | | | | |
| **Claims:** | | | | |
| **Miscellaneous-Filing:** | | | | |
| **Petition:** | | | | |
| **Patent-Appeals-and-Interference:** | | | | |
| **Post-Allowance-and-Post-Issuance:** | | | | |
| UTILITY APPL ISSUE FEE | 2501 | 1 | 600 | 600 |

| Description | Fee Code | Quantity | Amount | Sub-Total in USD($) |
|---|---|---|---|---|
| **Extension-of-Time:** | | | | |
| **Miscellaneous:** | | | | |
| | | | **Total in USD ($)** | **600** |

| Electronic Acknowledgement Receipt | |
|---|---|
| **EFS ID:** | 45913799 |
| **Application Number:** | 15931716 |
| **International Application Number:** | |
| **Confirmation Number:** | 4948 |
| **Title of Invention:** | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR |
| **First Named Inventor/Applicant Name:** | Theodore W. Jagger |
| **Customer Number:** | 26096 |
| **Filer:** | Timothy J. Murphy/Molly Anderson |
| **Filer Authorized By:** | Timothy J. Murphy |
| **Attorney Docket Number:** | 67853-012 PUS4 |
| **Receipt Date:** | 09-JUN-2022 |
| **Filing Date:** | 14-MAY-2020 |
| **Time Stamp:** | 11:09:25 |
| **Application Type:** | Utility under 35 USC 111(a) |

## Payment information:

| | |
|---|---|
| Submitted with Payment | yes |
| Payment Type | DA |
| Payment was successfully received in RAM | $600 |
| RAM confirmation Number | E202269B10251935 |
| Deposit Account | 501482 |
| Authorized User | Molly Anderson |
| The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:<br>37 CFR 1.17 (Patent application and reexamination processing fees)<br>37 CFR 1.19 (Document supply fees) | |

37 CFR 1.20 (Post Issuance fees)

37 CFR 1.21 (Miscellaneous fees and charges)

## File Listing:

| Document Number | Document Description | File Name | File Size(Bytes)/ Message Digest | Multi Part /.zip | Pages (if appl.) |
|---|---|---|---|---|---|
| 1 | Issue Fee Payment (PTO-85B) | Issue_Fee_Transmittal_12PUS4.pdf | 102785<br>274231d8b59f4da5248e5d5305bcff7d99992e5b | no | 1 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 2 | Fee Worksheet (SB06) | fee-info.pdf | 38331<br>e4e443000d62696fe44429b5d617c66bd1f1bfdd | no | 2 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| | | **Total Files Size (in bytes):** | 141116 | | |

**This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.**

**New Applications Under 35 U.S.C. 111**

**If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.**

**National Stage of an International Application under 35 U.S.C. 371**

**If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.**

**New International Application Filed with the USPTO as a Receiving Office**

**If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.**

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# NOTICE OF ALLOWANCE AND FEE(S) DUE

26096 7590 06/08/2022

CARLSON, GASKEY & OLDS, P.C.
400 WEST MAPLE ROAD
SUITE 350
BIRMINGHAM, MI 48009

| EXAMINER |
|---|
| JONES, CHRISTOPHER P |

| ART UNIT | PAPER NUMBER |
|---|---|
| 1776 | |

DATE MAILED: 06/08/2022

| APPLICATION NO. | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|---|---|---|---|---|
| 15/931,716 | 05/14/2020 | Theodore W. Jagger | 67853-012 PUS4 | 4948 |

TITLE OF INVENTION: REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR

| APPLN. TYPE | ENTITY STATUS | ISSUE FEE DUE | PUBLICATION FEE DUE | PREV. PAID ISSUE FEE | TOTAL FEE(S) DUE | DATE DUE |
|---|---|---|---|---|---|---|
| nonprovisional | SMALL | $600 | $0.00 | $0.00 | $600 | 09/08/2022 |

**THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT. PROSECUTION ON THE MERITS IS CLOSED. THIS NOTICE OF ALLOWANCE IS NOT A GRANT OF PATENT RIGHTS. THIS APPLICATION IS SUBJECT TO WITHDRAWAL FROM ISSUE AT THE INITIATIVE OF THE OFFICE OR UPON PETITION BY THE APPLICANT. SEE 37 CFR 1.313 AND MPEP 1308.**

**THE ISSUE FEE AND PUBLICATION FEE (IF REQUIRED) MUST BE PAID WITHIN THREE MONTHS FROM THE MAILING DATE OF THIS NOTICE OR THIS APPLICATION SHALL BE REGARDED AS ABANDONED. THIS STATUTORY PERIOD CANNOT BE EXTENDED. SEE 35 U.S.C. 151. THE ISSUE FEE DUE INDICATED ABOVE DOES NOT REFLECT A CREDIT FOR ANY PREVIOUSLY PAID ISSUE FEE IN THIS APPLICATION. IF AN ISSUE FEE HAS PREVIOUSLY BEEN PAID IN THIS APPLICATION (AS SHOWN ABOVE), THE RETURN OF PART B OF THIS FORM WILL BE CONSIDERED A REQUEST TO REAPPLY THE PREVIOUSLY PAID ISSUE FEE TOWARD THE ISSUE FEE NOW DUE.**

**HOW TO REPLY TO THIS NOTICE:**

I. Review the ENTITY STATUS shown above. If the ENTITY STATUS is shown as SMALL or MICRO, verify whether entitlement to that entity status still applies.

If the ENTITY STATUS is the same as shown above, pay the TOTAL FEE(S) DUE shown above.

If the ENTITY STATUS is changed from that shown above, on PART B - FEE(S) TRANSMITTAL, complete section number 5 titled "Change in Entity Status (from status indicated above)".

For purposes of this notice, small entity fees are 1/2 the amount of undiscounted fees, and micro entity fees are 1/2 the amount of small entity fees.

II. PART B - FEE(S) TRANSMITTAL, or its equivalent, must be completed and returned to the United States Patent and Trademark Office (USPTO) with your ISSUE FEE and PUBLICATION FEE (if required). If you are charging the fee(s) to your deposit account, section "4b" of Part B - Fee(s) Transmittal should be completed. If an equivalent of Part B is filed, a request to reapply a previously paid issue fee must be clearly made, and delays in processing may occur due to the difficulty in recognizing the paper as an equivalent of Part B.

III. All communications regarding this application must give the application number. Please direct all communications prior to issuance to Mail Stop ISSUE FEE unless advised to the contrary.

**IMPORTANT REMINDER: Maintenance fees are due in utility patents issuing on applications filed on or after Dec. 12, 1980. It is patentee's responsibility to ensure timely payment of maintenance fees when due. More information is available at www.uspto.gov/PatentMaintenanceFees.**

## PART B - FEE(S) TRANSMITTAL

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| (Typed or printed name) |
|---|
| (Signature) |
| (Date) |

| APPLICATION NO. | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|---|---|---|---|---|
| 15/931,716 | 05/14/2020 | Theodore W. Jagger | 67853-012 PUS4 | 4948 |

TITLE OF INVENTION: REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR

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| nonprovisional | SMALL | $600 | $0.00 | $0.00 | $600 | 09/08/2022 |

| EXAMINER | ART UNIT | CLASS-SUBCLASS |
|---|---|---|
| JONES, CHRISTOPHER P | 1776 | 096-121000 |

1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363).

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❑ "Fee Address" indication (or "Fee Address" Indication form PTO/AIA/47 or PTO/SB/47; Rev 03-02 or more recent) attached. **Use of a Customer Number is required.**

2. For printing on the patent front page, list
(1) The names of up to 3 registered patent attorneys or agents OR, alternatively,
(2) The name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.

1 _____
2 _____
3 _____

3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document must have been previously recorded, or filed for recordation, as set forth in 37 CFR 3.11 and 37 CFR 3.81(a). Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE (B) RESIDENCE: (CITY and STATE OR COUNTRY)

Please check the appropriate assignee category or categories (will not be printed on the patent) : ❑ Individual ❑ Corporation or other private group entity ❑ Government

4a. Fees submitted: ❑Issue Fee ❑Publication Fee (if required) ❑Advance Order - # of Copies _____

4b. Method of Payment: *(Please first reapply any previously paid fee shown above)*

❑ Electronic Payment via EFS-Web ❑ Enclosed check ❑ Non-electronic payment by credit card (Attach form PTO-2038)

❑ The Director is hereby authorized to charge the required fee(s), any deficiency, or credit any overpayment to Deposit Account No. _____

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❑ Applicant asserting small entity status. See 37 CFR 1.27

❑ Applicant changing to regular undiscounted fee status.

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NOTE: Checking this box will be taken to be a notification of loss of entitlement to small or micro entity status, as applicable.

NOTE: This form must be signed in accordance with 37 CFR 1.31 and 1.33. See 37 CFR 1.4 for signature requirements and certifications.

Authorized Signature _____ Date _____

Typed or printed name _____ Registration No. _____



GCE - Exhibit 1071, Page 8
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
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P.O. Box 1450
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| APPLICATION NO. | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|---|---|---|---|---|
| 15/931,716 | 05/14/2020 | Theodore W. Jagger | 67853-012 PUS4 | 4948 |

26096 7590 06/08/2022

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| EXAMINER | |
|---|---|
| JONES, CHRISTOPHER P | |
| **ART UNIT** | **PAPER NUMBER** |
| 1776 | |

DATE MAILED: 06/08/2022

**Determination of Patent Term Adjustment under 35 U.S.C. 154 (b)**
(Applications filed on or after May 29, 2000)

The Office has discontinued providing a Patent Term Adjustment (PTA) calculation with the Notice of Allowance.

Section 1(h)(2) of the AIA Technical Corrections Act amended 35 U.S.C. 154(b)(3)(B)(i) to eliminate the requirement that the Office provide a patent term adjustment determination with the notice of allowance. See Revisions to Patent Term Adjustment, 78 Fed. Reg. 19416, 19417 (Apr. 1, 2013). Therefore, the Office is no longer providing an initial patent term adjustment determination with the notice of allowance. The Office will continue to provide a patent term adjustment determination with the Issue Notification Letter that is mailed to applicant approximately three weeks prior to the issue date of the patent, and will include the patent term adjustment on the patent. Any request for reconsideration of the patent term adjustment determination (or reinstatement of patent term adjustment) should follow the process outlined in 37 CFR 1.705.

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Customer Service Center of the Office of Patent Publication at 1-(888)-786-0101 or (571)-272-4200.



PTOL-85 (Rev. 02/11)

GCE - Exhibit 1071, Page 9
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

## OMB Clearance and PRA Burden Statement for PTOL-85 Part B

The Paperwork Reduction Act (PRA) of 1995 requires Federal agencies to obtain Office of Management and Budget approval before requesting most types of information from the public. When OMB approves an agency request to collect information from the public, OMB (i) provides a valid OMB Control Number and expiration date for the agency to display on the instrument that will be used to collect the information and (ii) requires the agency to inform the public about the OMB Control Number's legal significance in accordance with 5 CFR 1320.5(b).

The information collected by PTOL-85 Part B is required by 37 CFR 1.311. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 30 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, Virginia 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, Virginia 22313-1450. Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

## Privacy Act Statement

**The Privacy Act of 1974 (P.L. 93-579)** requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether disclosure of these records is required by the Freedom of Information Act.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspection or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

| *Notice of Allowability* | **Application No.** 15/931,716 | **Applicant(s)** Jagger et al. | |
|---|---|---|---|
| | **Examiner** CHRISTOPHER P JONES | **Art Unit** 1776 | **AIA (FITF) Status** No |

***-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address--***

All claims being allowable, PROSECUTION ON THE MERITS IS (OR REMAINS) CLOSED in this application. If not included herewith (or previously mailed), a Notice of Allowance (PTOL-85) or other appropriate communication will be mailed in due course. **THIS NOTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT RIGHTS.** This application is subject to withdrawal from issue at the initiative of the Office or upon petition by the applicant. See 37 CFR 1.313 and MPEP 1308.

1. ☑ This communication is responsive to 05/24/2022.
   ☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2. ☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

3. ☑ The allowed claim(s) is/are 21-36 . As a result of the allowed claim(s), you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information, please see **http://www.uspto.gov/patents/init_events/pph/index.jsp** or send an inquiry to **PPHfeedback@uspto.gov.**

4. ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

   **Certified copies:**

   a) ☐ All b) ☐ Some* c) ☐ None of the:

   1. ☐ Certified copies of the priority documents have been received.
   2. ☐ Certified copies of the priority documents have been received in Application No. _____ .
   3. ☐ Copies of the certified copies of the priority documents have been received in this national stage application from the International Bureau (PCT Rule 17.2(a)).

   * Certified copies not received: _____ .

Applicant has THREE MONTHS FROM THE "MAILING DATE" of this communication to file a reply complying with the requirements noted below. Failure to timely comply will result in ABANDONMENT of this application.
**THIS THREE-MONTH PERIOD IS NOT EXTENDABLE.**

5. ☐ CORRECTED DRAWINGS (as "replacement sheets") must be submitted.
   ☐ including changes required by the attached Examiner's Amendment / Comment or in the Office action of Paper No./Mail Date _____ .

   **Identifying indicia such as the application number (see 37 CFR 1.84(c)) should be written on the drawings in the front (not the back) of each sheet. Replacement sheet(s) should be labeled as such in the header according to 37 CFR 1.121(d).**

6. ☐ DEPOSIT OF and/or INFORMATION about the deposit of BIOLOGICAL MATERIAL must be submitted. Note the attached Examiner's comment regarding REQUIREMENT FOR THE DEPOSIT OF BIOLOGICAL MATERIAL.

**Attachment(s)**

1. ☐ Notice of References Cited (PTO-892)
2. ☐ Information Disclosure Statements (PTO/SB/08), Paper No./Mail Date _____.
3. ☐ Examiner's Comment Regarding Requirement for Deposit of Biological Material _____.
4. ☐ Interview Summary (PTO-413), Paper No./Mail Date. _____.
5. ☐ Examiner's Amendment/Comment
6. ☐ Examiner's Statement of Reasons for Allowance
7. ☐ Other _____.

/CHRISTOPHER P JONES/
Primary Examiner, Art Unit 1776

| Issue Classification | Application/Control No. | Applicant(s)/Patent Under Reexamination |
|---|---|---|
| | 15/931,716 | Jagger et al. |
| | **Examiner** | **Art Unit** |
| | CHRISTOPHER P JONES | 1776 |

| CPC | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Symbol** | | | | | | **Type** | **Version** |
| A61M | / | 16 | / | 101 | | F | 2014-02-04 |
| A62B | / | 19 | / | 00 | | I | 2013-01-01 |
| B01D | / | 53 | / | 0476 | | I | 2013-01-01 |
| A61M | / | 16 | / | 10 | | I | 2013-01-01 |
| A61M | / | 16 | / | 107 | | A | 2014-02-04 |
| A61M | / | 16 | / | 0666 | | A | 2013-01-01 |
| B01D | / | 2259 | / | 455 | | A | 2013-01-01 |
| A61M | / | 2202 | / | 0208 | | A | 2013-01-01 |
| A61M | / | 2202 | / | 03 | | A | 2013-01-01 |

| CPC Combination Sets | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| **Symbol** | | | | | **Type** | **Set** | **Ranking** | **Version** |
| A61M | / | 2202 | / | 0208 | A | 1 | 1 | 2013-01-01 |
| A61M | / | 2202 | / | 0007 | A | 1 | 2 | 2013-01-01 |

| NONE | | **Total Claims Allowed:** | |
|---|---|---|---|
| (Assistant Examiner) | (Date) | 16 | |
| /CHRISTOPHER P JONES/<br>Primary Examiner, Art Unit 1776 | 31 May 2022 | O.G. Print Claim(s) | O.G. Print Figure |
| (Primary Examiner) | (Date) | 1 | 1 |

U.S. Patent and Trademark Office — Part of Paper No.: 20220531



GCE - Exhibit 1071, Page 12
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

| Issue Classification | Application/Control No. | Applicant(s)/Patent Under Reexamination |
|---|---|---|
| | 15/931,716 | Jagger et al. |
| | **Examiner** | **Art Unit** |
| | CHRISTOPHER P JONES | 1776 |

| INTERNATIONAL CLASSIFICATION | | | | |
|---|---|---|---|---|
| **CLAIMED** | | | | |
| B01D | / | 53 | / | 02 |

| NON-CLAIMED | | | | |
|---|---|---|---|---|
| | / | | / | |

| US ORIGINAL CLASSIFICATION | |
|---|---|
| **CLASS** | **SUBCLASS** |
| 96 | 121 |

| CROSS REFERENCES(S) | | | | | | |
|---|---|---|---|---|---|---|
| **CLASS** | **SUBCLASS (ONE SUBCLASS PER BLOCK)** | | | | | |
| | | | | | | |

| NONE | | Total Claims Allowed: | |
|---|---|---|---|
| (Assistant Examiner) | (Date) | 16 | |
| /CHRISTOPHER P JONES/<br>Primary Examiner, Art Unit 1776 | 31 May 2022 | O.G. Print Claim(s) | O.G. Print Figure |
| (Primary Examiner) | (Date) | 1 | 1 |

U.S. Patent and Trademark Office — Part of Paper No.: 20220531



GCE - Exhibit 1071, Page 13
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

| Issue Classification | Application/Control No. | Applicant(s)/Patent Under Reexamination |
|---|---|---|
| | 15/931,716 | Jagger et al. |
| | Examiner | Art Unit |
| | CHRISTOPHER P JONES | 1776 |

☐ Claims renumbered in the same order as presented by applicant ☐ CPA ☑ T.D. ☐ R.1.47

CLAIMS

| Final | Original | Final | Original | Final | Original | Final | Original | Final | Original | Final | Original | Final | Original | Final | Original |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 21 | 10 | 30 | | | | | | | | | | | | |
| 2 | 22 | 11 | 31 | | | | | | | | | | | | |
| 3 | 23 | 12 | 32 | | | | | | | | | | | | |
| 4 | 24 | 13 | 33 | | | | | | | | | | | | |
| 5 | 25 | 14 | 34 | | | | | | | | | | | | |
| 6 | 26 | 15 | 35 | | | | | | | | | | | | |
| 7 | 27 | 16 | 36 | | | | | | | | | | | | |
| 8 | 28 | | | | | | | | | | | | | | |
| 9 | 29 | | | | | | | | | | | | | | |

| | | | |
|---|---|---|---|
| NONE | | **Total Claims Allowed:** | |
| (Assistant Examiner) | (Date) | 16 | |
| /CHRISTOPHER P JONES/<br>Primary Examiner, Art Unit 1776 | 31 May 2022 | O.G. Print Claim(s) | O.G. Print Figure |
| (Primary Examiner) | (Date) | 1 | 1 |

U.S. Patent and Trademark Office Part of Paper No.: 20220531



GCE - Exhibit 1071, Page 14
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

| Search Notes | Application/Control No. | Applicant(s)/Patent Under Reexamination |
|---|---|---|
| | 15/931,716 | Jagger et al. |
| | Examiner | Art Unit |
| | CHRISTOPHER P JONES | 1776 |

| CPC - Searched* | | |
|---|---|---|
| **Symbol** | **Date** | **Examiner** |
| A61M2202/0208, 0007 | 04/06/2022 | CPJ |
| A61M16/0666, 10, 101, 107 | 04/06/2022 | CPJ |
| A61M2202/03 | 04/06/2022 | CPJ |
| A62B19/00 | 04/06/2022 | CPJ |
| B01D2259/455 | 04/06/2022 | CPJ |
| B01D53/0476 | 04/06/2022 | CPJ |

| CPC Combination Sets - Searched* | | |
|---|---|---|
| **Symbol** | **Date** | **Examiner** |
| | | |

| US Classification - Searched* | | | |
|---|---|---|---|
| **Class** | **Subclass** | **Date** | **Examiner** |
| | | | |

* See search history printout included with this form or the SEARCH NOTES box below to determine the scope of the search.

| Search Notes | | |
|---|---|---|
| **Search Notes** | **Date** | **Examiner** |
| Inventor search | 04/06/2022 | CPJ |
| Text search | 04/06/2022 | CPJ |

| /CHRISTOPHER P JONES/<br>Primary Examiner, Art Unit 1776 | |
|---|---|



GCE - Exhibit 1071, Page 15
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

| Search Notes | Application/Control No. | Applicant(s)/Patent Under Reexamination |
|---|---|---|
| | 15/931,716 | Jagger et al. |
| | Examiner | Art Unit |
| | CHRISTOPHER P JONES | 1776 |

| Interference Search | | | |
|---|---|---|---|
| **US Class/CPC Symbol** | **US Subclass/CPC Group** | **Date** | **Examiner** |
| A61M2202 | 0208, 0007 | 05/31/2022 | CPJ |
| A61M16 | 0666, 10, 101, 107 | 05/31/2022 | CPJ |
| A61M2202 | 03 | 05/31/2022 | CPJ |
| A62B19 | 00 | 05/31/2022 | CPJ |
| B01D2259 | 455 | 05/31/2022 | CPJ |
| B01D53 | 0476 | 05/31/2022 | CPJ |

| | |
|---|---|
| /CHRISTOPHER P JONES/<br>Primary Examiner, Art Unit 1776 | |



GCE - Exhibit 1071, Page 16
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

| *Application Number* * 15/931,716 * | Application/Control No. 15/931,716 | Applicant(s)/Patent under Reexamination Jagger et al. | |
|---|---|---|---|
| | Examiner JONES, CHRISTOPHER P | Art Unit 1776 | |

| Document Code - DISQ | Internal Document - DO NOT MAIL |
|---|---|

| TERMINAL DISCLAIMER | ☑ APPROVED | ☐ DISAPPROVED |
|---|---|---|
| Date Filed: 24 May 2022 | This patent is subject to a Terminal Disclaimer | |

**Approved/Disapproved by:**

/PAMELA A YOUNG/

**Technology Center:** OPLC

**Telephone:** (571)272-3622

2 TD

| *Application Number* * 15/931,716 * | Application/Control No. 15/931,716 | Applicant(s)/Patent under Reexamination Jagger et al. | |
|---|---|---|---|
| | Examiner JONES, CHRISTOPHER P | Art Unit 1776 | |

| Document Code - DISQ | Internal Document - DO NOT MAIL |
|---|---|

| TERMINAL DISCLAIMER | ☑ APPROVED | ☐ DISAPPROVED |
|---|---|---|
| Date Filed: 24 May 2022 | This patent is subject to a Terminal Disclaimer | |

**Approved/Disapproved by:**

/PAMELA A YOUNG/

**Technology Center: OPLC**

**Telephone: (571)272-3622**

2 TD

UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

| APPLICATION NO. | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|---|---|---|---|---|
| 15/931,716 | 05/14/2020 | Theodore W. Jagger | 67853-012 PUS4 | 4948 |

26096 7590 05/24/2022

CARLSON, GASKEY & OLDS, P.C.
400 WEST MAPLE ROAD
SUITE 350
BIRMINGHAM, MI 48009

| EXAMINER | |
|---|---|
| JONES, CHRISTOPHER P | |

| ART UNIT | PAPER NUMBER |
|---|---|
| 1776 | |

| NOTIFICATION DATE | DELIVERY MODE |
|---|---|
| 05/24/2022 | ELECTRONIC |

**Please find below and/or attached an Office communication concerning this application or proceeding.**

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

cgolaw@yahoo.com
ptodocket@cgolaw.com

| *Office Action Summary* | **Application No.** 15/931,716 | **Applicant(s)** Jagger et al. | |
|---|---|---|---|
| | **Examiner** CHRISTOPHER P JONES | **Art Unit** 1776 | **AIA (FITF) Status** No |

*-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --*

**Period for Reply**

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTHS FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

**Status**

1)☑ Responsive to communication(s) filed on 07/23/2020.

☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on _____.

2a)☐ This action is **FINAL.** 2b) ☑ This action is non-final.

3)☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on _____; the restriction requirement and election have been incorporated into this action.

4)☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

**Disposition of Claims***

5) ☑ Claim(s) 21-36 is/are pending in the application.

5a) Of the above claim(s) _____ is/are withdrawn from consideration.

6) ☐ Claim(s) _____ is/are allowed.

7) ☑ Claim(s) 21-36 is/are rejected.

8) ☐ Claim(s) _____ is/are objected to.

9) ☐ Claim(s) _____ are subject to restriction and/or election requirement

* If any claims have been determined allowable, you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information, please see http://www.uspto.gov/patents/init_events/pph/index.jsp or send an inquiry to **PPHfeedback@uspto.gov.**

**Application Papers**

10)☐ The specification is objected to by the Examiner.

11)☑ The drawing(s) filed on 05/14/2020 is/are: a)☑ accepted or b)☐ objected to by the Examiner.

Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).

Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).

**Priority under 35 U.S.C. § 119**

12)☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

**Certified copies:**

a)☐ All b)☐ Some** c)☐ None of the:

1.☐ Certified copies of the priority documents have been received.

2.☐ Certified copies of the priority documents have been received in Application No. _____.

3.☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

** See the attached detailed Office action for a list of the certified copies not received.

**Attachment(s)**

1) ☐ Notice of References Cited (PTO-892)

2) ☑ Information Disclosure Statement(s) (PTO/SB/08a and/or PTO/SB/08b) Paper No(s)/Mail Date _____.

3) ☐ Interview Summary (PTO-413) Paper No(s)/Mail Date _____.

4) ☐ Other: _____.

**DETAILED ACTION**

***Notice of Pre-AIA or AIA Status***

1. The present application is being examined under the pre-AIA first to invent provisions.

***Double Patenting***

2. The nonstatutory double patenting rejection is based on a judicially created doctrine grounded in public policy (a policy reflected in the statute) so as to prevent the unjustified or improper timewise extension of the "right to exclude" granted by a patent and to prevent possible harassment by multiple assignees. A nonstatutory double patenting rejection is appropriate where the conflicting claims are not identical, but at least one examined application claim is not patentably distinct from the reference claim(s) because the examined application claim is either anticipated by, or would have been obvious over, the reference claim(s). See, e.g., *In re Berg*, 140 F.3d 1428, 46 USPQ2d 1226 (Fed. Cir. 1998); *In re Goodman*, 11 F.3d 1046, 29 USPQ2d 2010 (Fed. Cir. 1993); *In re Longi*, 759 F.2d 887, 225 USPQ 645 (Fed. Cir. 1985); *In re Van Ornum*, 686 F.2d 937, 214 USPQ 761 (CCPA 1982); *In re Vogel*, 422 F.2d 438, 164 USPQ 619 (CCPA 1970); *In re Thorington*, 418 F.2d 528, 163 USPQ 644 (CCPA 1969).

A timely filed terminal disclaimer in compliance with 37 CFR 1.321(c) or 1.321(d) may be used to overcome an actual or provisional rejection based on nonstatutory double patenting provided the reference application or patent either is shown to be commonly owned with the examined application, or claims an invention made as a result of activities undertaken within the scope of a joint research agreement. See MPEP § 717.02 for applications subject to examination under the first inventor to file provisions of the AIA as explained in MPEP § 2159. See MPEP § 2146 *et seq.* for applications not subject to examination under the first inventor to file provisions of the AIA. A terminal disclaimer must be signed in compliance with 37 CFR 1.321(b).

The USPTO Internet website contains terminal disclaimer forms which may be used. Please visit www.uspto.gov/patent/patents-forms. The filing date of the application in which the form is filed determines what form (e.g., PTO/SB/25, PTO/SB/26, PTO/AIA/25, or PTO/AIA/26) should be used. A web-based eTerminal Disclaimer may be filled out completely online using web-screens. An eTerminal Disclaimer that meets all requirements is auto-processed and approved immediately upon submission. For more information about eTerminal Disclaimers, refer to www.uspto.gov/patents/process/file/efs/guidance/eTD-info-I.jsp.

3. Claims 21-32 are rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-23 of U.S. Patent No. 10,357,628 B2. Although the claims at issue are not identical, they are not patentably distinct from each other because all the limitations of claims 21-32 are found in claims 1-23 of USPN 10,357,628 B2. It is noted that claims 1-23 of USPN 10,357,628 B2 disclose that the bed of adsorbent is either in the line of the inlet or the line of the outlet. This anticipates the current claims where the adsorbent bed is in the line of the outlet.

4. Claims 33-36 are rejected on the ground of nonstatutory double patenting as being unpatentable over claims 1-17 of U.S. Patent No. 10,702,669 B2. Although the claims at issue are not identical, they are not patentably distinct from each other because all the limitations of claims 33-36 are found in claims 1-17 of U.S. Patent No. 10,702,669 B2.

***Conclusion***

5. Any inquiry concerning this communication or earlier communications from the examiner should be directed to CHRISTOPHER P JONES whose telephone number is (571)270-7383. The examiner can normally be reached 9AM-6PM EST M-F.

Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at http://www.uspto.gov/interviewpractice.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Jennifer Michener can be reached on (571)272-1424. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of published or unpublished applications may be obtained from Patent Center. Unpublished application information in Patent Center is available to registered users. To file and manage patent submissions in Patent Center, visit: https://patentcenter.uspto.gov. Visit https://www.uspto.gov/patents/apply/patent-center for more information about Patent Center and https://www.uspto.gov/patents/docx for information about filing in DOCX format. For additional questions, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/CHRISTOPHER P JONES/
Primary Examiner, Art Unit 1776

| Search Notes | Application/Control No. | Applicant(s)/Patent Under Reexamination |
|---|---|---|
| | 15/931,716 | Jagger et al. |
| | Examiner | Art Unit |
| | CHRISTOPHER P JONES | 1776 |

| CPC - Searched* | | |
|---|---|---|
| **Symbol** | **Date** | **Examiner** |
| A61M2202/0208, 0007 | 04/06/2022 | CPJ |
| A61M16/0666, 10, 101, 107 | 04/06/2022 | CPJ |
| A61M2202/03 | 04/06/2022 | CPJ |
| A62B19/00 | 04/06/2022 | CPJ |
| B01D2259/455 | 04/06/2022 | CPJ |
| B01D53/0476 | 04/06/2022 | CPJ |

| CPC Combination Sets - Searched* | | |
|---|---|---|
| **Symbol** | **Date** | **Examiner** |
| | | |

| US Classification - Searched* | | | |
|---|---|---|---|
| **Class** | **Subclass** | **Date** | **Examiner** |
| | | | |

* See search history printout included with this form or the SEARCH NOTES box below to determine the scope of the search.

| Search Notes | | |
|---|---|---|
| **Search Notes** | **Date** | **Examiner** |
| Inventor search | 04/06/2022 | CPJ |
| Text search | 04/06/2022 | CPJ |

| /CHRISTOPHER P JONES/<br>Primary Examiner, Art Unit 1776 | |
|---|---|



GCE - Exhibit 1071, Page 24
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

| Search Notes | Application/Control No. | Applicant(s)/Patent Under Reexamination |
|---|---|---|
| | 15/931,716 | Jagger et al. |
| | Examiner | Art Unit |
| | CHRISTOPHER P JONES | 1776 |

| Interference Search | | | |
|---|---|---|---|
| US Class/CPC Symbol | US Subclass/CPC Group | Date | Examiner |
| | | | |

| | |
|---|---|
| /CHRISTOPHER P JONES/<br>Primary Examiner, Art Unit 1776 | |



GCE - Exhibit 1071, Page 25
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

**EAST Search History**

**EAST Search History (Prior Art)**

| Ref # | Hits | Search Query | DBs | Default Operator | Plurals | Time Stamp |
|---|---|---|---|---|---|---|
| L1 | 2 | (("7841343") or ("6605136")).PN. | US-PGPUB; USPAT; USOCR | OR | OFF | 2022/04/06 17:34 |
| S1 | 49 | ((Theodore) near2 (Jagger)).INV. | US-PGPUB; USPAT | OR | ON | 2015/05/08 09:04 |
| S2 | 92 | ((Nicholas) near2 ("Van Brunt")).INV. | US-PGPUB; USPAT | OR | ON | 2015/05/08 09:04 |
| S3 | 51 | ((John) near2 (Kivisto)).INV. | US-PGPUB; USPAT | OR | ON | 2015/05/08 09:04 |
| S4 | 36 | ((Perry) near2 (Lonnes)).INV. | US-PGPUB; USPAT | OR | ON | 2015/05/08 09:04 |
| S5 | 1 | ("13795977").PN. | US-PGPUB; USPAT; USOCR | OR | OFF | 2015/05/08 09:19 |
| S6 | 2 | (("6585804") or ("6651658")).PN. | US-PGPUB; USPAT; USOCR | OR | OFF | 2015/05/08 09:19 |
| S7 | 2 | "13795977" | US-PGPUB; USPAT; USOCR | OR | ON | 2015/05/08 09:55 |
| S8 | 3,771 | (A61M16/0666 OR A61M16/10 OR A61M16/101 OR A62B19/00 OR B01D2259/455 OR B01D53/0476).CPC. | US-PGPUB; USPAT; USOCR | OR | ON | 2015/05/08 09:55 |
| S9 | 2 | ("11054341").PN. | US-PGPUB; USPAT; USOCR | OR | OFF | 2015/05/08 09:55 |
| S10 | 1 | ("13096362").PN. | US-PGPUB; USPAT; USOCR | OR | OFF | 2015/05/08 09:58 |
| S11 | 59 | (oxygen with concentrator) and lilsx | US-PGPUB; USPAT; USOCR | OR | ON | 2015/05/08 09:59 |
| S12 | 56 | (oxygen with concentrator) and lilsx and ((vacuum with swing) or vsa) | US-PGPUB; USPAT; USOCR | OR | ON | 2015/05/08 09:59 |
| S13 | 50 | (oxygen with concentrator) same portable and lilsx and ((vacuum with swing) or vsa) | US-PGPUB; USPAT; USOCR | OR | ON | 2015/05/08 10:01 |

| | | | | | | |
|---|---|---|---|---|---|---|
| S14 | 159 | (oxygen with concentrator) same portable and ((vacuum with swing) or vsa) | US-PGPUB; USPAT; USOCR | OR | ON | 2015/05/08 10:02 |
| S15 | 32 | (oxygen with concentrator) same portable and ((vacuum with swing) or vsa) and cartridge | US-PGPUB; USPAT; USOCR | OR | ON | 2015/05/08 10:02 |
| S16 | 1 | ("5413625").PN. | US-PGPUB; USPAT; USOCR | OR | OFF | 2015/05/08 10:46 |
| S17 | 1 | ("5268021").PN. | US-PGPUB; USPAT; USOCR | OR | OFF | 2015/05/08 11:25 |
| S18 | 1 | S6 and (motor same valve) | US-PGPUB; USPAT; USOCR | OR | ON | 2015/05/08 11:53 |
| S19 | 103 | S1 S2 S3 S4 | US-PGPUB; USPAT; USOCR | OR | ON | 2015/05/08 12:38 |
| S20 | 23 | S19 and (oxygen with concentrator).clm. | US-PGPUB; USPAT; USOCR | OR | ON | 2015/05/08 12:38 |
| S21 | 1 | ("6651658").PN. | US-PGPUB; USPAT; USOCR | OR | OFF | 2015/11/06 10:20 |
| S22 | 35 | (oxygen with concentrator) and ((vacuum with swing) or vsa) and (single near2 valve) | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 13:06 |
| S23 | 33 | (oxygen with concentrator) and ((vacuum with swing) or vsa) and (three$way near2 valve) | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 13:10 |
| S24 | 144 | (oxygen with concentrator) and (single near2 valve) | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 13:36 |
| S25 | 43 | (oxygen with concentrator) and (single near2 valve) with connect$4 | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:30 |
| S26 | 17 | (oxygen with concentrator) and (single near2 valve) with connect$4 not jagger | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:31 |
| S27 | 1 | ("13795977").PN. | US-PGPUB; USPAT; USOCR | OR | OFF | 2015/11/06 14:32 |
| S28 | 26 | (oxygen with concentrator) and (main near2 valve) with connect$4 not jagger | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:37 |

| | | | | | | |
|---|---|---|---|---|---|---|
| S29 | 0 | (oxygen with concentrator) and (main near2 valve) with connect$4 with inlet with outlet with separat$4 not jagger | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:40 |
| S30 | 185 | (main near2 valve) with connect$4 with inlet with outlet with separat$4 not jagger | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:41 |
| S31 | 89,499 | (("95") or ("96") or ("55")).CLAS. | US-PGPUB; USPAT; USOCR | OR | OFF | 2015/11/06 14:41 |
| S32 | 4 | S30 and S31 | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:41 |
| S33 | 82 | (single near2 valve) with connect$4 with inlet with outlet with separat$4 not jagger | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:42 |
| S34 | 4 | S31 and S33 | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:42 |
| S35 | 49 | ((Theodore) near2 (Jagger)).INV. | US-PGPUB; USPAT | OR | ON | 2015/11/06 14:54 |
| S36 | 26 | (single near2 valve) and S35 | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:54 |
| S37 | 0 | (single near2 valve).clm. and S35 | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:54 |
| S38 | 30,409 | (b01j20/00-3491, b01d53/02-185).cpc. | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:55 |
| S39 | 10,463 | (b01d2252/00-61, b01d2253/00-3425, y02c10/06-08).cpc. | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:56 |
| S40 | 31,398 | S38 or S39 | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:56 |
| S41 | 0 | S40 and S33 | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:56 |
| S42 | 5 | (single near2 valve) with connect$4 with inlet with outlet with adsorbent not jagger | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:58 |
| S43 | 50 | ("2944627" \| "3313091" \| "3396904" \| "3812882" \| "3923477" \| "4272265" \| "4399838" \| "4469494" \| "4532961" \| "4534346" \| "4554948" \| "4674537" \| "4787417" \| "4892566" \| "4900338" \| "4925464" \| "5074333" \| "5112367" \| | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:59 |

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| | | "5127438" \| "5268021" \| "5366541" \| "5370728" \| "5417083" \| "5593478" \| "5658371" \| "5679134" \| "5730778" \| "5785740" \| "5807423" \| "5814130" \| "5814131" \| "5876485" \| "5912426" \| "6056804" \| "6143056" \| "6192922" \| "6253778" \| "6311719" \| "6344069" \| "6457485" \| "6514319" \| "6936091" \| "7152620").PN. OR ("8210205").URPN. | | | | |
| S44 | 34 | S43 and S31 | US-PGPUB; USPAT; USOCR | OR | ON | 2015/11/06 14:59 |
| S45 | 1 | ("7621980").PN. | US-PGPUB; USPAT; USOCR | OR | OFF | 2015/11/09 09:32 |
| S46 | 1 | ("20070207917").PN. | US-PGPUB; USPAT; USOCR | OR | OFF | 2015/11/09 10:50 |
| S47 | 1 | "15931716" | US-PGPUB; USPAT | OR | ON | 2022/03/10 15:33 |
| S48 | 3,671 | adsorbant | US-PGPUB; USPAT | OR | ON | 2022/03/11 10:45 |
| S49 | 105,007 | adsorbent | US-PGPUB; USPAT | OR | ON | 2022/03/11 10:45 |
| S50 | 125 | ((("JAGGER") near3 ("Theodore")) OR (("VAN") near3 ("BRUNT") near3 ("Nicholas")) OR (("KIVISTO") near3 ("John")) OR (("LONNES") near3 ("Perry"))).INV. | US-PGPUB; USPAT; USOCR; EPO; JPO; DERWENT | OR | ON | 2022/04/06 11:52 |
| S51 | 28 | S50 and (oxygen near2 concentrator).clm. | US-PGPUB; USPAT | OR | ON | 2022/04/06 11:52 |
| S52 | 1 | "15931716" | US-PGPUB; USPAT | OR | ON | 2022/04/06 12:45 |
| S53 | 11,742 | (A61M2202/0208 OR A61M2202/0007 OR A61M16/0666 OR A61M16/10 OR A61M16/101 OR A61M16/107 OR A61M2202/03 OR A62B19/00 OR B01D2259/455 OR B01D53/0476).CPC. | US-PGPUB; USPAT | OR | ON | 2022/04/06 12:46 |
| S54 | 628 | (oxygen with concentrator) and portable and bed and (inlet and outlet) | US-PGPUB; USPAT | OR | ON | 2022/04/06 12:51 |
| S55 | 412 | (oxygen with concentrator) and portable and bed and (inlet and outlet) and (sorbent or adsorbent or absorbent) | US-PGPUB; USPAT | OR | ON | 2022/04/06 12:51 |
| S56 | 295 | S53 and S55 | US-PGPUB; USPAT | OR | ON | 2022/04/06 12:51 |
| S57 | 76 | S53 and S55 and cartridge | US-PGPUB; USPAT | OR | ON | 2022/04/06 12:51 |

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|---|---|---|---|---|---|---|
| S58 | 12,008 | (A61M2202/0208 OR A61M2202/0007 OR A61M16/0666 OR A61M16/10 OR A61M16/101 OR A61M16/107 OR A61M2202/03 OR A62B19/00 OR B01D2259/455 OR B01D53/0476).CPC. | US-PGPUB; USPAT; USOCR | OR | ON | 2022/04/06 12:52 |
| S59 | 412 | (oxygen with concentrator) and portable and bed and (inlet and outlet) and (sorbent or adsorbent or absorbent) | US-PGPUB; USPAT; USOCR | OR | ON | 2022/04/06 12:52 |
| S60 | 76 | S58 and S59 and cartridge | US-PGPUB; USPAT; USOCR | OR | ON | 2022/04/06 12:52 |
| S61 | 248 | ("10265491" \| "10343139" \| "10384028" \| "10478770" \| "10576455" \| "20030005928" \| "20030106430" \| "20030167924" \| "20040103894" \| "20050072298" \| "20050072306" \| "20050072423" \| "20050072426" \| "20050103341" \| "20050257686" \| "20050274381" \| "20060090759" \| "20060124128" \| "20060174871" \| "20060174875" \| "20060174877" \| "20060174880" \| "20060174881" \| "20060230924" \| "20060230928" \| "20060283325" \| "20060292654" \| "20070056583" \| "20070056584" \| "20070289446" \| "20080000353" \| "20080072907" \| "20080105258" \| "20080110338" \| "20080168901" \| "20080202337" \| "20080282883" \| "20090071333" \| "20090126736" \| "20090131763" \| "20100282084" \| "20100294133" \| "20110023712" \| "20110067566" \| "20110271833" \| "20110275866" \| "20120167883" \| "20120167886" \| "20120167887" \| "20120167888" \| "20120266883" \| "20130019752" \| "20130269519" \| "20130299023" \| "20130305929" \| "20140033918" \| "20140137741" \| "20140353886" \| "20150107585" \| "20150107592" \| "20150182720" \| "20150250973" \| "20150360167" \| "20160022950" \| "20170113013" \| "20170143926" \| "20170239640" \| "20170239641" \| "20170340851" \| "20170348501" \| "20170361052" \| "20180001048" \| "20180200474" \| "20180200475" \| "20180364119" \| "20190091651" \| "20190175860" \| "20190193018" \| "20190201835" \| "20190291078" \| "20200054986" \| | US-PGPUB; USPAT | OR | ON | 2022/04/06 13:15 |

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| | | "20200054987" \| "20200054988" \|<br>"3121625" \| "3236028" \| "3717974" \|<br>"3880616" \| "3922149" \| "4169715" \|<br>"4194890" \| "4222750" \| "4263018" \|<br>"4354859" \| "4386945" \| "4448592" \|<br>"4449990" \| "4472177" \| "4491459" \|<br>"4502873" \| "4576616" \| "4584001" \|<br>"4627860" \| "4648395" \| "4648888" \|<br>"4673415" \| "4681099" \| "4684377" \|<br>"4702903" \| "4801308" \| "4802899" \|<br>"4813977" \| "4816121" \| "4826510" \|<br>"4880443" \| "4892566" \| "4925460" \|<br>"4925464" \| "4948401" \| "4968329" \|<br>"4971609" \| "4973339" \| "5002591" \|<br>"5034023" \| "5069688" \| "5071449" \|<br>"5082473" \| "5084075" \| "5096469" \|<br>"5112367" \| "5114440" \| "5169506" \|<br>"5186793" \| "5228840" \| "5247826" \|<br>"5256172" \| "5266102" \| "5268021" \|<br>"5268023" \| "5275642" \| "5328503" \|<br>"5330561" \| "5340381" \| "5366541" \|<br>"5370112" \| "5411578" \| "5415683" \|<br>"5429666" \| "5441558" \| "5469372" \|<br>"5474595" \| "5487775" \| "5531807" \|<br>"5540758" \| "5565018" \| "5578115" \|<br>"5593478" \| "5656066" \| "5656067" \|<br>"5672195" \| "5711787" \| "5730778" \|<br>"5753010" \| "5762686" \| "5772737" \|<br>"5779766" \| "5797979" \| "5814130" \|<br>"5814131" \| "5820656" \| "5827358" \|<br>"5858062" \| "5871564" \| "5882380" \|<br>"5891217" \| "5893275" \| "5893944" \|<br>"5906672" \| "5917135" \| "5928189" \|<br>"5931159" \| "5935297" \| "5948142" \|<br>"5968236" \| "5979440" \| "5985001" \|<br>"5985113" \| "5988165" \| "5997611" \|<br>"5997617" \| "6010555" \| "6017315" \|<br>"6030435" \| "6033457" \| "6051050" \|<br>"6056804" \| "6068680" \| "6093379" \|<br>"6096115" \| "6146447" \| "6152134" \|<br>"6156101" \| "6162283" \| "6171371" \|<br>"6176897" \| "6183538" \| "6212904" \|<br>"6217635" \| "6287366" \| "6290759" \|<br>"6302107" \| "6311719" \| "6314957" \|<br>"6344069" \| "6346139" \| "6372026" \|<br>"6425938" \| "6428607" \| "6432170" \|<br>"6446630" \| "6457485" \| "6461410" \|<br>"6468328" \| "6471744" \| "6478850" \|<br>"6478857" \| "6511526" \| "6520176" \|<br>"6521143" \| "6524370" \| "6544318" \|<br>"6547851" \| "6551384" \| "6572688" \|<br>"6576043" \| "6585804" \| "6605136" \|<br>"6629525" \| "6641644" \| "6641645" \| | | | |

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|---|---|---|---|---|---|---|
| | | "6651653" \| "6651658" \| "6658894" \| "6687164" \| "6691702" \| "6698423" \| "6712087" \| "6712886" \| "6764534").PN. | | | | |
| S62 | 101 | ("6793719" \| "6802889" \| "6805729" \| "6811590" \| "6824590" \| "6866041" \| "6866950" \| "6878186" \| "6908503" \| "6935460" \| "6949133" \| "6953498" \| "6959728" \| "7011092" \| "7011898" \| "7025329" \| "7066985" \| "7066986" \| "7105038" \| "7135059" \| "7156903" \| "7171963" \| "7273051" \| "7279029" \| "7285154" \| "7291271" \| "7329304" \| "7350521" \| "7368005" \| "7390350" \| "7402193" \| "7425231" \| "7431032" \| "7438745" \| "7445663" \| "7452407" \| "7455717" \| "7473299" \| "7491040" \| "7500490" \| "7510601" \| "7517385" \| "7533872" \| "7585351" \| "7604004" \| "7604005" \| "7651549" \| "7686870" \| "7708802" \| "7713421" \| "7717981" \| "7722698" \| "7722700" \| "7730887" \| "7753996" \| "7758672" \| "7766010" \| "7771519" \| "7780768" \| "7794522" \| "7841343" \| "7857894" \| "7866315" \| "7879138" \| "7922789" \| "7947118" \| "8016918" \| "8142544" \| "8148583" \| "8262783" \| "8303683" \| "8366815" \| "8377181" \| "8440004" \| "8529674" \| "8568519" \| "8580015" \| "8603220" \| "8702841" \| "8753426" \| "8858689" \| "9101872" \| "9199190" \| "9220864" \| "9283346" \| "9592360" \| "9669349" \| "9731241" \| "9907926" \| "9925514" \| "9995645" \| "D510169" \| "D528212" \| "D700701" \| "D706211" \| "D749220" \| "D770608" \| "D851767" \| "D860459" \| "D876615" \| "RE35099").PN. | US-PGPUB; USPAT | OR | ON | 2022/04/06 13:15 |
| S63 | 0 | S61 and S62 and (portable same oxygen near2 concentrator) | US-PGPUB; USPAT | OR | ON | 2022/04/06 13:16 |
| S64 | 129 | (S61 or S62) and (portable same oxygen near2 concentrator) | US-PGPUB; USPAT | OR | ON | 2022/04/06 13:16 |
| S65 | 114 | S64 not S50 | US-PGPUB; USPAT | OR | ON | 2022/04/06 13:17 |
| S66 | 109 | S65 not S60 | US-PGPUB; USPAT | OR | ON | 2022/04/06 13:23 |
| S67 | 4 | "10357628" | US-PGPUB; USPAT | OR | ON | 2022/04/06 13:39 |

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|---|---|---|---|---|---|---|
| S68 | 2 | (("10357628") or ("10702669")).PN. | US-PGPUB; USPAT; USOCR | OR | OFF | 2022/04/06 13:39 |
| S69 | 2 | S68 not S61 not S62 | US-PGPUB; USPAT; USOCR | OR | ON | 2022/04/06 13:42 |

**EAST Search History (Interference)**

<This search history is empty>

4/6/2022 6:01:32 PM
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Doc code: IDS
Doc description: Information Disclosure Statement (IDS) Filed

PTO/SB/08a (01-10)
Approved for use through 07/31/2012. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
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**INFORMATION DISCLOSURE STATEMENT BY APPLICANT**
**( Not for submission under 37 CFR 1.99)**

| | |
|---|---|
| Application Number | |
| Filing Date | |
| First Named Inventor | Theodore W. Jagger |
| Art Unit | |
| Examiner Name | |
| Attorney Docket Number | 67853-012 PUS4 |

**U.S.PATENTS** Remove

| Examiner Initial* | Cite No | Patent Number | Kind Code[1] | Issue Date | Name of Patentee or Applicant of cited Document | Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear |
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| INFORMATION DISCLOSURE STATEMENT BY APPLICANT ( Not for submission under 37 CFR 1.99) | | |
|---|---|---|
| | Application Number | |
| | Filing Date | |
| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

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EFS Web 2.1.17

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| INFORMATION DISCLOSURE STATEMENT BY APPLICANT ( Not for submission under 37 CFR 1.99) | | |
|---|---|---|
| | Application Number | |
| | Filing Date | |
| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

| | | | | | | |
|---|---|---|---|---|---|---|
| | 20 | 4648888 | | 1987-03-10 | Robert O. Rowland | |
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| INFORMATION DISCLOSURE STATEMENT BY APPLICANT ( Not for submission under 37 CFR 1.99) | | |
|---|---|---|
| | Application Number | |
| | Filing Date | |
| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

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| | 37 | 5002591 | | 1991-03-26 | Raymond A. Stanford | |
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| | 40 | 5071449 | | 1991-12-10 | Shivaji Sircar | |
| | 41 | 5082473 | | 1992-01-21 | Bowie G. Keefer | |



ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /C.P.J/

| INFORMATION DISCLOSURE STATEMENT BY APPLICANT ( Not for submission under 37 CFR 1.99) | | |
|---|---|---|
| | Application Number | |
| | Filing Date | |
| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

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| | 44 | 5112367 | | 1992-05-12 | Charles C. Hill | |
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EFS Web 2.1.17

ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /C.P.J/

| INFORMATION DISCLOSURE STATEMENT BY APPLICANT ( Not for submission under 37 CFR 1.99) | | |
|---|---|---|
| | Application Number | |
| | Filing Date | |
| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

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Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

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See attached certification statement.

The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.

☒ A certification statement is not submitted herewith.

**SIGNATURE**

A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

| Signature | /Timothy J. Murphy/ | Date (YYYY-MM-DD) | 2020-05-14 |
|---|---|---|---|
| Name/Print | Timothy J. Murphy | Registration Number | 64,613 |

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5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.

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7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.

8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.

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Application No: 15/931,716

Foreign Priority claimed: ○ Yes ◉ No

35 USC 119 (a-d) conditions met: ☐ Yes ☐ No ☐ Met After Allowance

Verified and Acknowledged: /CHRISTOPHER P JONES/ (Examiner's Signature) | (Initials)

Title: REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR

| FILING or 371(c) DATE | CLASS | GROUP ART UNIT | ATTORNEY DOCKET NO. |
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| 05/14/2020 | 096 | 1776 | 67853-012 PUS4 |
| RULE | | | |

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**CONTINUING DATA**

This application is a CON of 14193504 02/28/2014 PAT 10702669

14193504 is a CON of 13795977 03/12/2013ABN

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05/21/2020

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Doc code: IDS
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| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

[1] See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. [2] Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). [3] For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. [4] Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. [5] Applicant is to place a check mark here if English language translation is attached.



ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /C.P.J/

| INFORMATION DISCLOSURE STATEMENT BY APPLICANT ( Not for submission under 37 CFR 1.99) | | |
|---|---|---|
| | Application Number | |
| | Filing Date | |
| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

**CERTIFICATION STATEMENT**

Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

**OR**

☐ That no item of information contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in 37 CFR 1.56(c) more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(2).

See attached certification statement.

The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.

☒ A certification statement is not submitted herewith.

**SIGNATURE**

A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

| Signature | /Timothy J. Murphy/ | Date (YYYY-MM-DD) | 2020-05-14 |
|---|---|---|---|
| Name/Print | Timothy J. Murphy | Registration Number | 64613 |

This collection of information is required by 37 CFR 1.97 and 1.98. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 1 hour to complete, including gathering, preparing and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**



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## Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these record s.

2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.

3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.

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5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.

6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).

7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.

8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.

9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.



ALL REFERENCES CONSIDERED EXCEPT WHERE LINED THROUGH. /C.P.J/

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

| | |
|---|---|
| First Inventor: | Theodore W. Jagger |
| Serial No.: | 15/931,716 |
| Filed: | May 14, 2020 |
| Group Art Unit: | 1776 |
| Examiner: | Christopher P. Jones |
| Confirmation: | 4948 |
| Title: | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR |

## RESPONSE

Without conceding to the rejections in the Non-Final Office Action dated May 24, 2022, terminal disclaimers have been submitted to render moot the double patenting rejections.

This application should now be allowed.

The Commissioner is authorized to charge Deposit Account No. 50-1482 in the name of Carlson, Gaskey & Olds, P.C. for any required fees or credit the account for any overpayment.

Respectfully Submitted,

**CARLSON, GASKEY & OLDS, P.C.**

/Timothy J. Murphy/

Timothy J. Murphy
Registration No. 64,613
400 West Maple, Suite 350
Birmingham, MI 48009
Telephone: (248) 283-0732



GCE - Exhibit 1071, Page 77
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

# Electronic Patent Application Fee Transmittal

| | |
|---|---|
| **Application Number:** | 15931716 |
| **Filing Date:** | 14-May-2020 |
| **Title of Invention:** | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR |
| **First Named Inventor/Applicant Name:** | Theodore W. Jagger |
| **Filer:** | Timothy J. Murphy/Molly Anderson |
| **Attorney Docket Number:** | 67853-012 PUS4 |

Filed as Small Entity

**Filing Fees for Utility under 35 USC 111(a)**

| Description | Fee Code | Quantity | Amount | Sub-Total in USD($) |
|---|---|---|---|---|
| **Basic Filing:** | | | | |
| **Pages:** | | | | |
| **Claims:** | | | | |
| **Miscellaneous-Filing:** | | | | |
| **Petition:** | | | | |
| **Patent-Appeals-and-Interference:** | | | | |
| **Post-Allowance-and-Post-Issuance:** | | | | |
| **Extension-of-Time:** | | | | |

| Description | Fee Code | Quantity | Amount | Sub-Total in USD($) |
|---|---|---|---|---|
| **Miscellaneous:** | | | | |
| STATUTORY OR TERMINAL DISCLAIMER | 2814 | 2 | 170 | 340 |
| | | **Total in USD ($)** | | **340** |

| Electronic Acknowledgement Receipt | |
|---|---|
| **EFS ID:** | 45788921 |
| **Application Number:** | 15931716 |
| **International Application Number:** | |
| **Confirmation Number:** | 4948 |
| **Title of Invention:** | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR |
| **First Named Inventor/Applicant Name:** | Theodore W. Jagger |
| **Customer Number:** | 26096 |
| **Filer:** | Timothy J. Murphy/Molly Anderson |
| **Filer Authorized By:** | Timothy J. Murphy |
| **Attorney Docket Number:** | 67853-012 PUS4 |
| **Receipt Date:** | 24-MAY-2022 |
| **Filing Date:** | 14-MAY-2020 |
| **Time Stamp:** | 13:08:18 |
| **Application Type:** | Utility under 35 USC 111(a) |

## Payment information:

| | |
|---|---|
| Submitted with Payment | yes |
| Payment Type | DA |
| Payment was successfully received in RAM | $340 |
| RAM confirmation Number | E20225ND08412059 |
| Deposit Account | 501482 |
| Authorized User | Molly Anderson |
| The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:<br>37 CFR 1.17 (Patent application and reexamination processing fees)<br>37 CFR 1.19 (Document supply fees) | |

37 CFR 1.20 (Post Issuance fees)

37 CFR 1.21 (Miscellaneous fees and charges)

## File Listing:

| Document Number | Document Description | File Name | File Size(Bytes)/ Message Digest | Multi Part /.zip | Pages (if appl.) |
|---|---|---|---|---|---|
| 1 | Terminal Disclaimer Filed | Terminal_Disclaimer_1.pdf | 169954<br>8941fd20a3ec2013e73367eab320be24da1f0010 | no | 2 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 2 | Terminal Disclaimer Filed | Terminal_Disclaimer_2.pdf | 165340<br>c8807fda7dd00c2f7272f6e7b2aa5e4b6f191ff3 | no | 2 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 3 | Amendment/Request for Reconsideration-After Non-Final Rejection | Response_012PUS4.pdf | 16335<br>d33dac55e9cd635d114a1105a0fef63854a8c37e | no | 1 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 4 | Fee Worksheet (SB06) | fee-info.pdf | 38278<br>afa9604a7ebf8b852cbf873cc7bc45bc21692df0 | no | 2 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| | | **Total Files Size (in bytes):** | 389907 | | |

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**New Applications Under 35 U.S.C. 111**

**If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.**

**National Stage of an International Application under 35 U.S.C. 371**

**If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.**

**New International Application Filed with the USPTO as a Receiving Office**

**If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.**

PTO/AIA/26 (04-14)
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U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE
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| **TERMINAL DISCLAIMER TO OBVIATE A DOUBLE PATENTING REJECTION OVER A "PRIOR" PATENT** | Docket Number (Optional) 67853-012 PUS4 |
|---|---|

In re Application of: Theodore W. Jagger

Application No.: 15/931,716

Filed: May 14, 2020

For:

The applicant, Vbox, Incorporated, owner of 100 percent interest in the instant application hereby disclaims, except as provided below, the terminal part of the statutory term of any patent granted on the instant application which would extend beyond the expiration date of the full statutory term of **prior patent** No. 10357628 as the term of said **prior patent** is presently shortened by any terminal disclaimer. The applicant hereby agrees that any patent so granted on the instant application shall be enforceable only for and during such period that it and the **prior patent** are commonly owned. This agreement runs with any patent granted on the instant application and is binding upon the grantee, its successors or assigns.

In making the above disclaimer, the applicant does not disclaim the terminal part of the term of any patent granted on the instant application that would extend to the expiration date of the full statutory term of the **prior patent**, "as the term of said **prior patent** is presently shortened by any terminal disclaimer," in the event that said **prior patent** later:

expires for failure to pay a maintenance fee;
is held unenforceable;
is found invalid by a court of competent jurisdiction;
is statutorily disclaimed in whole or terminally disclaimed under 37 CFR 1.321;
has all claims canceled by a reexamination certificate;
is reissued; or
is in any manner terminated prior to the expiration of its full statutory term as presently shortened by any terminal disclaimer.

Check either box 1 or 2 below, if appropriate.

1. ☐ The undersigned is the applicant. If the applicant is an assignee, the undersigned is authorized to act on behalf of the assignee.

I hereby acknowledge that any willful false statements made are punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than five (5) years, or both.

2. ☑ The undersigned is an attorney or agent of record. Reg. No. 64613

/Timothy J. Murphy/ — Signature
2022-05-24 — Date

Timothy J. Murphy — Typed or printed name

Title
248-988-8360 — Telephone Number

☑ Terminal disclaimer fee under 37 CFR 1.20(d) included.

**WARNING: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.**

This collection of information is required by 37 CFR 1.321. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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|---|---|

In re Application of: Theodore W. Jagger

Application No.: 15/931,716

Filed: May 14, 2020

For:

The applicant, Vbox, Incorporated, owner of 100 percent interest in the instant application hereby disclaims, except as provided below, the terminal part of the statutory term of any patent granted on the instant application which would extend beyond the expiration date of the full statutory term of **prior patent** No. 10702669 as the term of said **prior patent** is presently shortened by any terminal disclaimer. The applicant hereby agrees that any patent so granted on the instant application shall be enforceable only for and during such period that it and the **prior patent** are commonly owned. This agreement runs with any patent granted on the instant application and is binding upon the grantee, its successors or assigns.

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is held unenforceable;
is found invalid by a court of competent jurisdiction;
is statutorily disclaimed in whole or terminally disclaimed under 37 CFR 1.321;
has all claims canceled by a reexamination certificate;
is reissued; or
is in any manner terminated prior to the expiration of its full statutory term as presently shortened by any terminal disclaimer.

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/Timothy J. Murphy/ — Signature
2022-05-24 — Date

Timothy J. Murphy — Typed or printed name

Title
248-988-8360 — Telephone Number

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UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
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P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

| APPLICATION NUMBER | FILING OR 371(C) DATE | FIRST NAMED APPLICANT | ATTY. DOCKET NO./TITLE |
|---|---|---|---|
| 15/931,716 | 05/14/2020 | Theodore W. Jagger | 67853-012 PUS4 |

**CONFIRMATION NO. 4948**

26096
CARLSON, GASKEY & OLDS, P.C.
400 WEST MAPLE ROAD
SUITE 350
BIRMINGHAM, MI 48009

**PUBLICATION NOTICE**

*OC000000119524909*

**Title:**REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR

**Publication No.**US-2020-0269004-A1
**Publication Date:**08/27/2020

## NOTICE OF PUBLICATION OF APPLICATION

The above-identified application will be electronically published as a patent application publication pursuant to 37 CFR 1.211, et seq. The patent application publication number and publication date are set forth above.

The publication may be accessed through the USPTO's publically available Searchable Databases via the Internet at www.uspto.gov. The direct link to access the publication is currently http://www.uspto.gov/patft/.

The publication process established by the Office does not provide for mailing a copy of the publication to applicant. A copy of the publication may be obtained from the Office upon payment of the appropriate fee set forth in 37 CFR 1.19(a)(1). Orders for copies of patent application publications are handled by the USPTO's Public Records Division. The Public Records Division can be reached by telephone at (571) 272-3150 or (800) 972-6382, by facsimile at (571) 273-3250, by mail addressed to the United States Patent and Trademark Office, Public Records Division, Alexandria, VA 22313-1450 or via the Internet.

In addition, information on the status of the application, including the mailing date of Office actions and the dates of receipt of correspondence filed in the Office, may also be accessed via the Internet through the Patent Electronic Business Center at www.uspto.gov using the public side of the Patent Application Information and Retrieval (PAIR) system. The direct link to access this status information is currently https://portal.uspto.gov/pair/PublicPair. Prior to publication, such status information is confidential and may only be obtained by applicant using the private side of PAIR.

Further assistance in electronically accessing the publication, or about PAIR, is available by calling the Patent Electronic Business Center at 1-866-217-9197.

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Office of Data Managment, Application Assistance Unit (571) 272-4000, or (571) 272-4200, or 1-888-786-0101

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

| | |
|---|---|
| First Inventor: | Theodore W. Jagger |
| Serial No.: | 15/931,716 |
| Filed: | May 14, 2020 |
| Group Art Unit: | Unknown |
| Examiner: | Unknown |
| Confirmation: | 4948 |
| Title: | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR |

**PRELIMINARY AMENDMENT**

Applicant hereby submits a preliminary amendment.

A complete listing of the claims begins on page 2.

Remarks begin on page 8.

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## IN THE CLAIMS

This listing of claims will replace all prior listings.

1-20. (Cancelled)

21. (New) A portable oxygen concentrator, comprising:

a separation cartridge selectively removable from a receptacle by movement relative to the receptacle in a first direction, the separation cartridge comprising an inlet oriented in a second direction opposite the first direction, an outlet oriented in the second direction, and a bed of adsorbent material configured to preferentially adsorb nitrogen;

wherein a first line extending parallel to the first and second directions passes through the outlet,

wherein a second line spaced-apart from the first line and extending parallel to the first line passes through the inlet, and

wherein the separation cartridge is configured such that, when fluid flows through the separation cartridge, fluid flowing along the first line contacts the adsorbent material of the bed and fluid flowing along the second line does not contact the adsorbent material of the bed.

22. (New) The portable oxygen concentrator as recited in claim 21, wherein:

the outlet is arranged adjacent a first end of the bed, and

adjacent a second end of the bed opposite the first end, the separation cartridge includes a spring configured to hold the bed in place.



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23. (New) The portable oxygen concentrator as recited in claim 22, wherein:

adjacent the second end of the bed, the separation cartridge includes a porous filter, and

the spring contacts the second end of the bed via the porous filter.

24. (New) The portable oxygen concentrator as recited in claim 21, further comprising:

a power pack removable from the portable oxygen concentrator;

a pump electrically coupled to the power pack, the pump operable to cause a flow of fluid to move relative to the bed of adsorbent material;

a user interface configured to allow a patient to adjust and monitor operation of the portable oxygen concentrator; and

a carrying device for transporting the portable oxygen concentrator.

25. (New) The portable oxygen concentrator as recited in claim 21, wherein the bed comprises a bed of zeolite which provides preferential adsorption of nitrogen.

26. (New) The portable oxygen concentrator as recited in claim 25, wherein the zeolite is an LiLSX adsorbent.

27. (New) The portable oxygen concentrator as recited in claim 21, wherein the first and second lines are straight lines.

28. (New) The portable oxygen concentrator as recited in claim 21, wherein the separation cartridge is configured such that, when fluid flows through the separation cartridge, fluid flows

along the first line in the second direction and fluid flows along the second line in the first direction.

29. (New) The portable oxygen concentrator as recited in claim 21, wherein the separation cartridge includes at least one additional outlet oriented in the second direction and spaced-apart from the second line.

30. (New) The portable oxygen concentrator as recited in claim 21, wherein the first line intersects adsorbent material of the bed and the second line does not intersect adsorbent material of the bed.

31. (New) The portable oxygen concentrator as recited in claim 21, wherein the inlet is configured to receive ambient air and the outlet is configured to expel product gas.

32. (New) A separation cartridge for a portable oxygen concentrator, comprising:

an inlet oriented in a direction;

an outlet oriented in the direction;

a bed of adsorbent material configured to preferentially adsorb nitrogen; and

wherein a first line extending parallel to the direction passes through the outlet,

wherein a second line spaced-apart from the first line and extending parallel to the first line passes through the inlet, and

wherein the separation cartridge is configured such that, when fluid flows through the separation cartridge, fluid flowing along the first line contacts the adsorbent material of the bed and fluid flowing along the second line does not contact the adsorbent material of the bed.

33. (New) A portable oxygen concentrator, comprising:

a first bed of adsorbent material configured to preferentially adsorb nitrogen;

a first inlet oriented in a direction, wherein the first inlet is configured to direct a flow of ambient air to the first bed;

a first outlet oriented in the direction, wherein the first outlet is configured to expel product gas flowing downstream of the first bed;

a second bed of adsorbent material configured to preferentially adsorb nitrogen;

a second inlet oriented in the direction, wherein the second inlet is configured to direct a flow of ambient air to the second bed;

a second outlet oriented in the direction, wherein the second outlet is configured to expel product gas flowing downstream of the second bed;

a power pack removable from the portable oxygen concentrator;

a pump electrically coupled to the power pack, the pump operable to cause a flow of fluid to move relative to the first and second beds of adsorbent material;

a user interface configured to allow a patient to adjust and monitor operation of the portable oxygen concentrator;

a carrying device for transporting the portable oxygen concentrator;

wherein a first line extending parallel to the direction passes through the first outlet,



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wherein a second line spaced-apart from the first line and extending parallel to the first line passes through the first inlet,

wherein the portable oxygen concentrator is configured such that, during use, fluid flowing along the first line contacts the adsorbent material of the first bed and fluid flowing along the second line does not contact the adsorbent material of the first bed,

wherein a third line extending parallel to the direction passes through the second outlet,

wherein a fourth line spaced-apart from the third line and extending parallel to the third line passes through the second inlet, and

wherein the portable oxygen concentrator is configured such that, during use, fluid flowing along the third line contacts the adsorbent material of the second bed and fluid flowing along the fourth line does not contact the adsorbent material of the second bed.

34. (New) The portable oxygen concentrator as recited in claim 33, wherein the portable oxygen concentrator is configured such that, during use:

fluid flows along the first line in the direction,

fluid flows along the second line opposite the direction,

fluid flows along the third line in the direction, and

fluid flows along the fourth line opposite the direction.

35. (New) The portable oxygen concentrator as recited in claim 34, wherein:

the first outlet is arranged adjacent a first end of the first bed,

a first spring is arranged adjacent a second end of the first bed opposite the first end,

the first spring is configured to hold the first bed in place,



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the second outlet is arranged adjacent a first end of the second bed,

a second spring is arranged adjacent a second end of the second bed opposite the first end, and

the second spring is configured to hold the second bed in place.

36. (New) The portable oxygen concentrator as recited in claim 33, wherein the first and second beds are contained within a common separation cartridge.



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## REMARKS

This preliminary amendment cancelled claims 1-20 and has added claims 21-36. No new matter has been added. Applicant requests examination and allowance of the pending claims.

Applicant believe that no fees are due at this time. However, the Commissioner is authorized to charge to Deposit Account No. 50-1482, in the name of Carlson, Gaskey & Olds, for any required fees or credit any overpayment.

Respectfully Submitted,

**CARLSON, GASKEY & OLDS, P.C.**

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IPR2023-00527

# Electronic Acknowledgement Receipt

| | |
|---|---|
| **EFS ID:** | 40083692 |
| **Application Number:** | 15931716 |
| **International Application Number:** | |
| **Confirmation Number:** | 4948 |
| **Title of Invention:** | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR |
| **First Named Inventor/Applicant Name:** | Theodore W. Jagger |
| **Customer Number:** | 26096 |
| **Filer:** | Timothy J. Murphy/Carolyn Hannenberg |
| **Filer Authorized By:** | Timothy J. Murphy |
| **Attorney Docket Number:** | 67853-012 PUS4 |
| **Receipt Date:** | 23-JUL-2020 |
| **Filing Date:** | 14-MAY-2020 |
| **Time Stamp:** | 11:21:32 |
| **Application Type:** | Utility under 35 USC 111(a) |

## Payment information:

| | |
|---|---|
| Submitted with Payment | no |

## File Listing:

| Document Number | Document Description | File Name | File Size(Bytes)/ Message Digest | Multi Part /.zip | Pages (if appl.) |
|---|---|---|---|---|---|
| 1 | | Preliminary_Amendment_012PUS4.pdf | 33217<br>d6a78e63a96324c9a4b3c04ff62670eab1e4ac3d | yes | 8 |

| | Multipart Description/PDF files in .zip description | | |
|---|---|---|---|
| | **Document Description** | **Start** | **End** |
| | Preliminary Amendment | 1 | 1 |
| | Claims | 2 | 7 |
| | Applicant Arguments/Remarks Made in an Amendment | 8 | 8 |
| **Warnings:** | | | |
| **Information:** | | | |
| | **Total Files Size (in bytes):** | 33217 | |

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PTO/SB/06 (09-11)
Approved for use through 1/31/2014. OMB 0651-0032
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| **PATENT APPLICATION FEE DETERMINATION RECORD**<br>Substitute for Form PTO-875 | Application or Docket Number<br>15/931,716 | Filing Date<br>05/14/2020 | ☐ To be Mailed |
|---|---|---|---|

**ENTITY:** ☐ LARGE ☑ SMALL ☐ MICRO

**APPLICATION AS FILED - PART I**

| FOR | (Column 1)<br>NUMBER FILED | (Column 2)<br>NUMBER EXTRA | | RATE ($) | FEE ($) |
|---|---|---|---|---|---|
| ☐ BASIC FEE<br>(37 CFR 1.16(a), (b), or (c)) | N/A | N/A | | N/A | |
| ☐ SEARCH FEE<br>(37 CFR 1.16(k), (i), or (m)) | N/A | N/A | | N/A | |
| ☐ EXAMINATION FEE<br>(37 CFR 1.16(o), (p), or (q)) | N/A | N/A | | N/A | |
| TOTAL CLAIMS<br>(37 CFR 1.16(i)) | minus 20 = | * | | x $50 = | |
| INDEPENDENT CLAIMS<br>(37 CFR 1.16(h)) | minus 3 = | * | | x $230 = | |
| ☐ APPLICATION SIZE FEE (37 CFR 1.16(s)) | If the specification and drawings exceed 100 sheets of paper, the application size fee due is $310 ($155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s). | | | | |
| ☐ MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j)) | | | | | |
| * If the difference in column 1 is less than zero, enter "0" in column 2. | | | | TOTAL | |

**APPLICATION AS AMENDED - PART II**

| | | (Column 1) | | (Column 2) | (Column 3) | | | |
|---|---|---|---|---|---|---|---|---|
| AMENDMENT | 07/23/2020 | CLAIMS REMAINING AFTER AMENDMENT | | HIGHEST NUMBER PREVIOUSLY PAID FOR | PRESENT EXTRA | | RATE ($) | ADDITIONAL FEE ($) |
| | Total<br>(37 CFR 1.16(i)) | * 16 | Minus | ** 20 | = 0 | | x $50 = | 0 |
| | Independent<br>(37 CFR 1.16(h)) | * 3 | Minus | *** 3 | = 0 | | x $230 = | 0 |
| | ☐ Application Size Fee (37 CFR 1.16(s)) | | | | | | | |
| | ☐ FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j)) | | | | | | | |
| | | | | | | | TOTAL ADD'L FEE | 0 |

| | | (Column 1) | | (Column 2) | (Column 3) | | | |
|---|---|---|---|---|---|---|---|---|
| AMENDMENT | | CLAIMS REMAINING AFTER AMENDMENT | | HIGHEST NUMBER PREVIOUSLY PAID FOR | PRESENT EXTRA | | RATE ($) | ADDITIONAL FEE ($) |
| | Total<br>(37 CFR 1.16(i)) | * | Minus | ** | = | | x $0 = | |
| | Independent<br>(37 CFR 1.16(h)) | * | Minus | *** | = | | x $0 = | |
| | ☐ Application Size Fee (37 CFR 1.16(s)) | | | | | | | |
| | ☐ FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j)) | | | | | | | |
| | | | | | | | TOTAL ADD'L FEE | |

| | |
|---|---|
| * If the entry in column 1 is less than the entry in column 2, write "0" in column 3. | SLIE |
| ** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20". | /SHARON M WEST/ |
| *** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3". | |
| The "Highest Number Previously Paid For" (Total or Independent) is the highest number found in the appropriate box in column 1. | |

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| APPLICATION NUMBER | FILING or 371(c) DATE | GRP ART UNIT | FIL FEE REC'D | ATTY.DOCKET.NO | TOT CLAIMS | IND CLAIMS |
|---|---|---|---|---|---|---|
| 15/931,716 | 05/14/2020 | 3785 | 785 | 67853-012 PUS4 | 20 | 2 |

**CONFIRMATION NO. 4948**

**FILING RECEIPT**

26096
CARLSON, GASKEY & OLDS, P.C.
400 WEST MAPLE ROAD
SUITE 350
BIRMINGHAM, MI 48009
UNITED STATES OF AMERICA

OC000000117193050

Date Mailed: 05/22/2020

Receipt is acknowledged of this non-provisional utility patent application. The application will be taken up for examination in due course. Applicant will be notified as to the results of the examination. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF FIRST INVENTOR, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection.

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**Inventor(s)**

Theodore W. Jagger, White Bear Lake, MN;
Nicholas P. Van Brunt, White Bear Lake, MN;
John A. Kivisto, Oak Grove, MN;
Perry B. Lonnes, White Bear Lake, MN;

**Applicant(s)**

Vbox, Incorporated, White Bear Lake, MN;

**Power of Attorney:** The patent practitioners associated with Customer Number 26096

**Domestic Priority data as claimed by applicant**

This application is a CON of 14/193,504 02/28/2014
which is a CON of 13/795,977 03/12/2013 ABN
which is a CON of 13/096,362 04/28/2011 ABN
which is a CON of 11/054,341 02/09/2005 PAT 7954490

**Foreign Applications** for which priority is claimed (You may be eligible to benefit from the **Patent Prosecution Highway** program at the USPTO. Please see http://www.uspto.gov for more information.) - None.
*Foreign application information must be provided in an Application Data Sheet in order to constitute a claim to foreign priority. See 37 CFR 1.55 and 1.76.*

**Permission to Access Application via Priority Document Exchange:** Yes

**Permission to Access Search Results:** Yes

Applicant may provide or rescind an authorization for access using Form PTO/SB/39 or Form PTO/SB/69 as appropriate.

**If Required, Foreign Filing License Granted:** 05/21/2020
The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 15/931,716**
**Projected Publication Date:** 08/27/2020
**Non-Publication Request:** No
**Early Publication Request:** No
**** SMALL ENTITY ****
**Title**

REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR

**Preliminary Class**

128

**Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications:** No

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this website includes self-help "toolkits" giving innovators guidance on how to protect intellectual property in specific countries such as China, Korea and Mexico. For questions regarding patent enforcement issues, applicants may call the U.S. Government hotline at 1-866-999-HALT (1-866-999-4258).

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## PATENT APPLICATION FEE DETERMINATION RECORD

Substitute for Form PTO-875

Application or Docket Number: 15/931,716

### APPLICATION AS FILED - PART I

| FOR | (Column 1) NUMBER FILED | (Column 2) NUMBER EXTRA | SMALL ENTITY RATE($) | SMALL ENTITY FEE($) | OR | OTHER THAN SMALL ENTITY RATE($) | OTHER THAN SMALL ENTITY FEE($) |
|---|---|---|---|---|---|---|---|
| BASIC FEE (37 CFR 1.16(a), (b), or (c)) | N/A | N/A | N/A | 75 | | N/A | |
| SEARCH FEE (37 CFR 1.16(k), (i), or (m)) | N/A | N/A | N/A | 330 | | N/A | |
| EXAMINATION FEE (37 CFR 1.16(o), (p), or (q)) | N/A | N/A | N/A | 380 | | N/A | |
| TOTAL CLAIMS (37 CFR 1.16(i)) | 20 minus 20 = | * | x 50 = | 0.00 | OR | | |
| INDEPENDENT CLAIMS (37 CFR 1.16(h)) | 2 minus 3 = | * | x 230 = | 0.00 | | | |
| APPLICATION SIZE FEE (37 CFR 1.16(s)) | If the specification and drawings exceed 100 sheets of paper, the application size fee due is $310 ($155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s). | | | 0.00 | | | |
| MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j)) | | | | 0.00 | | | |
| * If the difference in column 1 is less than zero, enter "0" in column 2. | | | TOTAL | 785 | | TOTAL | |

### APPLICATION AS AMENDED - PART II

| | | (Column 1) CLAIMS REMAINING AFTER AMENDMENT | | (Column 2) HIGHEST NUMBER PREVIOUSLY PAID FOR | (Column 3) PRESENT EXTRA | SMALL ENTITY RATE($) | SMALL ENTITY ADDITIONAL FEE($) | OR | OTHER THAN SMALL ENTITY RATE($) | OTHER THAN SMALL ENTITY ADDITIONAL FEE($) |
|---|---|---|---|---|---|---|---|---|---|---|
| AMENDMENT A | Total (37 CFR 1.16(i)) | * | Minus | ** | = | x = | | OR | x = | |
| | Independent (37 CFR 1.16(h)) | * | Minus | *** | = | x = | | OR | x = | |
| | Application Size Fee (37 CFR 1.16(s)) | | | | | | | | | |
| | FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j)) | | | | | | | OR | | |
| | | | | | | TOTAL ADD'L FEE | | OR | TOTAL ADD'L FEE | |

| | | (Column 1) CLAIMS REMAINING AFTER AMENDMENT | | (Column 2) HIGHEST NUMBER PREVIOUSLY PAID FOR | (Column 3) PRESENT EXTRA | RATE($) | ADDITIONAL FEE($) | | RATE($) | ADDITIONAL FEE($) |
|---|---|---|---|---|---|---|---|---|---|---|
| AMENDMENT B | Total (37 CFR 1.16(i)) | * | Minus | ** | = | x = | | OR | x = | |
| | Independent (37 CFR 1.16(h)) | * | Minus | *** | = | x = | | OR | x = | |
| | Application Size Fee (37 CFR 1.16(s)) | | | | | | | | | |
| | FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j)) | | | | | | | OR | | |
| | | | | | | TOTAL ADD'L FEE | | OR | TOTAL ADD'L FEE | |

* If the entry in column 1 is less than the entry in column 2, write "0" in column 3.
** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20".
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| **INFORMATION DISCLOSURE STATEMENT BY APPLICANT** ( **Not for submission under 37 CFR 1.99)** | | |
|---|---|---|
| | Application Number | |
| | Filing Date | |
| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

| **U.S.PATENTS** | | | | | | Remove |
|---|---|---|---|---|---|---|
| Examiner Initial* | Cite No | Patent Number | Kind Code[1] | Issue Date | Name of Patentee or Applicant of cited Document | Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear |
| | 1 | 3121625 | | 1964-02-18 | D. B. Broughton | |
| | 2 | 3236028 | | 1966-02-22 | G. A. Rutan | |
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| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

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| | 1 | Recommendations of the Fifth Oxygen Consensus Conference, 1999 | |
| | 2 | "AirSep lowers boom", www.hmenews.com/2004.12/depts/vendors/vendors1.htm. | |
| | 3 | PETER L BLISS, ROBERT W MCCOY, ALEXANDER B ADAMS, A Bench Study Comparison of Demand Oxygen Delivery Systems and Continuous Flow Oxygen, Respiratory Care, August 1999, Vol 44. No 8 | |

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| Examiner Signature | | Date Considered | |
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| INFORMATION DISCLOSURE STATEMENT BY APPLICANT ( Not for submission under 37 CFR 1.99) | | |
|---|---|---|
| | Application Number | |
| | Filing Date | |
| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

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Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

**OR**

☐ That no item of information contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in 37 CFR 1.56(c) more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(2).

See attached certification statement.

The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.

☒ A certification statement is not submitted herewith.

**SIGNATURE**

A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

| Signature | /Timothy J. Murphy/ | Date (YYYY-MM-DD) | 2020-05-14 |
|---|---|---|---|
| Name/Print | Timothy J. Murphy | Registration Number | 64,613 |

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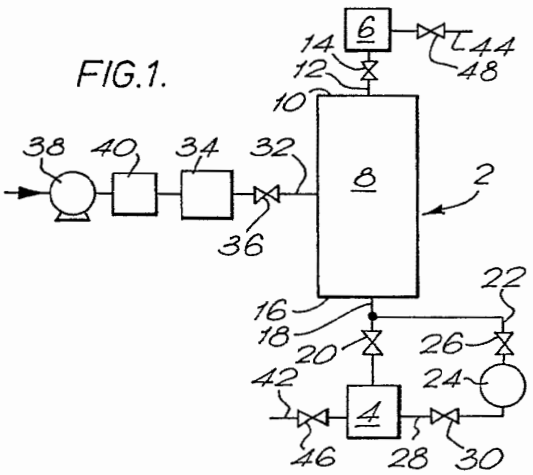
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# (54) Separation of a gas mixture

(57) A gas separation is effected by passing gas from a reservoir 4 into a bed 8 of adsorbent material so as to raise the pressure therein. More readily adsorbable gas is thus adsorbed. Gas flow from the reservoir 4 to the bed 8 continues at substantially constant pressure with gas being displaced from the bed 8 into the reservoir 6. An expansion or pressure reduction in the bed 8 is then performed by withdrawing gas from the bed 8 by means of pump 24 and passing the gas into the reservoir 4. As a result of the pressure reduction more readily adsorbable gas is desorbed. The withdrawal of gas is continued but a substantially constant pressure is maintained in the bed 8 by allowing gas to flow into the bed 8 from the reservoir 6. Product gases may from time-to-time be withdrawn from the reservoirs 4 and 6 and an equal volume of feed gas mixture introduced into the column 2 through the inlet 32. As a result of this cycle there is a net upward flow of the less readily adsorbable component of the gas mixture and a net downward flow of the more readily adsorbable component of the gas mixture. A multiplicity of operating cycles are typically needed to separate each increment of feed gas mixture. The method and apparatus may be employed to separate air into oxygen-rich and nitrogen-rich fractions at relatively high efficiency.

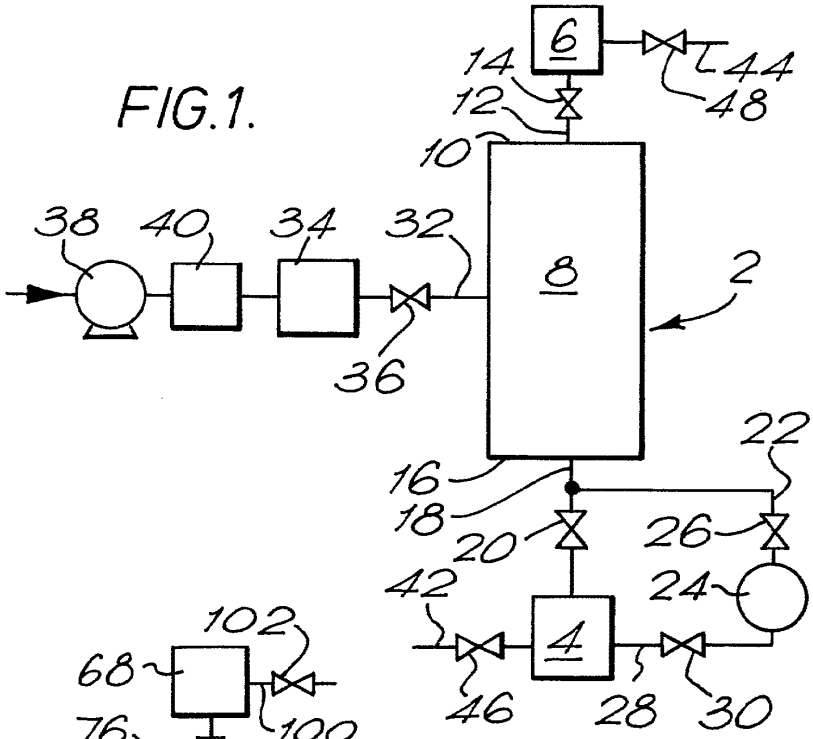
FIG.1.




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FIG.1.



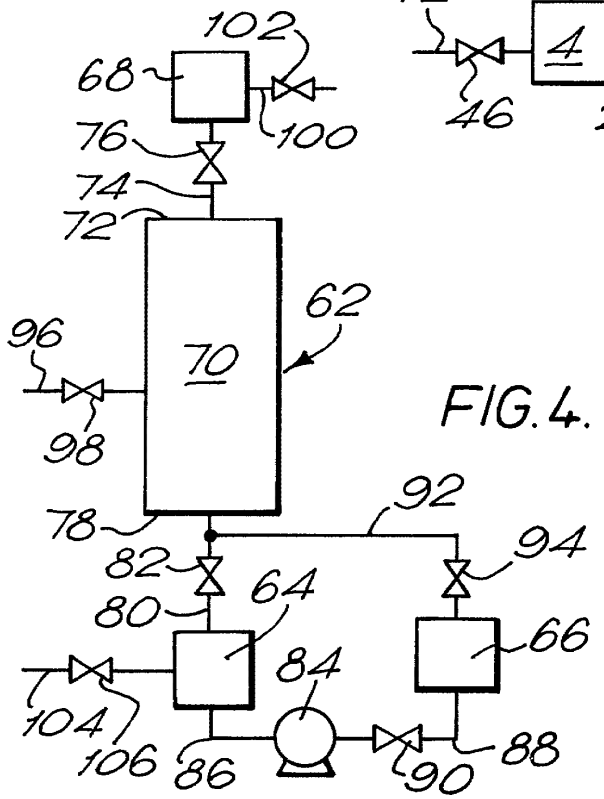




FIG.4.

GCE - Exhibit 1071, Page 128
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

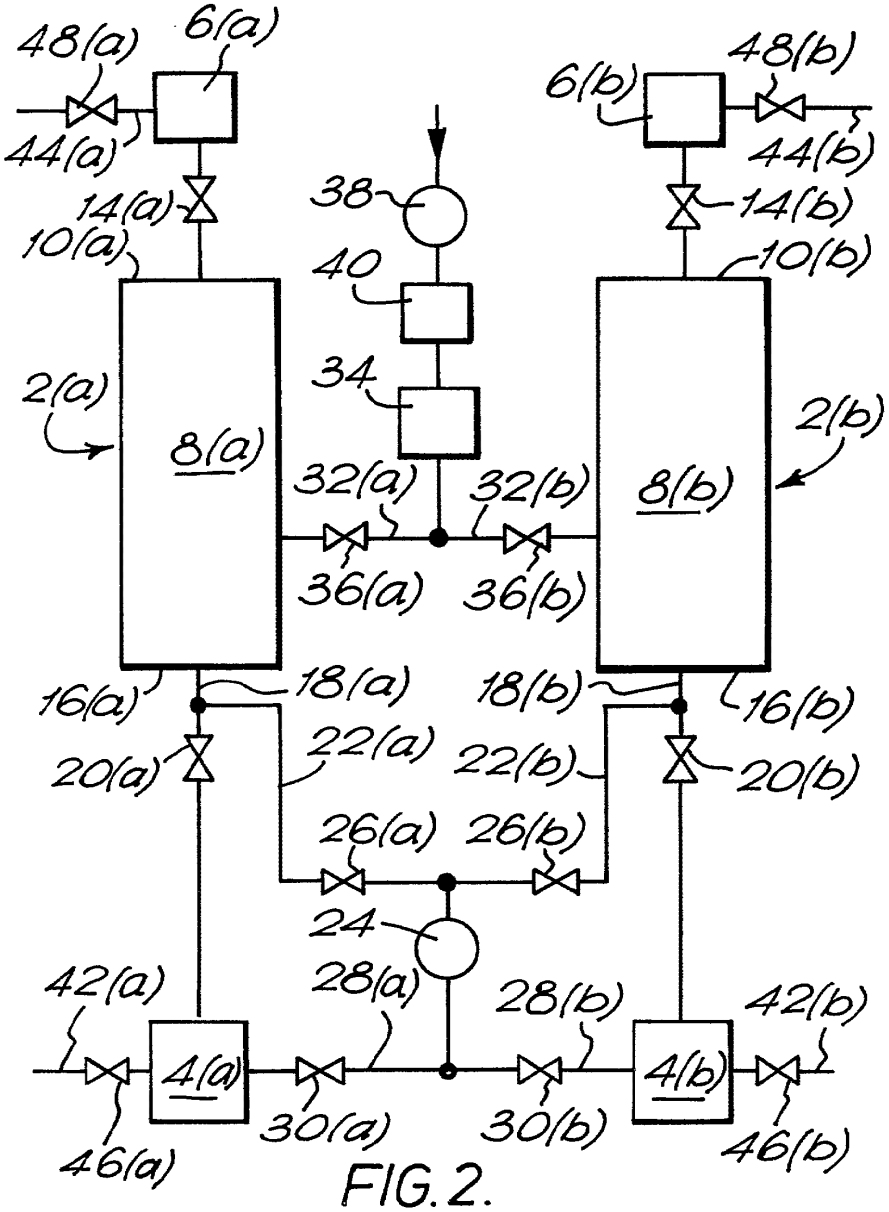

48(a)
6(a)
44(a)
14(a)
10(a)
2(a)
8(a)
38
40
34
32(a)
32(b)
36(a)
36(b)
48(b)
6(b)
44(b)
14(b)
10(b)
2(b)
8(b)
16(a)
18(a)
18(b)
16(b)
20(a)
22(a)
22(b)
20(b)
26(a)
26(b)
24
42(a)
28(a)
28(b)
42(b)
4(a)
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30(a)
30(b)
46(a)
46(b)

FIG. 2.

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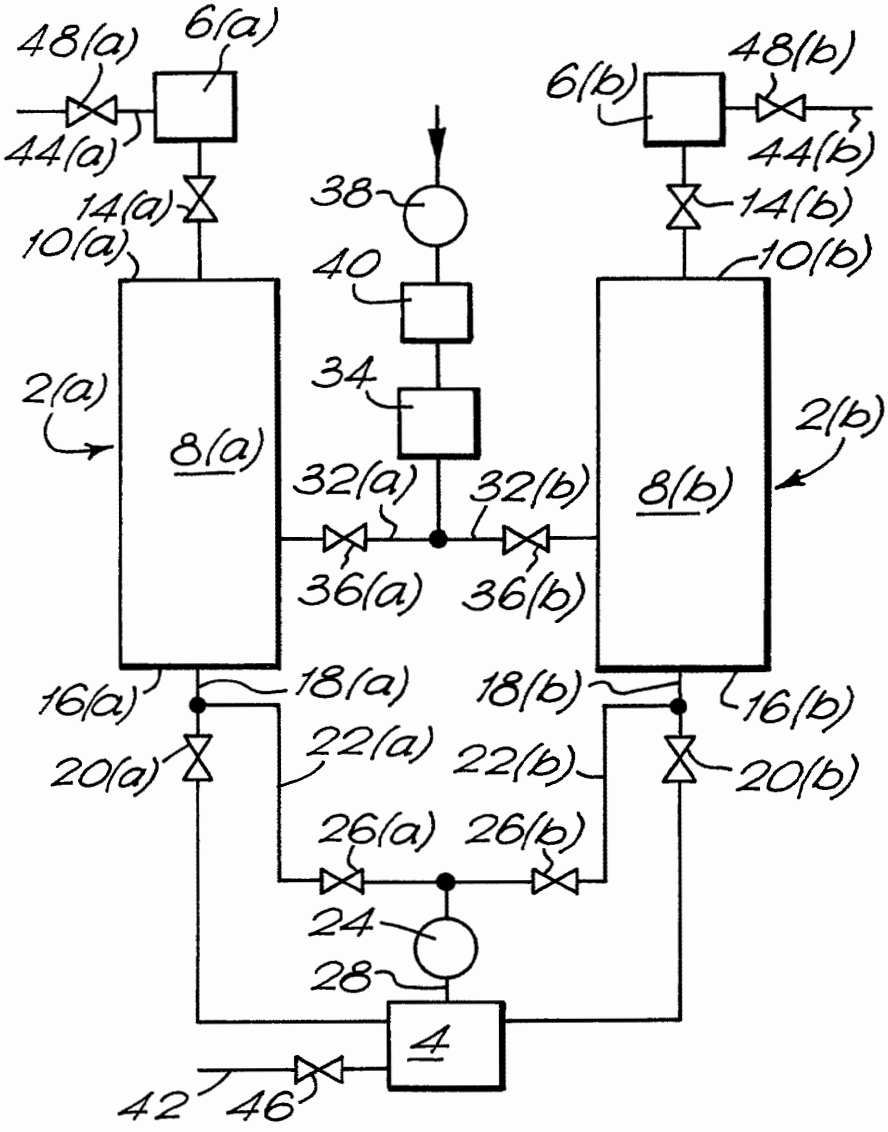



FIG. 3.

SPECIFICATION

**Separation of a gas mixture**

This invention relates to the separation of a gas mixture. It is particularly concerned with the separation of gas mixtures employing adsorbent material. By appropriate selection of the adsorbent it is known to separate a feed gas mixture into two gas streams, one relatively rich in the more readily adsorbed constituent or constituents of the gas mixture, the other relatively rich in the less readily adsorbed constituent or constituents of the gas mixture. Typically, one of the gas streams is taken as product and the other stream is vented to the atmosphere. Normally, a plurality of beds is employed, each bed being regenerated in turn.

A large number or processes for the separation of gas mixtures employ adsorbent in such a way. Typically, adsorption takes place at a relatively elevated pressure and the adsorbent is regenerated by subjecting it to a relatively low pressure. Such a mode of operation is attractive in its simplicity, but does involve large losses of energy and of the yield.

If these so called "pressure swing adsorption" processes for separating gas mixtures are analysed thermodynamically it will be seen that they involve the performance of a large quantity of irreversible work. It is an aim of the present invention to provide a more thermodynamically efficient method and apparatus for separating gas using an adsorbent.

According to the present invention, there is provided a method of separating a feed gas mixture into a first gaseous fraction and a second gaseous fraction, comprising performing the following steps in repeated sequence:

(a) fractionally raising the pressure of a gas mixture which is resident in a bed of adsorbent capable of selectively adsorbing at least one constituent of the feed gas mixture and which comprises the same constituents as said feed gas mixture, and thereby adsorbing said at least one selectively adsorbable constituent;

(b) performing a first incremental translation at approximately constant pressure of the unadsorbed gas in one direction;

(c) fractionally reducing the pressure of the gas in the bed and thereby desorbing previously adsorbed gas from the adsorbent;

(d) performing a second incremental translation at approximately constant pressure of the gas in the bed in a direction opposite to the direction of translation in step (b),

wherein during the repeated performance of said steps first and second gaseous fractions of different composition from each other and from feed gas mixture are withdrawn and the feed gas mixture for separation flows into the bed at a region intermediate its ends;

The invention also provides apparatus for performing the above method comprising a column containing a bed of adsorbent capable of selectively adsorbing at least one constituent of the feed gas mixture and having associated therewith an outlet for a first gaseous fraction spaced apart from an outlet for a second gaseous fraction and an inlet for gas mixture to be separated intermediate the said two ends of the column; at least one first reservoir able to be placed in communication with one end of the column by operation of at least one first valve, at least one second reservoir able to be placed in communication with the other end of the column by operation of at least one second valve, and means for extracting gas from one end of the column and passing it to the said reservoir associated with that end.

In a preferred feature of the present invention, the pressure raising (or compression) step is performed by allowing gas to flow into the adsorbent bed from a reservoir associated with one end of the bed by virtue of a pressure different between the reservoir and the bed. No gas is discharge from the bed during this step. The first translation is then preferably performed by continuing to allow gas to flow into the adsorbent bed but allowing a substantially equal quantity to be displaced from the other end, at least some of which displaced gas flows into another reservoir associated with that end. The pressure reduction (or expansion) step is preferably performed by pumping gas from said one end of the bed into a reservoir associated with that end. The second translation is preferably performed by continuing to pump gas into the reservoir but allowing a substantially equal quantity of gas to flow into said bed from the reservoir associated with said other end.

In order to maximise the use made of a mechanical pump employed to carry out such pumping, feed gas may be separated in two separate, parallel beds operating out-of-phase with one another such that steps (a) and (b) of a cycle are performed on one bed, while steps (c) and (d) are performed on the other and vice versa. If desired, the ends of the respective columns served by the pump may share a common reservoir. Any convenient kind pump may be employed. The invention thus does not rely on the use of reciprocating machinery.

In separating a binary mixture into a first gaseous fraction rich in the more readily adsorbed constituent of the mixture and a second gaseous fraction rich in the less readily adsorbed consitituent of the mixture, the outlets for the gaseous fractions are preferably associated with the respective ends of the adsorbent bed.

The volume of gas translated during each first and each second translation is small compared with the free gas space in said bed. Moreover, the difference between the pressure that obtains in the bed at the end of each step (a) and the pressure that obtains in the bed at the end of each step (c) is preferably small compared with the former of these pressures. Thus, in a preferred methods according to the invention, there is no rapid to-and-fro glow of gas between say the top of an adsorption bed and the lower half of the bed. Rather, there is in effect a net gradual passage of the more adsorbable constituent or constituents of the gas mixture towards one end of the bed and a net gradual passage of the less adsorbable constituent or constituents towards the other end of the bed even though each complete cycle of steps (a) to

(d) may only take say a few seconds (or less) to complete. Consider the example of the separation of air (purified by the removal of water vapour and carbon dioxide) in a vertical bed of carbon molecular sieve of the kind that adsorbs oxygen more rapidly than nitrogen. During the pressure raising step of the method there is adsorption of oxygen and hence a direct (and say) upward flow of the unadsorbed gas. This upward flow is greater than that which would be predicted from a mere consideration of the "pressure swing" that takes place between the start and end of each pressure raising step. The extent of the upward flow is increased since oxygen is adsorbed. Translation in the same direction then takes place. There is thus further upward flow of the unadsorbed gas and some of this gas will enter the reservoir associated with the top of the bed. The pressure reduction step results in desorption of oxygen and a downward flow of gas comprising unadsorbed nitrogen and desorbed oxygen. The downward flow of nitrogen is not as great as that which would be predicted by mere consideration of the "pressure swing" that takes place between the start and end of each pressure reduction step. The extent of the downward flow is decreased by the desorbing of oxygen. Further downward flow of gas takes place during the second translation step. Considering now the cycle as a whole, there is a net upward flow of unadsorbed gas (nitrogen) and a net downward flow of adsorbed gas (oxygen). Introduction of air into the bed and withdrawal of relatively pure oxygen and nitrogen fractions is arranged so as not to prevent the establishment in the bed of such a flow regime. Since there is a net upward flow of nitrogen the gas and a net downward flow of oxygen, the gas mixture in the bed tends to be relative rich in oxygen at the bottom of the bed, and then to become progressively leaner in oxygen as one ascends the bed with a relatively nitrogen-rich fraction obtaining at the top of the bed. Thus, oxygen-rich gas collects in one reservoir and nitrogen-rich gas in the other reservoir.

The pressure in the bed during each of the first and second translation steps preferably deviates very little from a constant value, whereby the amount of irreversible work that is performed in the bed during these steps is kept to a minimum. Accordingly, a separation of the gas mixture relatively efficient thermodynamically in comparison with conventional pressure swing adsorption processes for the separation of gas mixtures is made possible. Such deviations as do take place during the first translation should be in the direction of increasing the pressure, and such deviations as do take place during the second translation should be in the direction of reducing the pressure.

Introduction of the feed gas mixture for separation and extraction of the first and second gaseous fractions may be carried out in any convenient part of the cycle. Preferably, feed gas mixture is introduced into the bed in a discrete step immediately following the second isobaric displacement step and immediately preceding the pressure raising step. The volume of the feed gas introduced per cycle of operations is preferably small compared with the free gas space in the bed. Preferably, the introduction of the feed gas into the bed is performed such that the pressure deviates in the bed very little from an constant value and that the pressure is returned to its value at the start of step (a) of the method. During each operating cycle, the volume of gas withdrawn as product preferably equals the volume of gas introduced into the bed for separation during each operating cycle. The first and second gaseous fraction can be extracted from the bed at any convenient time during each operating cycle. For example, a first gaseous fraction rich in the preferentially adsorbed gas may be taken for at least part of the time in which feed gas mixture is introduced into the bed for separation and a second gaseous fraction relatively rich in the less preferentially adsorbed constituent or constituents of the feed gas mixture may be withdrawn as product during at least part of the first translation step. It is generally arranged for the pressure in the gas reservoirs to be super-ambient and thus it is possible to withdraw the products at any time or continuously.

Typically, the pressure swing involved in each operating cycle is in the order of 5 psi, and the volume of gas translated in each translation step is less than 5% of the total bed capacity.

The method and apparatus according to the present invention will now be further described by way of example with reference to the accompanying drawings, in which:

*Figure 1* is a schematic drawing of one plant including a single column for separating air according to the present invention.

*Figure 2* is a schematic drawing illustrating a second plant, including two adsorption columns, for separating air in accordance with the invention;

*Figure 3* is a schematic drawing of yet another plant for performing the present invention;

*Figure 4* is a schematic drawing of another plant for performing the present invention.

Referring to Figure 1 of the drawings, a plant for the separation of air includes a column 2, a first reservoir 4, and a second reservoir 6. The column 2 and reservoirs 4 and 6 are constructed as pressure vessels. The column 2 contains a bed 8 of carbon molecular sieve capable of preferentially adsorbing oxygen from a gas mixture comprising oxygen and nitrogen. The bed 8 occupies substantially the whole internal volume of the column 2. The top end 10 of the bed 8 communicates with a conduit 12 which extends from the top of the column 2 to the second reservoir 6. A valve 14 is located in the conduit 12. The bottom end 16 of the bed 8 communicates with a conduit 18 which extends from the bottom of the column 2 to the first reservoir 4. A valve 20 is located in the conduit 18. A conduit 22 terminates at one end in the conduit 18 at a region thereof intermediate the valve 20 and the bottom of the column 2 and at its other end in the inlet to a pump 24. A valve 26 is located in the conduit 22. A further conduit 28 extends from the outlet of the pump 24 and terminates in the reservoir 4. A valve 30 is located in the conduit 28.

The column 2 is also provided with an inlet 32 for compressed and dried air to be separated. Such air is

supplied from a third reservoir 34. The reservoir 34 is constructed as a pressure vessel. The outlet of the reservoir 34 communicates with the inlet 32 and is provided with a valve 36. The inlet of the reservoir 34 communicates with the outlet of air compressor 38 via conventional means for removing water, particulates and carbon dioxide located at 40.

The reservoirs 4 and 6 are provided with outlets 42 and 44 respectively having valves 46 and 48 respectively disposed therein.

All the above mentioned valves may be pneumatically or hydraulically operated and the relative timings of their opening and closing may be effected by means of a central programmable controller (not shown) of a kind conventional in the art.

In operation, with the pressure in the bed 8 and reservoir 6 being equal to one another at a value below that in the reservoir 4, with the reservoir 4 containing oxygen-rich gas and the reservoir 6 containing nitrogen-rich gas, with the void spaces in the bed 8 containing a gas mixture comprising nitrogen and oxygen, with a flow of air just having been admitted to the bed 8 via the inlet 32, with some oxygen product just having been withdrawn from the reservoir 4 through the conduit 42, and with the valves 36 and 46 just having been closed and the valve 20 opened, gas flows from the reservoir 4 into the bed 8 by virtue of the pressure difference therebetween. Thus the gas in the bed 8 is compressed and a proportion of the oxygen in the bed is adsorbed in consequence of the increasing pressure in the bed 8. As a result of the increasing pressure in the void spaces of the bed 8 and the removal of some oxygen from these void spaces by virtue of its adsorption there is an upward flow of nitrogen molecules in the void spaces of the bed.

Appreciably before the pressure in the reservoir 4 equalises with the pressure in the bed 8, the valve 14 is opened and gas thus flows from the bed 8 into the reservoir 6. The continuing flow of gas into the bed 8 from the reservoir 4 approximately balances the flow of gas out of the bed 8 into the reservoir 6 so that the pressure in the bed 8 increases only slightly during the time that the valve 14 is open. Thus, during this time there is an upward translation or displacement of unadsorbed gas, but the oxygen that has been previously adsorbed during the previous compression step remains adsorbed. There is thus a net upward displacement of nitrogen relative to oxygen during the compression and upward translation steps.

For a period during the upward displacement step, nitrogen is withdrawn from the reservoir 6 via the outlet 44. To this end, valve 48 is opened and nitrogen is allowed to flow under pressure from the reservoir 6 to a suitable collection vessel (not shown). Typically the withdrawal of nitrogen product takes place during the latter half of the upward displacement step.

In order to maintain approximately isobaric conditions in the bed 8 during the upward displacement step the flow of gas into the bed 8 from the reservoir 4 is maintained as close to equality as possible with the flow of gas out of the bed 8 into the reservoir 6. Thus, the difference between the pressure in the reservoir 4 and the bed 8 and the difference between the pressure in the bed 8 and the reservoir 6 at the start of the upward translation step are chosen so as to ensure that approch closely to equality throughout the upward translation step.

Typically, but not necessarily, the upward translation step continues until the pressures in the reservoirs 4 and 6 and the bed 8 become equal with one another. Valves 14, 20 and 44 close and valves 26 and 30 open so as to start a pressure reduction step. Throughout this step the pump 24 is operated to withdraw gas from the bottom of bed 8 and to pass it into the reservoir 4. The pressure in the bed 8 thus falls causing some oxygen molecules to be desorbed from the adsorbent and then to flow downwards through the bed 8. There is thus a net downward flow of oxygen. The gas in the voids spaces at the start of the pressure reduction step flows downwards during this step by a distance which is less than that which would be expected from a mere consideration of the pressure obtaining at the start of this step and the pressure obtaining at the end of this step. This is because some of the downward flow of gas that takes place during the pressure reduction step is constituted by oxygen that is desorbed. The pressure reduction step is concluded when the pressure in the bed reaches the value that obtained at the start of the foregoing pressure raising the value that obtained at the start of the foregoing pressure raising step. In that step there was an enhanced upward flow of unadsorbed gas as a result of the adsorption of some oxygen molecules. Comparing the pressure reduction step with the pressure raising step it can thus be seen that there is an overall upward flow of unadsorbed gas and an overall downward flow of adsorbed gas. Thus there is a net upward flow of nitrogen and a net downward flow of oxygen.

The duration of the pressure reduction step is typically arranged to be equal to the duration of the pressure raising or compression step. Change over from pressure reduction to isobaric downward translation of gas in the void spaces of the bed 8 is effected by opening valve 14. It is to be appreciated that during the pressure reduction step a pressure difference between the reservoir 6 and the bed 8 is built up. During the downward translation step, gas flows from the reservoir 6 into the void spaces of the bed 8 to take the place of the gas that is withdrawn from the void spaces of the bed 8 by the pump 24. Thus approximately isobaric conditions with the bed pressure falling only slightly are able to be maintained in the bed 8 during the downward translation. During the downward translation step there is a downward flow of oxygen molecules previously desorbed during the pressure reduction step. When the pressure in the reservoir 6 reaches equality with that in the void spaces of the bed, the valve 14 is closed and the valve 36 opens to admit compressed dry air which is free of carbon dioxide into the bed 8 at an intermediate level. If desired, the pressure of the incoming air may be selected to be a little in excess of the pressure that obtains at the instant the valve 14 is closed and the valve 36 opens. It is thus possible to maintain a pressure in the bed 8 that

deviates only slightly from a constant value even though gas continues to be withdrawn therefrom by the pump 24. For at least a part of the period in which air is admitted to the bed oxygen product is withdrawn from the reservoir 4. To this end, the valve 46 is opened and the reservoir 4 placed in communication with for example a storage vessel for product oxygen (not shown). The volumes of nitrogen product and oxygen product withdrawn during each cycle are equal to the volume of air admitted during each cycle. The ratio of volume of nitrogen product to volume of oxygen product withdrawn during each cycle is arranged to approxmate to the ratio of nitrogen to oxygen in the incoming air, and the reservoirs 6 and 4 have a similar volume ratio.

The total duration of each downward displacement step and the following air admission step is typically arranged to be equal to the duration of each upward displacement step. The pressure of the bed 8 at the end of the air feed step equals that at the start of the preceding pressure raising or compression step.

As the downward translation step proceeds so the pressure in the reservoir 4 is built up and thus by the end of the air admission step a pressure in the reservoir has been established that is sufficient to enable the next pressure raising (or compression) and upward displacement steps to be carried out.

To bring the air admission step to an end, the valves 26, 30, 36 and 46 are closed; and the valve 20 is simultaneously opened to commence the next pressure raising or compression step. Thus, the above described cycle of steps may be continuously repeated.

Considering now the volume of air that is admitted to the bed during the air admission step of the cycle, this volume will initially flow downwards. A "band" of "new" air is thus established in the column immediately below the level of the inlet 32. During the following compression step there is a small upward displacement of this air and, moreover, at least some of the oxygen molecules are adsorbed. There is thus a net upward displacement of nitrogen molecules relative to oxygen molecules as the action of the adsorbent in adsorbing oxygen molecules from the air band starts with the commencement of the compression step. In the following upward translation step the oxygen molecules remain adsorbed and the remainder of the air band is displaced upward. Thus the net upward displacement of the nitrogen molecules relative to the oxygen molecules increases. In the pressure reduction or expansion step the previously adsorbed oxygen molecules are desorbed and thus enter the void spaces of the bed at the positions where they were initially adsorbed. The desorbed oxygen molecules limit the downward displacement of the previously unadsorbed portion of the air band to a small extent so that the downward flow of unadsorbed gas molecules during the expansion step does not quite match the upward displacement of unadsorbed gas molecules during the previous compression step. The downward displacement step then takes place, during which step the air band now relatively rich in oxygen at its lower front flows downwards. There is thus a net downward flow of oxygen molecules and a net upward flow of nitrogen molecules. In other words during one operating cycle the incoming air band has been re-ordered to that it no longer contains uniform concentrations of oxygen and nitrogen but instead has a greater concentration of oxygen molecules towards its lower "fring". Moreover, the initial "band" had become spread with the upper fringe of the band (which is relatively rich in nitrogen) being situated above the level of the inlet 32 by a distance corresponding to the extent of upward flow or displacement, and the lower "fringe" of the band having been displaced downwards by a distance corresponding to the extent of the downward flow in the cycle. It can thus be seen that in one operating cycle there has been a downward flow of oxygen molecules and an upward flow of nitrogen molecules. Considering now the next complete cycle, by analogy with the first cycle, it can be seen that the "oxygen-rich" fringe will be displaced upwards and further enriched in nitrogen. While it is possible to arrange the apparatus such that the "oxygen-rich" fringe could reach the bottom of the column and the "nitrogen-rich" fringe the top of the column in say one or two cycles no advantage accrues from such a procedure. Indeed, the thermodynamic efficiency of the process for example in terms of specific power consumption tends to increase the greater the numer of individual operating cycles it takes for the initial air band to be separated into nitrogen rich gas at the top of the column 2 and the oxygen-rich gas at the bottom of the column 2. Thus, it is preferable for the "pressure swing" between the pressure at the start and end of the compression step to be small and the volume of gas entering or leaving the bed to and from the reservoirs to be only a small increment of the total volume of the bed. Although this has the consequence that the degree of enrichment acheived by one operating cycle may be small, and the "pressure swing" small, say a few psi, by allowing each increment of gas admitted to the bed to be in effect subjected to a multiplicity of operating cycles (typically more than ten), nitrogen and oxygen products of acceptable commercial purity may be produced by the method according to the invention. In this respect the invention is a radical departure in the art as prior processes have relied upon only one or a few operating cycles to effect separation.

The operating pressure will typically be chosen in accordance with the desired product pressure, typically 2 to 4 bars.

It may help towards a better understanding of the invention to draw an analogy between the column 2 and a rectification column of the kind typically used to separate mixtures of oxygen and nitrogen formed from air by initial extraction of water vapour and carbon dioxide. In a rectification column, a descending liquid phase undergoes mass transfer with an ascending phase. As the vapour phase ascends the column so it becomes richer in nitrogen (the more volatile constituent) and as the liquid phase descends the column so it becomes richer in oxygen (the less volatile constituent). Thus liquid oxygen collects at the bottom of the column and nitrogen

vapour rises to the top of the column. In the method according to the invention, the gas in the void spaces of the bed 8 is progressively richer in oxygen (the preferentially adsorbed constituent) as one descends the column end progressively rich in nitrogen as one ascends the column.

The invention is not limited to the use of carbon molecular sieve. Suppose the zeolite which is suitable for separation of nitrogen- oxygen mixtures and which adsorbs nitrogen in preference to oxygen is employed. Then, nitrogen-enriched air is collected at the bottom of the bed and oxygen-enriched air is collected at the top of the bed.

It is not possible to obtain both an absolutely pure oxygen fraction and an absolutely pure nitrogen fraction in the method according to the invention. Quite apart from the argon content of the air (which will tend to be collected in the unadsorbed gas fraction), a perfect separation is not possible. The operating parameters of the method and the positions of the outlets may thus be selected according to the desired purity of the respective fractions.

In order to utilise the pump 24 fully, two or more columns may be operated out of phase with one another to separate air. Referring now to Figure 2 of the accompanying drawings, a plant comprising two columns is illustrated. Like parts in Figures 1 and 2 are identified by the same reference numerals. The suffix (a) and the suffix (b) are used in Figure 2 to distinguish between the two columns.

The two columns 2(a) and 2(b) share a common source of compressed and purified air, as described with reference to Figure 1. When the bed 8(a) is being subjected to the compression step, the bed 8(b) is being subjected to the expansion or pressure reduction step, and vice versa. During the time that the bed 8(a) is subjected to the upward translation step, the bed 8(b) is subjected to the downward translation step and air feed steps, and vice versa. It is therefore necessary for each compression step to have the same duration as each pressure reduction step and for each upward translation step to have the same duration as each combined downward translation and air feed step so as to avoid discontinuous use of the pump, although such use may be reduced by means of a suitable buffer. The pump 24 has two inlet conduits 22(a) and 22(b) respectively and two outlet conduits 28(a) and 28(b) respectively. Thus, the pump 24 withdraws gas from the bed 8(a) and passes it to the reservoir 4(a) during the downward translation and air feed steps performed using the bed 8(a), and once the air feed step has ended the pump then withdraws gas from the bed 8(b) and passes it to the reservoir 4(b) during the downward translation and air feed steps performed using the bed 8(b). Thus, the pump is able to be used continuously.

The plant shown in Figure 3 is identical to that shown in Figure 2 save that the two reservoirs 4(a) and 4(b) are replaced by a common reservoir 4. Operation of the plant shown in Figure 3 is therefore identical to that shown in Figure 2.

The plant shown in Figure 4 is generally similar to that shown in Figure 1 save that a different arrangement is employed to withdraw gas from the bottom of the adsorption bed and to return it to the bed. A key feature of this different arrangement is that a vacuum pump such as a Roots blower is employed to subject the adsorption bed to a vacuum during the downward translation and air feed steps.

The plant shown in Figure 4 has a vertical column 62, reservoirs 64 and 66 associated with the lower end of the column 62 and a reservoir 68 associated with the upper end of the column 62. The column 62 contains a bed 70 of carbon molecular sieve capable of preferentially adsorbing oxygen from a gas mixture comprising oxygen and nitrogen. the bed 70 occupies substantially the whole internal volume of the column 62. The top end 72 of the bed 68 communicates with a conduit 74 which extends from the top of the column 62 to the reservoir 68. A valve 76 is located in the conduit 70. The bottom end 78 of the bed 70 communicates with a conduit 80 which extends from the bottom 78 of the column 62 to the top of a reservoir 64. A valve 82 is located in the conduit 80. the bottom of the reservoir 64 is connected to the inlet of a Roots blower 84 by a conduit 86. The outlet of the Roots blower 84 is connected by a conduit 88 to the lower end of the reservoir 66. A valve 90 is located in the conduit 88. A conduit 92 extends from the upper end of the reservoir 64 and terminates in the conduit 80 intermediate the valve 82 and the column 62. The valve 94 is located in the conduit 92.

The column 62 is also provided with an inlet 96 for compressed, purified air. The air may be compressed and purified by conventional plant, for example, of the kind shown in Figure 1 for this purpose. The inlet 96 is provided with a valve 98. The reservoir 68 is provided with an outlet 100 having a valve 102 disposed therein and the reservoir 66 is provided with an outlet 104 having a valve 106 disposed therein.

All the above mentioned valves may be pneumatically or hydraulically operated and the relative timings of their opening and closing may be effected by a central programmable controller (not shown) of a kind conventional in the art.

The operation of the plant shown in Figure 4 is generally similar to that shown in Figure 1 and will not be described again in detail, but some differences do arise as a result of the different arrangement of reservoirs associated with the bottom of the column. Thus, the pressure raising step is carried out with all valves closed save the valve 94 so that gas flows from the reservoir 66 through the conduit 92 into the bed 70. The upward translation step is initiated by opening the valve 76 and allowing gas to flow into the reservoir 68 from the bed 70 and thereby maintaining isobaric conditions in the bed 70. Product nitrogen is withdrawn during this step through outlet 100 and the valve 102 is opened to enable such withdrawal to take place. The pressure reduction step is initiated by opening valves 82 and 90 (simultaneously with the closing of valves 94 and 74) and operating the Roots blower 84 so as to withdraw gas from the bed 70 via the reservoir 64 and to pass this gas to the reservoir 66. The downward translation step commences with the opening of valve 76. At the end of the downward

translation step the valve 76 closes and valve 98 opens to admit air (free of carbon dioxide and water vapour) into the bed. During this step, the valve 106 is opened and product oxygen is withdrawn. The cycle of operations is performed repeatedly.

CLAIMS

1. A method of separating a feed gas mixture into a first gaseous fraction and a second gaseous fraction, comprising performing the following steps in repeated sequence.

(a) fractionally raising the pressure of a gas mixture which is resident in a bed of adsorbent capable of selectively adsorbing at least one constituent of the feed gas mixture and which comprises the same constituents as said feed gas mixture, and thereby adsorbing said at least one selectively adsorbable constituent;

(b) performing a first incremental translation at approximately constant pressure of the unadsorbed gas in one direction;

(c) fractionally reducing the pressure of the gas in the bed and thereby desorbing previously adsorbed gas from the adsorbent;

(d) performing a second incremental translation at approximately constant pressure of the gas in the bed in a direction opposite to the direction of translation in step (b),

wherein during the repeated performance of said steps first and second gaseous fractions of different composition from each other and from the feed gas mixture are withdrawn and the feed gas mixture for separation flows in the bed at a region intermediate its ends.

2. A method as claimed in claim 1, in which the pressure raising step is performed by allowing gas to flow into the adsorbent bed from a reservoir associated with one end of the bed by virtue of a pressure difference between the reservoir and the bed, and no gas is discharged from the bed during this step.

3. A method as claimed in claim 2, in which the first translation is performed by continuing to allow gas to flow into the adsorbent bed but allowing a substantially equal quantity of gas to be displaced from the other end of the bed, at least some of which displaced gas flows into another reservoir associated with that end.

4. A method as claimed in claim 2 or claim 3, in which the pressure reduction step is performed by pumping gas from said one end of the bed into a reservoir associated with that end.

5. A method as claimed in claim 4, in which the second translation is performed by continuing to pump gas into the reservoir but allowing a substantially equal quantity of gas to flow into said bed from the reservoir associated with said other end.

6. A method as claimed in claim 5, in which the feed gas mixture is introduced into said bed after said second translation step has been terminated by stopping flow into the bed from the reservoir associated with said other end of the bed, and introduction of said feed gas mixture into the bed is stopped before starting the next pressure raising step.

7. A method as claimed as claimed in claim 6, in which the pressure in the bed is maintained substantially constant during said introduction of the air feed.

8. A method as claimed in any one of the preceding claims, in which the feed gas mixture is air from which water vapour and carbon dioxide has been removed.

9. A method as claimed in any one of the preceding claims, in which each increment of gas admitted to the bed is on average subjected to at least 10 operating cycles.

10. A method of separating a gas mixture substantiall as herein described with reference to Figures 1, 2, 3 or 4 of the accompanying drawings.

11. Apparatus for performing the method claimed in claim 1, comprising a column containing a bed of adsorbent capable of selectively adsorbing at least one constituent of the feed gas mixture and having associated therewith an outlet for a first gaseous fraction, spaced apart from an outlet for a second gaseous fraction and an inlet for gas mixture to be separated intermediate the said two ends of the column; at least one first reservoir able to be placed in communication with one end of the column by operation of at least one first value, at least one second reservoir able to be placed in communication with the other end of the column by operation of at least one second valve, and means for extracting gas from one end of the column and passing it to the said reservoir associated with that end.

12. Apparatus as claimed in claim 11, wherein said gas extracting means is a pump.

13. Apparatus as claimed in claim 11, in which there are two reservoirs associated with said one end of the column, one such reservoir being adapted to receive gas from the bed, and the other such reservoir being adapted to pass gas to the bed, and a Roots blower or other vacuum pump is adapted to pass gas from the receiving reservoir to the other reservoir associated with said one end of the column.

14. Two apparatuses as claimed in claim 12 or claim 13 sharing a common pump or common Roots blower.

15. Apparatus for separating a gas mixture substantially as herein described with reference to any one of Figures 1 to 4 of the accompanying drawings.

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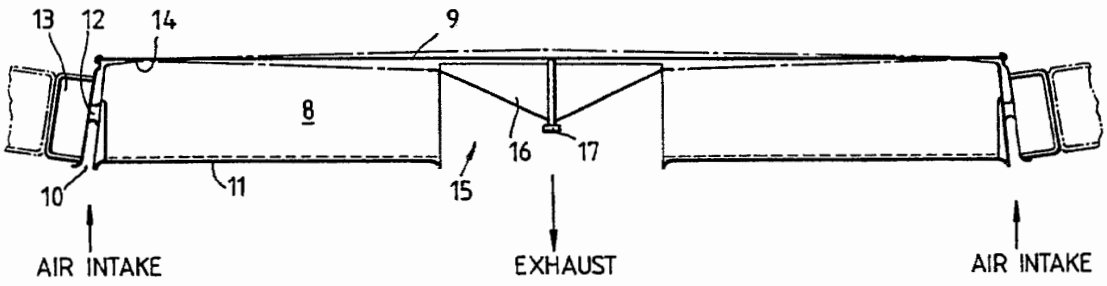
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# (54) Pressure swing gas separation.

(57) A pressure swing gas separator is operated with a pressure difference between its presurised and depressurised states of less than 0.1 bar. Preferably it operates at a repetition frequency greater than one cycle per second and preferably between 50 and 200 cycles per second. Preferby the pressure swing gas separator includes a bed of adsorbent material (16, 108, 147) and forms part of a resonant system. Means (1, 101, 140, 141) are included operating at the resonant frequency of the resonant system, to pressurise and depressurise the bed of adsorbent material (16, 108, 147) so that the pressure energy of the desorbed gas mixture is substantially recovered. The means may include a tuned diaphragm (109, 133, 141) adjacent but spaced from one face of the bed of adsorbent material (16, 108, 147), driving means (1, 101, 150) arranged to drive the diaphragm (109, 133, 141) at its resonant frequency towards and away from the bed of adsorbent material (16, 108, 147), valve means (110, 115, 131) to introduce a gas mixture to and exhaust a depleted gas mixture from, a space between the diaphragm (109, 133, 141) and the bed (16, 108, 147), and a gas outlet (112, 113, 149) in communication with the other face of the adsorbent bed (16, 108, 147). In use, the movement of the diaphragm towards and away from the adsorbent bed pressurises and depressurises it.

Fig.3.




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## Pressure Swing Gas Separation

Pressure swing gas separators include a bed of adsorbent material which preferentially adsorbs at least one gas from a mixture of gases. A pressure swing gas separator uses this preferential adsorption to preferentially remove that gas from a mixture of gases either to provide a gas mixture having a higher concentration of the preferentially adsorbed gas or a depleted gas mixture which has a lower concentration of the preferentially adsorbed gas. In such pressure swing gas separators the adsorbent bed is pressurised with the gas mixture and the preferentially adsorbed component is adsorbed onto the bed until the bed is saturated with that one component of the gas mixture. The bed is then vented and the gas mixture depleted by the preferentially adsorbed component is discharged. The bed is then subjected to a reduced pressure and under these circumstances the gas adsorbed onto the bed is desorbed to produce a gas which is rich in the gas which is preferentially adsorbed.

Recently lots of work has been carried out in this field particularly in the so called rapid pressure-swing adsorption regime where the cycle time of the above operation is typically less than 30 seconds and where the adsorbent bed is arranged so that the mixture of gases is introduced into the bed from one side and the gas mixture rich in the preferentially adsorbed component is removed from that same side after desorbtion but the gas or gases which are not preferentially adsorbed onto the adsorbent bed are removed from the opposite side of the adsorbent bed which is continuously vented. Examples of such systems are described in EP-A-0008619; EP-A-0013680; and EP-A-0055160.

According to a first aspect of this invention a pressure swing gas separator is operated with a pressure difference between its pressurised and depressurised states of less than 0.1 bar.

With existing rapid pressure swing adsorption gas separators the bed is subjected to a pressure swing of at least half and more usually two-thirds of a bar. A considerable amount of energy is required to provide such a large pressure swing. By operating the pressure swing gas separator in accordance with the first aspect of this invention over a very much lower pressure difference and over a pressure difference of less than 0.1 bar very much less energy is required although, typically, on each pressure swing, a lower volume of gas is produced.

According to a second aspect of this invention the pressure swing gas separator is operated at a repetition frequency greater than one cycle per second.

Preferably the pressure swing gas separator operates at a repetition frequency greater than 10 cycles per second and typically operates at a repetition frequency of between 50 and 200 cycles per second. Accordingly, a pressure swing gas separator in accordance with a second aspect of this invention is very much faster than the earlier proposals for rapid pressure swing adsorption by a factor of as much as one thousand. Preferably the pressure swing gas separator includes a combination of both the first and second aspects of this invention and, under these circumstances the high repetition frequency of its operation more than makes up for the low volume of gas produced in each cycle. A combination of the first and second aspects of this invention provide a good throughput of gas with only a low energy requirement.

According to a third aspect of this invention a pressure swing gas separator includes a bed of adsorbent material and forms part of a resonant system, the pressure swing gas separator including means operating at the resonant frequency of the resonant system to pressurise and depressurise the bed of adsorbent material so that the pressure energy of the desorbed gas mixture is substantially recovered.

By using a resonant system in accordance with the third aspect of this invention much of the energy of compression is conserved since it is possible to conserve the pressure energy contained in the gas component scavenged from the one side of the bed. With the arrangement in accordance with the third aspect of this invention it is substantially only the friction losses in the adsorbent bed which are lost. Accordingly, a further reduction in the amount of energy required to operate a pressure swing gas separator is achieved in accordance with this aspect of the present invention.

Preferably the first three aspects of this invention are combined. The inventor of this application has coined the expression "acoustic pressure swing gas separation" to describe this system.

EP-A-0267727 describes and claims a gas resonance device comprising a resonance tube which expands in cross-section along its length from one end to the other, a heat source located at one end of the resonance tube, and means to trigger oscillations in a gas in the resonance tube. The resonance tube is preferably of ogival form and the heat source and means that trigger oscillations in a gas in the resonance tube are preferably formed by a pulsed combustor having a pulse repetition frequency corresponding to a resonance frequency of the gas resonance tube. The pressure waves generated by such a thermally driven gas resonance device are described and claimed to operate a pressure swing gas separator.

The second and third aspects of this invention do not include the combinations disclosed and claimed



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in EP-A-0267727.

According to a fourth aspect of this invention a pressure swing gas separator includes a bed of adsorbent material, a tuned diaphragm adjacent but spaced from one face of the bed of adsorbent material, driving means arranged to drive the diaphragm at its resonant frequency towards and away from the bed of adsorbent material, valve means to introduce a gas mixture to and exhaust a depleted gas mixture from, a space between the diaphragm and the bed, and a gas outlet in communication with the other face of the adsorbent bed, in use, the movement of the diaphragm towards and away from the adsorbent bed pressurising and depressurising it.

The drive means may be formed by a thermally driven gas resonance device as described in EP-A-0267727 or may be formed by a gas resonance device comprising a pulsed combustor located in the middle of a substantially spherical resonance chamber. Such a thermally driven gas resonance device has the advantage that the wall friction losses discussed in EP-A-0267727 are eliminated altogether. The pulsed combustor produces a substantially spherical expanding wave front which encounters no wall friction whatsoever as it moves from the middle of the resonance chamber to the outside of the resonance chamber.

Preferably the gas resonance device includes means to provide an explosive gas mixture to the pulsed combustor. Preferably the pulsed combustor has a pulse repetition frequency corresponding to the resonant frequency of the substantially spherical resonance chamber which, largely, is dependent upon its radius. The means to supply the explosive mixture to the pulsed combustor may itself include a tuned valve or a resonance tube so that the pulsed combustor receives a pulsed supply of explosive mixture. In this case the explosive mixture is preferably supplied at the resonance frequency of the substantially spherical resonance chamber.

Preferably the pressure swing gas separator comprises a bed of molecular sieve material located at the periphery of the substantially spherical resonance chamber. Preferably the resonance chamber is formed as an icosahedron. In this case each bed of molecular sieve material preferably has a generally circular configuration with the beds being located in the side wall of the resonance chamber with three circular beds contained wholly within each substantially triangular segment of the icosahedron and one bed at each point of the iscosahedron.

The use of a diaphragm separating the inside of the resonance chamber from the bed of molecular sieve material may be used with resonance chambers that are generally conical as described in EP-A-0267727 or with resonance chambers that are substantially spherical. The mechanical oscillations of the gas cause corresponding oscillation of the diaphragm but the diaphragm provides a physical barrier which separates the bed of adsorbent material from the inside of the resonance chamber and so prevents combustion products from the pulsed combustor contaminating the bed of adsorbent material. Preferably the diameter, thickness and stiffness of the diaphragm are selected so that the diaphragm has a natural resonant frequency corresponding to that of the resonance chamber.

The drive means may be formed by an electrical actuator. In this case, it is preferred that the electrical actuator comprises an armature coil and a stator one of which is connected to the diaphragm and the other of which is held by a fixed surrounding framework, in use, an oscillating current applied to the armature causing the armature, and hence the diaphragm, to oscillate with respect to the stator. The stator may be formed by a permanent magnet but is preferably formed by an electromagnet. When the electrical actuator is powered from the mains it is preferred that the diaphragm is tuned to the mains frequency of 50 or 60 Hz and that the device is driven from a single phase supply.

The drive means may, alternatively, be formed by a motor, either an electric motor, or an internal combustion engine. In this case the diaphragm may be coupled to a rotary shaft of the motor via a crank or other eccentric connection or the motor may be mounted on the diaphragm and be arranged to rotate an unbalanced load.

Preferably the valve means to introduce gas mixture into, and exhaust gas mixture from, the space between the diaphragm and the adsorbent bed comprises an annular valve seat which engages the diaphragm and the diaphragm forms the valve closure member which forms a gas-tight seal against the annular diaphragm but which, when the diaphragm moves away from the adsorbent bed unseats to allow gas to pass between the seat and the diaphragm.

The valve means may include an exhaust valve located in the middle of the diaphragm so that the gas mixture moves radially inwards from the annular valve seat towards the middle of the diaphragm. In this case the exhaust valve is arranged to open after the inlet valve but closes at substantially the same time as the inlet valve. One way of achieving this is to have the exhaust valve closure member free to slide within limits on a spindle mounted on the diaphragm so that, in use, as the exhaust valve closure member moves with the diaphragm away from the adsorbent bed it is in a first position on the spindle but as it moves with



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the diaphragm towards the adsorbent bed it is in a second position on the spindle.

Alternatively the valve formed by the annular valve seat and the dipahragm acts as both an inlet and an exhaust valve. In this case it is preferred that a harmonic vibration is applied to the diaphragm so that, in addition to its fundamental oscillation towards and away from the adsorbent bed there is also a superimposed secondary vibration which appears as a wave moving from one side to the other of the diaphragm. In this way as the diaphragm is moving away from the bed one side of the diaphragm lifts from its annular seat initially. A reduced pressure generated in the bed whilst the diaphragm is moving away from it draws gas mixture into the space between the diaphragm and the bed via the unseated diaphragm valve seat on the one side of the diaphragm. After a delay the other side of the dipahragm also unseats from the annular seat. The gas introduced into the one side of the diaphragm has a sideways momentum which is continued and encouraged by the flexing of the diaphragm and causes a gas flow from the one side to the other side of the diaphragm which causes the depleted gas from the space between the bed and the diaphragm to be exhausted from the other side of the diaphragm. As the diaphragm returns towards the adsorbent bed the one side of the diaphragm seats first against the annular valve seat followed by the other side. As the diaphragm continues to move towards the adsorbent bed it pressurises the bed and then moves away from the bed to depressurise it whereupon the one side of the diaphragm again lifts from its valve seat first and the cycle is repeated. In this way not only is a pressurising and depressurising operation carried out on the adsorbent bed by the fundamental oscillation of the diaphragm but also, as a result of the harmonic oscillation imposed upon the diaphragm, a transverse wave motion on the diaphragm is created which drives the gas transversely across the top of the adsorbent bed. This provides particularly effective scavenge operation which scavenges the depleted gas mixture from the top of the bed on each oscillation of the diaphragm.

This additional harmonic motion can be applied to the diaphragm by, for example, applying an eccentric mass to one side of the diaphragm, preferably matched by a corresponding lightening of the diaphragm on the other side. This is particularly useful when the diaphragm is driven by a heat engine. Alternatively when the diaphragm is driven by an electrical actuator or a mechanical connection this is located off centre. When the diaphragm is driven via a crank this additional harmonic oscillation can also be achieved by directly coupling one end of the crank to the diaphragm so that the angular tilting of a connecting rod connecting the crank to the diaphragm is applied directly to and results in a corresponding flexing of the diaphragm.

Preferably the pressure swing gas separator in accordance with this invention is arranged so that, in use, the gas mixture enters the bed at a bed entry velocity which is sufficiently low to ensure laminar flow of the gas takes place inside the adsorbent bed. The bed entry velocity at which laminar flow occurs is a function of the particle size of the bed, as will be explained in more detail subsequently, and accordingly, it is preferred that the particle size of the bed is substantially a quarter of a millimetre or less. By establishing laminar flow in the bed firstly the friction losses which occur in the acoustic pressure swing gas separator system are reduced which gives rise to a further reduction in the energy required for separation. However, in addition to this, with laminar flow a more efficient separation occurs between the depleted gas mixture and that which is rich in the gas which is not adsorbed by the adsorbent bed moving from the one face to the other face of the adsorbent bed.

Preferably the adsorbent bed is of a wide and shallow configuration in contrast to the arrangements of adsorbent bed used in the conventional rapid pressure swing adsorption systems in which a relatively narrow but deep bed is required for maximum efficiency. Typically the volume of the material in the adsorbent bed is generally similar to that used in the rapid pressure swing adsorption systems for equivalent production of preferred gas.

Various examples of and a discussion of some of the theory behind gas pressure swing separation in accordance with this invention will now be described with reference to the accompanying drawings, in which:-

Figure 1 is a cross-section through a first example;

Figure 2 is a partially cut-away plan of the first example;

Figure 3 is a section through a first example of gas-separator module;

Figure 4 is a graph showing the displacement with respect to time of the valves of the first example of gas-separator module;

Figure 5 is a section through a second example of gas-separator module;

Figure 6 is a cross-section to an enlarged scale through the diaphragm;

Figure 7 is a series of cross-sections through the diaphragm of the second example of gas-separator module showing how it flexes; and,

Figure 8 is a cross-section through a second example.



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A thermally driven gas resonance device comprises a generally spherical chamber 1 and a pulsed combustor 2 positioned centrally within the chamber. The combustor 2 includes a tuned non-return valve such as that described in our earlier application EP-A-0267727. A combustion mixture of air and an appropriate fuel gas is admitted to the chamber 1 via the tuned non-return valve. The combustion mixture is periodically ignited at a frequency matched to the resonant frequency of the spherical chamber 1. A sparking plug is provided in the combustor to cause the initial ignition but subsequently the pulsed combustion is self-sustaining.

As an alternative to the use of a tuned non-return valve in the combustor the combustion mixture may be supplied to the chamber 1 via a resonant gas-supply tube which extends into the chamber 1 and is tuned to supply the combustion mixture in pulses at the required pulse repetition frequency.

The operation of the combustor in the manner described above provides a pulsed heat source at the centre of the chamber 1 which produces a substantially spherical expanding wave front. Since the frequency of the combustor is matched to the resonant frequency of the chamber the combustor drives resonant modes of the chamber, maximising the efficiency with which the heat energy of combustion is converted into the mechanical energy associated with the pressure swings in the gas within the chamber 1. In the first example described this mechanical energy is used to drive a gas separator used for oxygen enrichment.

In the first example of the present invention shown in Figures 1 and 2 the generally spherical chamber 1 is defined by a hollow icosohedral shell 3 formed of generally triangular elements 4. Open-ended exhaust tubes 5 extend into the chamber 1 from the outer surface of the shell 3. The radially innermost ends of the tubes 5 lie at points spaced from the centre of the spherical chamber 1 by about three-quarters of the sphere radius. There is a pressure null point in the resonant mode of the chamber at this radius. Combustion products can therefore be vented from the chamber 1 at these points with minimum power loss and external noise. If it is required to take the exhaust further away from the chamber 1 then the tubes 5 may project beyond the surface of the shell 3.

Pipes connected to an external water supply (not shown) are positioned concentrically within the exhaust tubes 5. These pipes provide inwardly directed water sprays. A small proportion of the water is converted to steam by the heat source during thermal pulses and so supplements the production of mechanical power. The remaining mist of water droplets cools the gases in the chamber during the subsequent expansion cycle, prior to recompression, so removing excess heat energy and further increasing the efficiency of the device. Heated water aggregated from water droplets returned to the internal spherical surface of the chamber 1 is drained away at the base and may be cooled and recirculated.

The modules in the chamber 1 are formed as gas-separator modules such as that shown with its outer surface cut-away in the element 4′ of Figure 2. Each gas-separator module comprises a number of circular elements 7, each element 7 comprising a dished zeolite bed 8 and a diaphragm 9 covering the bed 8 on its inwardly facing surface. Each element 7 is 0.8 metres in diameter and up to 72 such elements may be provided across the inner surface of a chamber 1 or 2 metres radius. In use, the pressure-swings at the periphery of the chamber 1 drive the diaphragm 8 back and forth in the radial direction, that is the direction transverse to the surface of the bed 8. Air is drawn in through an annular inlet 10 surrounding the periphery of the bed 8 and driven through the bed 8 in the manner described in further detail below. Oxygen-enriched gas leaving the bed 8 at its lower surface is collected under a skirt 11 and transferred via ducts 12 to the tubular framework 13 of the icosohedral shell 3. The tubular framework 13 is connected in common to all the gas separator modules 6 and provides a path from the modules 6 to an outlet (not shown) from which the the oxygen-enriched gas is piped away.

As well as serving to isolate the zeolite bed 8 from the combustion gases within the chamber 1 the diaphragm 9 also serves to provide a valving arrangement which ensures that air is admitted and depleted air exhausted at appropriate points in the oscillatory cycle. The diaphragm 9 is dimensioned to have the same resonant frequency as the chamber 1. In the present example the diaphragm 9 is formed of steel with a diameter of 0.8 metres and a thickness of 6 mm. The diaphragm 9 is suspended at its edges outside the bed 8 so that in its equilibrium position it clears the periphery of the bed 8 but touches the bed 8 at a valve seat 14 formed some 10 cm in from the outer edge of the bed 8. The part of the bed 8 extending radially outwards from this valve seat 14 is sealed so as to be impervious to air and the region of contact between the diaphragm 9 and the bed 8 serves to provide a valve which controls the intake of air from the port 10 to the main part of the bed 8. In the centre of the bed 8 there is a cylindrical exhaust outlet 15 which, when the diaphragm is in its equilibrium position, is sealed by a conical valve member 16. The valve member 16 rides on a spindle 17 fixed to the centre of the diaphragm 9.

The operational cycle of the pressure-swing gas separator is now described with reference to Figure 4 which shows the displacement of the inlet valve and the outlet valve with respect to time. At time $t_0$ the



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diaphragm 9 is in its equilibrium position. Subsequently as the pressure in the gas in the chamber immediately adjacent the diaphragm drops, the diaphragm is drawn back in the radially inwards direction away from the bed 8. The diaphragm 9 immediately clears the seat 14 thereby opening the valve inlet. However the exhaust valve remains closed since there is free movement between the spindle 17 and the valve member 16 so that although the spindle 17 is drawn up the valve member 16 remains seated. As a result during the period $t_0$ to $t_1$ a partial vacuum is created under the diaphragm 9 which draws in and accelerates air from the inlet 10 over the seat 14. At time $t_1$ as the diaphragm 9 moves further upwards the end of the spindle 17 engages the valve member 15, unseating it and so opening the exhaust valve. The air drawn under the diaphragm 9 from the inlet 10 is therefore drawn across the bed 8 and its momentum encourages it to continue to move radially in to the centre of the bed 8. As it does so it scavenges the depleted air initially present between the bed 8 and the diaphragm 9 and drives it out through the exhaust outlet 15.

Having reached its point of maximum displacement the diaphragm 9 then moves downwards towards the bed 8 again. At time $t_2$ the diaphragm 9 again reaches its equilibrium position and closes the inlet and exhaust valves. It should be noted that although there is a delay between the opening of the inlet and outlet valve resulting from the play on the spindle 17 when they close at time $t_2$ they do so simultaneously. During the subsequent phase of the cycle the pressure under the diaphragm 9 increases driving the fresh air drawn through the annular inlet 10 into the zeolite bed 8. As the air moves forwards through the bed 8 the zeolite acts as a molecular sieve, preferentially adsorbing the nitrogen from the air so that the gas passing out through the bottom of the bed 8 is oxygen enriched/nitrogen depleted. Then from time $t_3$ which is point of maximum displacement in the opposite direction, towards the bed 8, the diaphragm 9 moves back towards its equilibrium position reducing the pressure above the bed 8. Under this reduced pressure nitrogen is desorbed from the bed 8 and drawn back towards the surface. As the diaphragm 9 reaches its equilibrum point the cycle is repeated with the next intake of air driving the oxygen depleted, nitrogen-rich gas from the surface of the bed 8 to the exhaust outlet 15. The cycle accelerates the gas throughout its passage across the face of the bed so that when it reaches the outlet 15 its momentum carries it away from the chamber 1 as a puff of gas which does not mix with the intake to the bed. During successive cycles the oxygen is driven down through the bed to its outer surface where it is conducted away in the manner already described.

A modification of the gas separator module is shown in Figures 5, 6 and 7. In this example an annular valve seat 31 surrounds a dished bed of adsorbent material 32. A thick tuned diaphragm 33 resonates at the resonance frequency of the substantially spherical chamber 1 (not shown in Figure 5). The bed 32 and annular valve seat 31 are generally similar to the bed 8 and seat 14 of the first example. The diaphragm 33 is unbalanced and includes an added mass 34 on one side and a cut-away portion 35, shown in Figure 6, on its other side. This assymetry in the diaphragm 33 leads to a harmonic oscillation being generated in the diaphragm 33 as it is oscillated backwards and forwards in the radial direction at the fundamental frequency by the spherical wavefronts propagating in the chamber 1. This harmonic oscillation has the form of a transverse wave moving from left to right as shown in Figure 5.

Figure 7 illustrates this effect diagrammatically. In Figure 7 the diaphragm is represented by a line with the amplitude of the transverse oscillation greatly exaggerated to illustrate how, as the diaphragm 33 is moving away from the bed 32, first the left hand side, as shown in Figures 5, 6 and 7 lifts away from the annular seat 31 so allowing fresh air to be drawn in to the gap formed between the diaphragm 33 and the valve seat 31 at the left hand side of the gas separator module. Further movement of the diaphragm 33 away from the adsorbent bed 32 further reduces the pressure above the adsorbent bed 32, draws in more fresh air from the left hand side of the bed 32 and draws out more of the depleted nitrogen rich gas from the bed 32. As the fresh air moves inwards into the space between the bed 32 and diaphragm 33 it has a significant velocity in a direction from left to right as shown in Figures 5 and 6. After reaching its maximum deflection the diaphragm 33 starts to return towards the dished bed 32. Again the left hand side of the diaphragm moves first, as shown most clearly in Figure 7 and this leads to the left hand side of the diaphragm 33 forming a seal with the annular seat 31 at the left hand side of the diaphragm whilst the right hand side of the diaphragm is still out of contact with the right hand side of the annular seat 31, again as shown in Figure 7. The transverse movement of air across the face of the dished bed 32 leads to an effective scavenge of the depleted, nitrogen rich air from the surface of the bed 32 and out of the right hand side of the gas separator module between the right hand side of the annular seat 31 and the diaphragm 33. As the diaphragm 33 continues to move towards the adsorbent bed it eventually seats against the entire circumference of the annular seal 31 so closing the valve and pressurising the fresh air introduced into the module and forcing it into the adsorbent bed 32. After reaching a point of maximum deflection towards the bed 32 the diaphragm again starts to return and move away from the bed 32. Again it is the left hand side



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of the diaphragm which first lifts from its annular seat 31 to allow fresh air to enter the module from the left hand side and the process is repeated. The transverse wave action that is induced in the diaphragm 33 also encourages the movement of air in the transverse direction from the left hand side to the right hand side of the gas separator module.

A pressure-swing gas separator such as that described above offers both greater efficiency and lower capital costs by comparison with a conventional oxygen production plant such as a cryogenic oxygen recovery unit. A 2 metre radius sphere having its surface entirely covered by gas-separation modules is capable of producing luo tonnes per day of 98% oxygen with an oxygen recovery ratio, that is the ratio by volume of oxygen input to oxygen output, of 100:15 and a specific energy consumption, that is the mechanical energy input required to produce 1 kilogram of oxygen, of 0.125 kW hr/kg. The 98% oxygen mixture produced is suitable for use in industrial processes such as iron and steel-making but may also be used for other purposes such as sewage-treatment or the generation of oxgyen for medical use. For these alternative uses the scale of the device and the concentration of the oxygen produced may be varied to suit the needs of the particular application. Where only low volumes of oxygen are required a gas-separator unit formed by a single circular bed and diaphragm such as that described above may be used in an ogival gas resonance device such as that described in the European application cited above.

The second example of pressure swing gas separator uses a bed and diaphragm arrangement similar to that described in the modification of the first example but the diaphragm is driven by an electrical actuator 40. The gas separator comprises a diaphragm 41 having its periphery clamped between an O-ring 42 and a number of equiangularly spaced balls 43. The O-ring 42 is received in an annular groove in the top part of a housing 44 whilst the balls 43 are received in the bottom part 45 of the housing. The two halves of the housing are clamped together by bolts 46 to clamping the diaphragm 41 between the O-ring 42 and balls 43. A dished bed 47 filled with adsorbent material is located in the bottom half 45 of the housing and surrounded by an annular seal 48 which extends close to the periphery of the diaphragm 41. A gas outlet 49 in communication with the rear face of the bed 47 leads away from the lower half 45 of the housing. The upper part 44 of the housing supports the electrical actuator 40 which consists of a stator 50 which may be a permanent magnet or preferably, is an electro-magnetic energised by a DC field coil. An armature, 51 formed by a moving coil is connected to the diaphragm 41 eccentrically. Figure 8 illustrates the offset OFF between the centre of the diaphragm and the centre of the armature. The arrangement of the electrical actuator 40 is generally similar to that used for driving loudspeakers.

Typically a single phase mains AC current is connected to the moving coil armature and, in response to the alternating current the armature moves up and down, as shown in Figure 8 to cause the diaphragm to oscillate. The diaphragm is arranged to oscillate at the mains frequency. Because of the lateral offset between the axes of the diaphragm and that of the armature the diaphragm oscillates as described above with reference to Figure 7.

Outline of theory behind present invention

Adsorption/desorbtion with rising and falling fluid pressure is capable of separation of species, such as nitrogen and oxygen from air using a zeolite molecular sieve in the form of a porous bed of graded or uniformly sized particles. Typically, mixed gases are introduced at the top of the bed, and one component is withdrawn at a lower flow rate at the base of the bed. Throughputs are determined by the required purity of the separated fraction. The energy efficiency of separation is potentially high for provision of separated gas delivered at atmospheric pressure, and is already higher in practical applications than with any of the cryogenic separation routes.

Molecular selectivity is the basis for separations conducted by adsorption and it is known, for instance, that nitrogen is preferentially adsorbed at a zeolite surface because of its different molecular configuration from that of oxygen. The adsorption process is also completely reversible with change of pressure. The molecular selectivity of zeolite for nitrogen from $N_2/O_2$ mixtures is held to be 2.8 over a wide range of pressures at ambient temperatures. Since molecular processes are intrinsically fast, it is evident that rates of adsorption are limited by diffusion rather than molecular adsorption, since inert components such as argon in air are invariably present and effectively form a barrier between the nitrogen and the adsorbent bed. Furthermore, since adiabatic compression is also reversible, much of the energy of compression may be conserved if a resonant acoustic pressurisation system is used, so that the two dominant factors with regard to energy efficiency in the improved process are likely to be frictional flow resistance in the bed, coupled with scavenge flow resistance at atmospheric pressure at the top of the bed. These factors are now examined.



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Selective adsorption

The adsorption of two species B and C of similar densities at a clean surface is considered to be governed by the product of the molecular selectivity s and the relative rates of arrival of the species at the surface, in terms of an inert boundary layer of species A and thickness $\delta$ impeding the flow to the surface, and the mutual diffusivity D of the other two species through this boundary layer. Stefan's law indicates that the overall mass diffusion rate q per unit area of adsorbed species at the interface is,

$$q = -\frac{D\rho_o}{\delta} \ln c_A \qquad [1]$$

where $\rho_o$ is the overall density and $c_A$ is the small fractional concentration of the inert species A. (The negative sign accounts for the negative logarithm). A qualitative evaluation of equation [1] shows that comparatively high rates of adsorption can be foreseen in an adsorption bed of small particles.

Ignoring density differences, if the species B and C are present as fractional volume concentrations $c_B$ and $c_C$ (such that $c_B + c_C + c_A = 1$) it may be postulated that the effect of the molecular selectivity s, referring to species B and C respectively, is that

$$\frac{q_B}{q} = \frac{sc_B}{sc_B + c_C} \quad , \quad \frac{q_C}{q} = \frac{c_C}{sc_B + c_C} \quad , \quad \text{and } q_B + q_C = q \qquad [2]$$

Equation [2] demonstrates that the instantaneous effective selectivity $\sigma$ in the presence of the boundary layer is simply,

$$\sigma = \frac{q_B}{q_C} = \frac{sc_B}{c_C} \qquad [3]$$

which exceeds s at the start of the process and is progressively reduced.

In order to follow the changes of concentration it is necessary to postulate an initial volume V of mixed gases approaching the interface, progressively reduced in time by adsorption (density differences between species B and C are ignored, together with $c_A$ at this stage). Then,

$$c_B = \frac{V_B - \int q_B\, dt}{V - qt} \quad , \quad c_C = \frac{V_C - \int q_C\, dt}{V - qt} \quad , \quad c_B + c_C = 1 \qquad [4]$$

Dividing by V, putting $\frac{qt}{V} = x$, where x is the proportion of mixed gas which is adsorbed, and substituting the $q_B$ and $q_C$ from equation [2],

$$c_B = \frac{c_{B.0} - \int \frac{sc_B\, dx}{(s-1)c_B + 1}}{1 - x} \quad , \quad c_C = \frac{c_{C.0} - \int \frac{c_C\, dx}{(1-s)c_C + s}}{1 - x} \qquad [5]$$

where $c_{B.0}$ and $c_{C.0}$ are initial values. From the first of these, by differentiation and separation of variables,

$$\frac{dx}{1 - x} = -\frac{(s-1)c_B + 1}{(s-1)c_B(1 - c_B)} \cdot dc_B \qquad [6]$$

which leads by integration of both sides and insertion of limits to,



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$$1 - x = \left[ \frac{(1 - c_{B.0})^s}{c_{B.0}} \cdot \frac{c_B}{(1 - c_B)^s} \right]^{\frac{1}{s-1}} \quad [7]$$

Formulation in terms of $c_C$ in equations [5], [6] and [7] leads as expected to an identical result. Equation [7] may be written as

$$1 - x = \left( \frac{k_0}{k_n} \right)^{\frac{1}{s-1}}$$

$$\text{if } k = \frac{(1 - c)^s}{c} .$$

Then, for successive pressure swings relating to the non-adsorbed delivery fraction, corresponding with concentration terms $k_0$, $k_1$, $k_2$,....$k_n$, the cumulative value $\overline{1 - x}$ is given by,

$$\overline{1 - x} = \left[ \frac{k_0}{k_1} \cdot \frac{k_1}{k_2} \cdot \frac{k_2}{k_3} \cdots \frac{k_{n-1}}{k_n} \right]^{\frac{1}{1-s}} = 1 - x \quad [8]$$

so that the incremental result is the same as for one pressure swing.

Laminar flow within the bed

The Reynold's numbers for small particles and small velocities being small (<100), an assumption of laminar flow is well-founded. If z is distance below the entry surface of the bed the mass flow per unit area of bed may be defined as $q_o \frac{dp}{dz}$, where p is pressure excess, and the adsorption mass flow rate is Qp per unit bed volume. Then the pressure decay through the bed is governed by,

$$q_o \frac{d^2p}{dz^2} = Qp, \quad \text{such that} \quad p = Ae^{-\sqrt{\frac{Q}{q_o}} z} \quad [9]$$

and the active bed depth and proportional flow rate are unchanged whether A is constant or proportional to sin wt (where w is angular frequency and t is time), provided that Q is constant (i.e. not limited by saturation of adsorption) and laminar flow conditions are maintained. This simply expressed and important result demonstrates that operating frequency can be varied within wide limits. It is convenient in the following to put $A = p\, p_o$, where p is non-dimensional and $p_o$ is atmospheric pressure.

In bed volume V, close packed spheres of uniform diameter d each occupy the volume $\frac{\pi d^3}{6}$. The number n in volume V with packing space ratio $\eta$ is $\frac{6\eta V}{\pi d^3}$. The aggregate surface area in unit volume is

$$\pi d^2 \frac{6\eta}{\pi d^3} = \frac{6\eta}{d} ,$$

hence the equivalent thickness of parallel channel is



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$$\frac{(1-\eta)}{3\eta}\, d \quad .$$

Recognising that the dimensions of the area $\frac{6\eta}{d}$ are $\frac{L^3}{L} = L^2$, the attributions of mean path length and channel cross-section width, per unit volume, are $(3\eta)^{\frac{1}{2}}$ and

$$(3\eta)^{\frac{1}{2}},$$

respectively, which account for pore volume ration $1^{d}- \eta$ when multiplied together and with channel thickness

$$\frac{(1-\eta)}{3\eta}\, d \, .$$

The effective pressure gradient is then

$$\frac{p' p_o}{(3\eta)^{\frac{1}{2}}}$$

to account for the stretched path, where $p'$ is a non-dimensional pressure gardient.

The laminar flow rate $q = q_0 p'$ is given by,

$$q = \frac{(3\eta)^{\frac{1}{2}}}{d} \cdot \frac{p' p_o \rho_o}{(3\eta)^{\frac{1}{2}}} \left(\frac{1-\eta}{3\eta}\right)^3 \frac{d^2}{12\mu} = \frac{p' p_o \rho_o d^2}{12\mu} \left(\frac{1-\eta}{3\eta}\right)^3 \qquad [10]$$

where $\mu$ is viscosity, of dimensions $\frac{M}{LT}$.

The corresponding adsorption mass flow rate is given by,

$$Q = \frac{6\eta}{d} \left(\frac{6\eta}{1-\eta}\right) \frac{p\, \rho_o D \ln c_A}{d} = \frac{p\, \rho_o\, D \ln c_A (6\eta)^2}{d^2 (1-\eta)} \qquad [11]$$

so that the ratio $\sqrt{\frac{Q}{q_o}} = \frac{1}{a}$ is then given by,

$$a = \left(\frac{1-\eta}{6\eta}\right)^2 \frac{d^2}{3} \sqrt{\frac{p_o}{\eta\, \mu\, D \ln c_A}} \qquad [12]$$

and the bed entry velocity v by,

$$v = p\, \frac{1-\eta}{3\eta} \sqrt{\frac{\eta\, p_o\, D \ln c_A}{\mu}} \qquad [13]$$

It may be noted from equation [12] that active bed depth, which is a multiple of a, is proportional to $d^2$, whereas

from equation [13], v is independent of d. Both expressions show a significant influence of packing ratio $\eta$,



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since both active bed depth and v are reduced as $\eta$ is increased.

It may further be noted that average work rate per unit bed area during cyclic operations is

$$\frac{P\ P_o\ v}{2},$$

and that the component of specific energy of separation attributed to bed friction (kWhr/m$^3$ of component) is

$$\frac{\pi p}{72(1 - x)},$$

where 1 - x is the recovery ratio related to a particular concentration, derived from equation [7]. These expressions continue to be independent of operating frequency, and the specific energy is also independent of v. The relevant expression may be applied similarly to characterise non-resonant separation beds, and is well correlated with results obtained from them. The enhanced economy of resonant operation with much lower pressure amplitudes in accordance with this invention is thereby emphasized.

Bed flow model comparison

Bead volume = $\frac{\pi d^3}{6}$

Number of beads $\frac{6\eta}{\pi d^3}$ per unit volume.

The comparison of results shown below arises from the alternative of a tube nest model (bore diameter D) to the original model envisaging a stack of separated plates. The results are similar, but lead to simpler expressions indicative of smaller adsorptions but larger mass flow rates, entry velocities, and active bed lengths. As such, they are probably optimistic, and the plate model is preferred. The difference is small, about 20%, for the important bed entry velocity criterion V.

Aggregate area $\frac{6\eta}{d}$ per unit volume.



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| Plate Analogy | Tube Analogy |
|---|---|
| Channel face area $2 \times \frac{3\eta}{d}$ | Wall face area $m\pi D = \frac{6\eta}{d}$ |
| Channel thickness $\frac{1-\eta}{3\eta}d$ | Tube volume $\frac{\pi D^2}{m\,4} = 1-\eta$ |
| Channel width $\frac{(3\eta)^{\frac{1}{2}}}{d}$ | Tube diameter $D = \frac{2(1-\eta)}{3\eta}d$ |
| Path length $(3\eta)^{\frac{1}{2}}$ | Number of tubes $m = \frac{(3\eta)^2}{\pi d^2(1-\eta)}$ |
| Pore volume ratio $(1-\eta)$ | |
| Boundary layer thickness $\frac{(1-\eta)}{6\eta}d$ | Boundary layer thickness $\frac{(1-\eta)}{3\eta}d$ |
| Mass flow rate $q = \frac{p'po\rho od^2}{12\mu}\left(\frac{1-\eta}{3\eta}\right)^3$ | Mass flow rate $q = \frac{p'po\rho od^2}{8\mu}\frac{(1-\eta)^3}{(3\eta)^2}$ ; $\left(\times \frac{9\eta}{2}\right)$ |
| Mass adsorption $Q = \frac{po\rho oDlnC_A(6\eta)^2}{d^2(1-\eta)}$ | Mass adsorption $Q = \frac{po\rho oDlnC_A(6\eta)^2}{2d^2(1-\eta)}$ ; $(\times\frac{1}{2})$ |
| $a = \sqrt{\frac{q}{Q}} = \left(\frac{1-\eta}{6\eta}\right)^2 \frac{d^2}{3}\sqrt{\frac{po}{\eta\mu DlnC_A}}$ | $a = \sqrt{\frac{q}{Q}} = \left(\frac{1-\eta}{6\eta}\right)^2 d^2\sqrt{\frac{po}{\mu DlnC_A}}$ ; $(\times 3\sqrt{\eta})$ |
| $V = p\frac{1-\eta}{3\eta}\sqrt{\frac{\eta poDlnC_A}{\mu}}$ | $V = p\frac{1-\eta}{2}\sqrt{\frac{poDlnC_A}{\mu}}$ ; $\left(\times \frac{3\sqrt{\eta}}{2}\right)$ |

By way of conservatism both models incorporate the maximum possible boundary layer thickness, and neglect adsorption surfaces within the beads. However, they do not account for the irregularities of cross-section of the gas paths, which will have less effect with laminar flow than with turbulent flow.

The distinctions between acoustic pressure swing gas separation and rapid pressure swing adsorption.

The most obvious, but superficial difference is one of frequency, where the rapid pressure swing adsorption system (RPSA) employs sequenced valves to alternately pressurise and depressurise, and the acoustic system uses resonant oscillations in order to conserve the pressure energy contained in the gas component rejected after separation of another component. On this basis the usable frequency bands of the two systems are likely to be contiguous, and without overlap.

The bed loading analysis which this statement supports verifies that smaller pressure swing amplitudes and entry velocities are preferred for the acoustic variant than for RPSA in order to reduce bed friction losses. For a given throughput bed area is therefore larger with the acoustic variant, but is compensated by



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much less depth. A substantial increase of energy efficiency of separation is obtained in this way for acoustic separation.

The use of small beads (<250 μm) is preferred for both variants if pure gas is required from the separation. This may be attributed to the preponderance of laminar flow over turbulent flow through the bed in such cases, so that turbulent back-diffusion and therefore loss of the separated component is suppressed. The criterion for laminar flow is a low value of Reynold's number, essentially dependent upon the product of bead size and flow velocity. Laminar flow is therefore better established for the acoustic variant at a given bead size with lower bed entry velocities.

The most fundamental distinction between RPSA and acoustic pressure swing separation by adsorption, is that the former makes use of saturation of adsorption at bed entry conditions, whereas the acoustic variant does not. Thus, with RPSA when pressure is applied it rises quickly and then stays constant at entry for most of the cycle while the bed is charged. A wave of pressure and adsorption then passes down the bed, although with exponential type decay of amplitude. This accounts for the need for comparatively long beds, but extends the throughput. As shown in the analysis, a sufficient degree of adsorption is obtainable at enhanced frequency to obviate the need to make use of saturation, and this is why the acoustic bed can achieve the same volumetric loadings at lower entry pressures and velocities and higher energy efficiencies.

Both variants use a superimposed steady gas flow through the bed to extract the separated component, and the proportion extracted in this way determines its purity. Of necessity therefore, both variants make use of saturation at the low, constant pressure at the base of the bed, since adsorption eventually ceases when pressure is constant at any value.

In summary, the bed loading criterion for acoustic pressure swing separation is entry velocity and not frequency. At high frequencies bed diameters are preferably small in order to limit energy losses from high scavenge velocities.

## Claims

1. A pressure swing gas separator operating with a pressure difference between its presurised and depressurised states of less than 0.1 bar.

2. A pressure swing gas separator operating at a repetition frequency greater than one cycle per second.

3. A pressure swing gas separator including a bed of adsorbent material (16, 108, 147) and forming part of a resonant system, the pressure swing gas separator including means (1, 101, 140, 141) operating at the resonant frequency of the resonant system to pressurise and depressurise the bed of adsorbent material so that the pressure energy of the desorbed gas mixture is substantially recovered.

4. A pressure swing gas separator according to claim 2 or 3, arranged to operate at a repetition frequency of between 50 and 200 cycles per second.

5. A pressure swing gas separator including a bed of adsorbent material (16,108, 147), a tuned diaphragm (109, 133, 141) adjacent but spaced from one face of the bed of adsorbent material (16, 108, 147), driving means (1, 101, 150) arranged to drive the diaphragm (109, 133, 141) at its resonant frequency towards and away from the bed of adsorbent material (16, 108, 147), valve means (110, 115, 131) to introduce a gas mixture to and exhaust a depleted gas mixture from, a space between the diaphragm (109, 133, 141) and the bed (16, 108, 147), and a gas outlet (112, 113, 149) in communication with the other face of the adsorbent bed (16, 108, 147), in use, the movement of the diaphragm towards and away from the adsorbent bed pressurising and depressurising it.

6. A pressure swing gas separator according to claim 3, 4 or 5, in which the drive means operating at the resonant frequency of the system is formed by a pulsed combustor (2, 102) located in the middle of a substantially spherical resonance chamber (101) or at the end of an ogival resonance chamber (1).

7. A pressure swing gas separator according to claim 5, in which the drive means is formed by an electrical actuator (150) comprising an armature coil and a stator one of which is connected to the diaphragm (141) and the other of which is held by a fixed surrounding framework (146), in use, an oscillating current applied to the armature causing the armature, and hence the diaphragm (141), to oscillate with respect to the stator.

8. A pressure swing gas separator according to any one of claims 5, 6 or 7, in which the valve means to introduce gas mixture into, and exhaust gas mixture from, the space between the diaphragm (103, 133, 141) and the adsorbent bed (108, 147) comprises an annular valve seat (131, 148) which engages the diaphragm (103, 133, 141) and the diaphragm (103, 133, 141) forms the valve closure member which forms a gas-tight



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seal against the annular valve seat (131, 148) but which, when the diaphragm moves away from the adsorbent bed unseats to allow gas to pass between the seat and the diaphragm (103, 133, 141).

9. A pressure swing gas separator according to calim 8, in which the valve formed by the annular valve seat and the diaphragm acts as both an inlet and an exhaust valve, and in which a harmonic vibration is applied to the diaphragm so that, in addition to its fundamental oscillation towards and away from the adsorbent bed there is also a superimposed secondary vibration which appears as a wave moving from one side to the other of the diaphragm.

10. A pressure swing gas separator according to any one of the preceding claims, in which the particle size of the adsorbent bed (16, 108, 147) is substantially a quarter of a millimetre or less.



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Neu eingereicht / Newly filed
Nouvellement déposé

Fig.1.

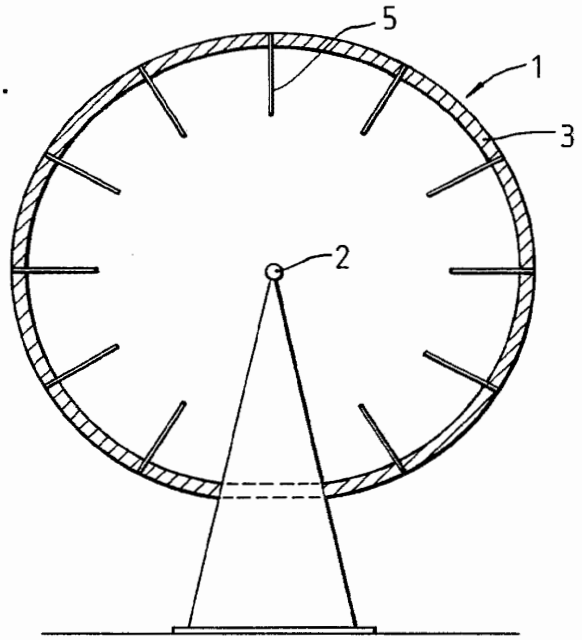
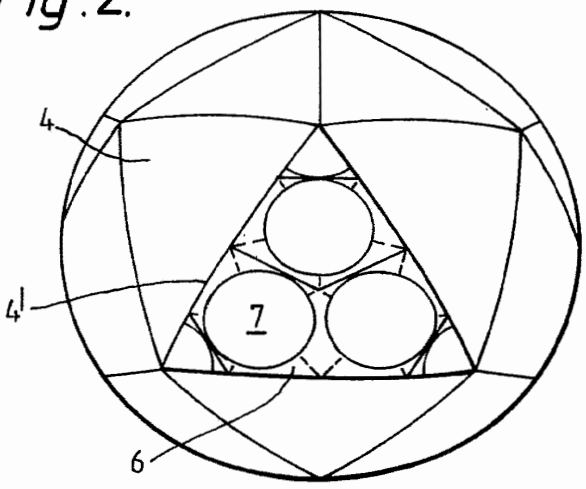



Fig.2.



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Fig.3.

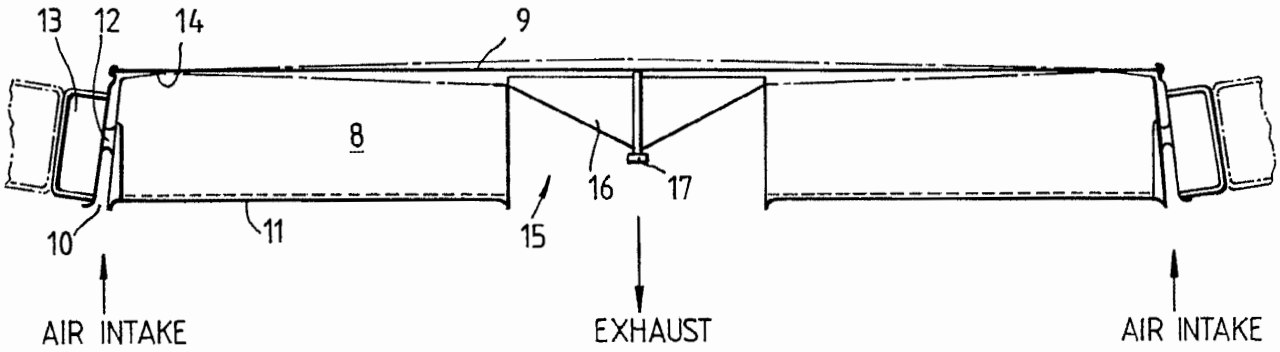
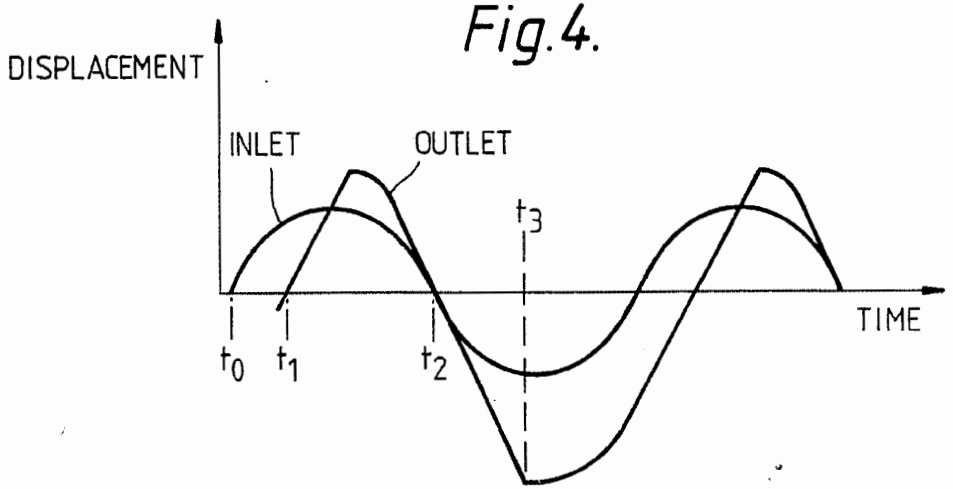



Fig.4.



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Fig.5.

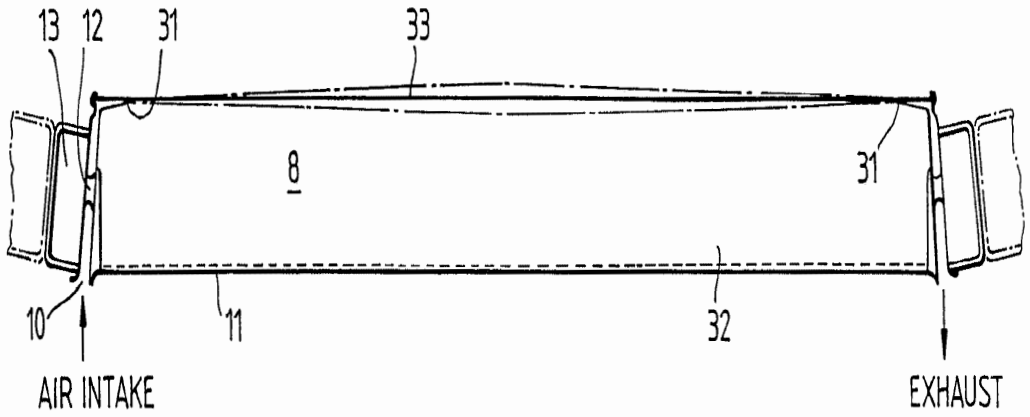
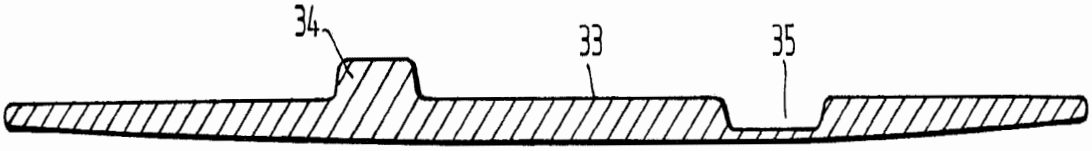
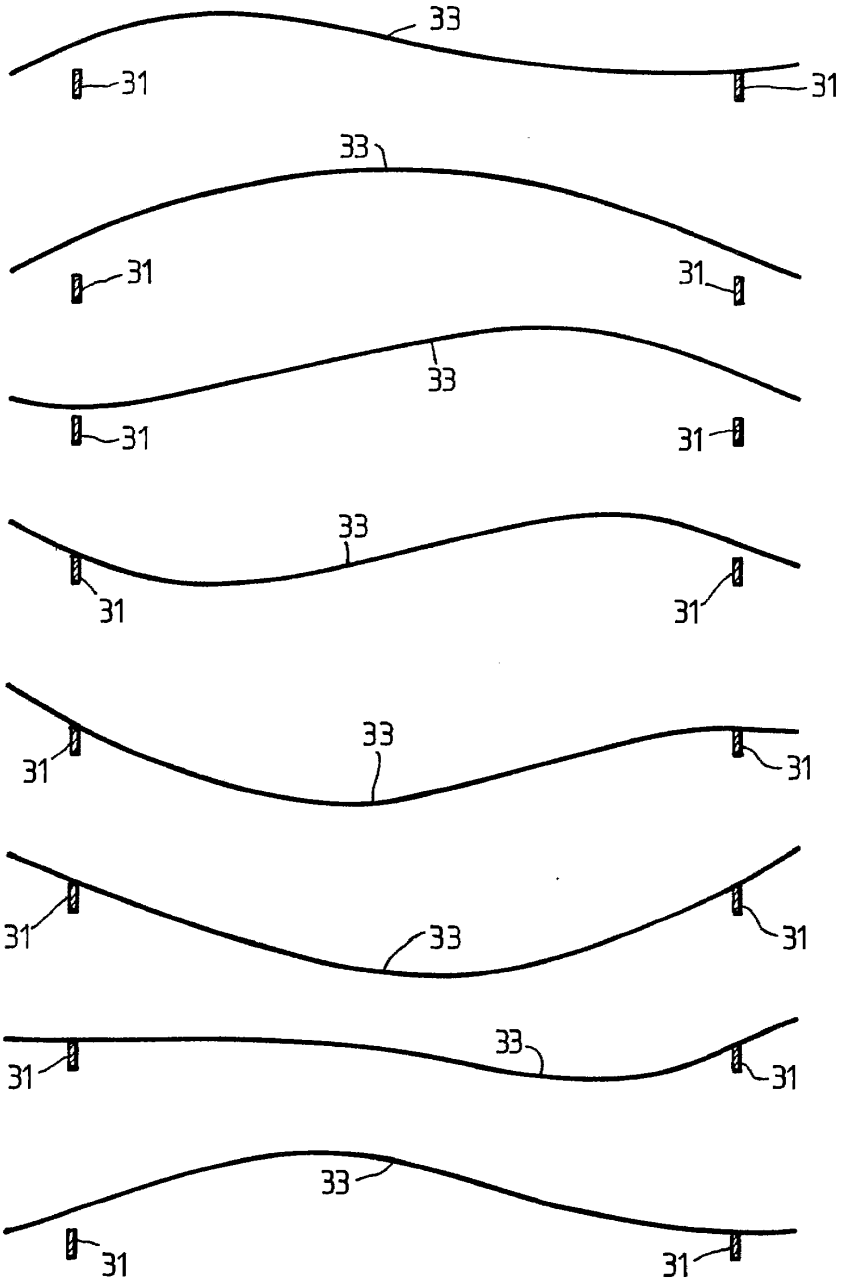



Fig.6.



Nouvelle page
Nouvelle page

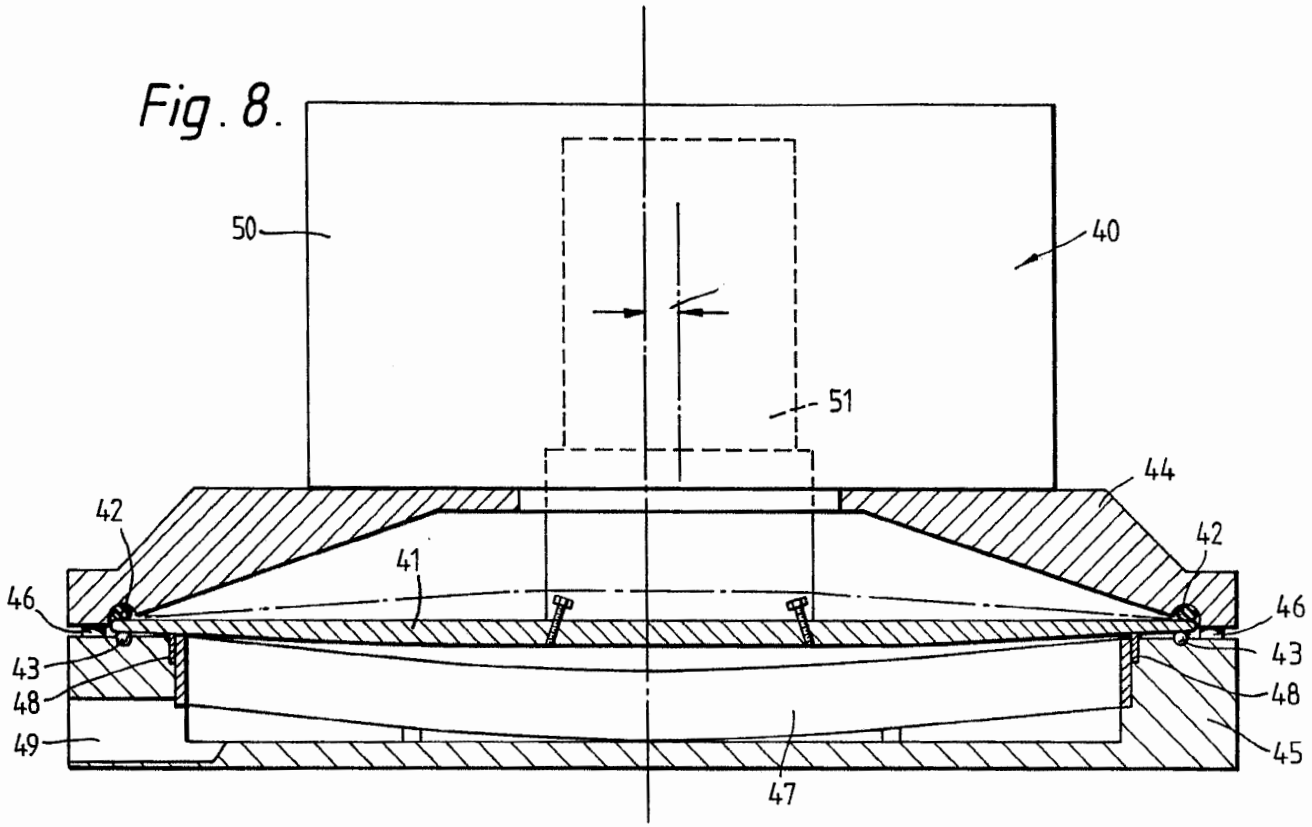
Fig. 7.



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Fig. 8.



European Patent Office

# EUROPEAN SEARCH REPORT

Application Number

EP 89 31 1584

## 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 ) |
|---|---|---|---|
| A,D | EP-A-8619 (UNION CARBIDE CORP.)<br>* abstract *<br>--- | 1, 2 | B01D53/04<br>F02G3/00<br>F25B9/00 |
| A,D | EP-A-13680 (UNION CARBIDE CORP.)<br>* abstract; claims 1-11 *<br>--- | 1, 2 | F23C11/04 |
| A | US-A-4767314 (MUTCHLER)<br>* abstract *<br>* column 2, lines 52 - 64; figure 1 *<br>--- | 5, 6 | |
| A,D | EP-A-267727 (WELLS)<br>* abstract *<br>* page 5, line 12 - page 6, line 7; figure 1 *<br>--- | 1-6, 8, 10 | |
| A | US-A-4732580 (JAIN)<br>--- | | |
| A | FR-A-1382688 (GESELLSCHAFT FUR LINDE'S EISMACHINEN)<br>----- | | |

| TECHNICAL FIELDS SEARCHED (Int. Cl.5 ) |
|---|
| B01D<br>F02G<br>F25B<br>F23C |

The present search report has been drawn up for all claims

| Place of search | Date of completion of the search | Examiner |
|---|---|---|
| THE HAGUE | 19 DECEMBER 1989 | ERNST J.L. |

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EPO FORM 1503 03.82 (P0401)

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
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D : document cited in the application
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## (54) Oxygen concentrator monitoring system

(57) A molecular sieve oxygen concentrator is provided with sensors for monitoring the oxygen flow and concentration and the pressure drop across the molecular sieve beds. The sieve beds are switched between gas separation and gas purge cycles based on gas pressure and the cycle times are measured. Information on the settings, the maintenance history and the operation of the concentrator is stored in a digital memory. The stored information may be accessed and updated by a service person through a portable hand held unit (9). The information may be transferred to a memory in the hand held unit and later transferred to the dealer's computer or, optionally, the information may be transferred via a modem and the telephone system directly to the dealer's computer (46). More accurate information is provided for timely servicing oxygen concentrators.

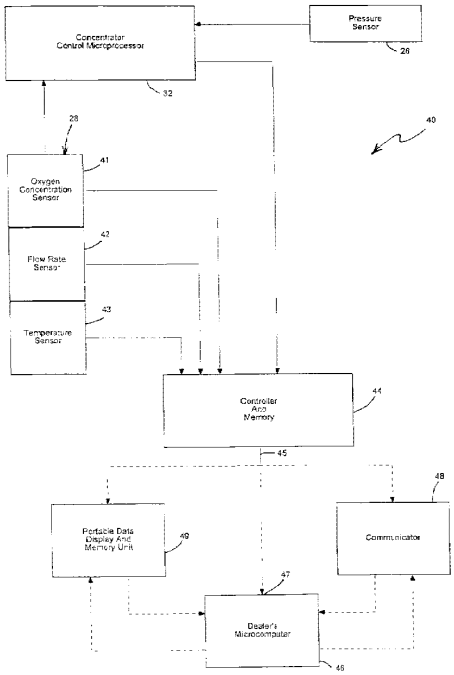



Fig. 2

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## Description

### Technical Field

The invention relates to oxygen concentrators and more particularly to a system for monitoring the operation of a pressure swing molecular sieve medical oxygen concentrator.

### Background Art

Oxygen concentrators are used, for example, as a source of high purity oxygen for medical applications. An oxygen concentrator will separate nitrogen from air to produce a gas stream which consists primarily of oxygen. A pressure swing oxygen concentrator typically has at least two molecular sieve beds. Each molecular sieve bed consists of a closed cylindrical container partially filled with a sieve material, such as a zeolite, which will pass a flow of oxygen molecules while blocking or adsorbing the larger nitrogen molecules. In operation, a compressor applies filtered pressurized air through a flow control valve to an inlet port on one of the molecular sieve beds and about 95% pure oxygen flows under pressure from an outlet port on such bed. The oxygen may flow to an accumulator and then pass through a pressure regulator, an optional flow meter and a final filter to the patient.

As oxygen flows through the sieve bed, the separated nitrogen is retained in the sieve bed. After a predetermined short operating time, one or more valves are changed to apply the pressurized air to the inlet port of a second sieve bed and to vent the inlet port of the first sieve bed. A small portion of the pressurized oxygen output from the second sieve bed is delivered to the outlet port of the first sieve bed. This oxygen flows in a reverse direction through the first sieve bed to purge nitrogen and any other trapped gases from the first sieve bed. The valves are periodically reversed to alternate the sieve beds between the gas separating cycle and the trapped gas purging cycle. Preferably, an accumulator holds a volume of the concentrated oxygen under pressure to provide a continuous oxygen flow to the patient as the valves are cycled.

The molecular sieve beds may be operated on a fixed cycle time. After a particular molecular sieve bed operates to produce concentrated oxygen for a fixed time, the valves are changed to switch gas separation to a different molecular sieve bed and to purge the previously used bed. Or, preferably, a system controller monitors the pressure drop across the molecular sieve bed which is in use. As nitrogen is accumulated in the molecular sieve bed, the pressure drop will increase and the concentrated oxygen output will decrease. When a predetermined pressure drop is reached, the valve is switched. Over a long period of operation, the molecular sieve beds will lose their efficiency. If the valve is switched on a timed basis, the oxygen output from the oxygen concentrator may decrease over time. Also, the valve may be operated more often then necessary, thus shortening the operating life of the oxygen concentrator. If the valve is switched in response to the pressure drop, the valve initially will be switched less frequently. Further, valve operation will be a function of the quantity of concentrated oxygen being produced. As the molecular sieve beds lose efficiency over time, the molecular sieve beds will be cycled more frequently, but the oxygen concentrator will continue to have its rated output.

Over a period of use, an oxygen concentrator requires regular maintenance. For example, air filters periodically need to be cleaned or replaced and eventually the molecular sieve beds may need replacing. As filters and/or the molecular sieve beds become clogged, the quantity and quality of the concentrated oxygen output may deteriorate. Since medical oxygen may be life supporting, it is important that the oxygen concentrator be timely serviced. In the past, there has been no satisfactory way for the service technician to determine the actual usage time of an oxygen concentrator or when service is needed. Consequently, a conservative maintenance schedule is followed. Maintenance is scheduled more frequently then may be required in order to assure that the oxygen concentrator continues to operate at peak performance. It would be desirable for the service technician to have a way of quickly determining the number of hours that an oxygen concentrator has been operated, to determine when a condition requiring servicing has occurred and to have a way to keep uniform records for each serviced oxygen concentrator. Also, it may be desirable for the physician who has prescribed oxygen therapy to easily determine patient compliance with the prescribed therapy.

Unnecessary maintenance increases the operating cost for an oxygen concentrator. With increased emphasis by health care providers for controlling health care costs, it is desirable to provide only necessary maintenance, but to provide such maintenance in a timely manner.

### Disclosure Of Invention

The invention is directed to a method and apparatus for monitoring the operation of a medical oxygen concentrator to determine when maintenance is needed, to indicate the nature of any needed maintenance and to record various data relating to the operation of an oxygen concentrator. The oxygen concentrator includes at least two molecular sieve beds for separating oxygen from air. The sieve beds are alternately operated in an oxygen separation mode and in a



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nitrogen purge mode. At least one valve, preferably a two position four way valve, is used to cycle the sieve beds between the gas separation and purge cycles. The oxygen concentration and flow rate from the molecular sieve beds are monitored by a sensor and stored in a digital memory. A microprocessor controls operation of the concentrator by operating the valve in response to the sensed pressure drop across the molecular sieve bed in the oxygen separation cycle. The pressure drop is compared with a high pressure alarm level, a valve switch level and a low pressure alarm level. In the event of an excessively high pressure, an alarm signal is stored in the digital memory, an alarm may be sounded and the concentrator is switched off. Only an alarm signal is stored for a low pressure. The computer also stores in the memory the valve cycle times which indicate the gas separation time for each sieve bed. If the cycle time is outside a predetermined range, an alarm message also is stored and the concentrator is turned off. Information on the total operating time and the oxygen output pressure and/or flow rate also may be collected and stored in the memory. When the concentrator is initially set up for operation, it also may be programmed with the serial number of the concentrator, the identity of the patient, the prescribed oxygen therapy and any other desired information. The memory in the concentrator also may serve as a notebook for storing information on the concentrator setup and past maintenance.

The operational data stored in the system memory may be accessed in any of several ways. The service person may have a digital hand held unit which includes a keyboard, a memory and a display. The hand held unit is connected to a serial input/output data port on the oxygen concentrator. The hand held unit allows the service person to access and display information on the past service history of the oxygen concentrator, on usage of the oxygen concentrator and on its operation including the cause of any alarms. As service calls are made, the data from each oxygen concentrator can be stored in the hand held unit for later transfer to the dealer's computer.

The input/output port also may be connected through a modem to a telephone line for communication with a dealer's computer. The oxygen concentrator may be programmed to automatically dial the dealer's computer at specific times and to download data. When the oxygen concentrator is at the dealer's shop either prior to delivery and installation at the patient's home or for servicing, the dealer's computer may be conn3ected directly to the input/output port on the oxygen concentrator. The computer may be used to download operational data from the oxygen concentrator or to program the oxygen concentrator.

The monitoring system for an oxygen concentrator provides several advantages which reduce the operating costs for the concentrator. Improved service can be provided to the patient at a lower cost to the provider. The time for performing preventative maintenance checks is reduced, allowing the service technical to service a greater number of concentrators. Data is made available for performance tracking which will help to schedule maintenance when the unit requires service verses the prior art system of scheduling maintenance at preset time intervals. Data also is available for preparing reports for the dealer and the physician.

Accordingly, it is an object of the invention to provide a monitoring system for a molecular sieve oxygen concentrator.

Other objects and advantages of the invention will become apparent from the following detailed description of the invention and the accompanying drawings.

## Brief Description Of The Drawings

Fig. 1 is a schematic diagram of an exemplary oxygen concentrator suitable for use with the monitoring system of the invention;

Fig. 2 is a block diagram of a monitoring system for an oxygen concentrator according to the invention;

Fig. 3 is a program flow chart illustrating operation of the oxygen concentrator for switching the molecular sieve bed selection valve and for generating messages relating to the operation of the molecular sieve beds;

Figs. 4a and 4b are a program flow chart illustrating operation of the oxygen concentrator program cycle for storing and transferring data;

Fig. 5 is a plan view of a hand held portable data display and memory unit for displaying service information regarding an oxygen concentrator and for transferring data to a dealer's microcomputer; and

Figs. 6a - 6e are a program flow chart illustrating operation of the portable data display and memory unit.

## Best Mode For Carrying Out The Invention

Referring to Fig. 1 of the drawings, an exemplary pressure swing molecular sieve oxygen concentrator 10 is illustrated. The oxygen concentrator 10 is of the type used to produce concentrated oxygen for medical applications, although the concentrated oxygen also may be used for other applications. The oxygen concentrator 10 includes two molecular sieve beds 11 and 12 which are partially filled with a material which will adsorb nitrogen while permitting a flow of oxygen between inlet ports 13 and 14 and outlet ports 15 and 16 on the beds 11 and 12, respectively. Various nitrogen adsorbent materials are known. Frequently, zeolite is used as the adsorbent material.

A compressor 17 driven by a motor 18 draws room air from an inlet 19 through a primary filter 20, a secondary filter 21 and a muffler 22. The air is discharged from the compressor 17 through a high efficiency filter 23 to a two



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position four way valve 24. The valve 24 is illustrated in its "on" or actuated position. When turned "off", the valve 24 moves to the left, as illustrated by the arrow 24'. In the illustrated position, the valve 24 applies the filtered compressed air to the inlet port 13 on the sieve bed 11. Concentrated oxygen flows from the outlet port 15, while nitrogen is retained in the bed 11. The concentrated oxygen flows through a check valve 25 and a pressure sensor 26 to an accumulator 27. The pressure sensor 26 may be adapted to measure both the concentrated oxygen pressure at the accumulator 27 and the pressure drop across the operating one of the molecular sieve beds 11 or 12. From the accumulator 27, the oxygen flows through a sensor 28 which measures the oxygen concentration, the oxygen flow rate and the oxygen temperature, through a final filter 29 and a check valve 30 to an oxygen outlet 31. The outlet 31 is connected, for example, through a suitable hose and mask or cannula (not shown) to supply concentrated oxygen to the respiratory track of a patient.

A microprocessor or computer 32 controls the operation of the oxygen concentrator 10. The microprocessor 32 is connected to control the motor 18 and the valve 24 in response to information from the pressure sensor 26 and the sensors 28. If desired, the speed of the motor 18 may be adjusted to provide a desired gas pressure and/or flow rate or a flow or pressure regulator (not shown) may be provided. As nitrogen is separated from oxygen in the sieve bed 11, the pressure drop across the sieve bed 11 increases and hence the pressure of the concentrated oxygen delivered to the accumulator 27 will decrease. When the microprocessor 32 senses a predetermined drop in the oxygen pressure or a predetermined pressure increase across the sieve bed 11 or 12, the valve 24 is operated to apply the compressed air to the inlet port 14 to the sieve bed 12. Concentrated oxygen then flows from the outlet port 16 through a check valve 33, through the pressure sensor 26 and to the accumulator 27. The accumulator 27 functions to maintain a more uniform flow pressure while the valve 24 is operated and the next sieve bed 11 or 12 comes up to pressure.

While the sieve bed 11 is operated to separate nitrogen from oxygen, the other sieve bed 12 is purged of retained nitrogen. The valve 24 connects the inlet port to the sieve bed 12 to a suction pump 34. The suction pump 34 also may be driven by the motor 18 which drives the compressor 17. The suction pump 34 reduces the pressure at the inlet port 14. A calibrated orifice 35 connects the sieve bed outlet ports 15 and 16 to permit a limited flow of concentrated oxygen from the pressurized bed 15 or 16 to the outlet port 16 or 15 of the bed being purged. The pressurized oxygen at the outlet port 16 and the reduced pressure at the inlet port 14 causes a reverse flow through the sieve bed 12 and any trapped nitrogen is purged or desorbed by the oxygen flow. The oxygen and desorbed nitrogen are discharged through a muffler 36 and an exhaust port 37 to atmosphere. Optionally, the suction pump 34 and the muffler 36 may be omitted and the valve 24 may connect the inlet port 13 or 14 of the bed 11 or 12 being purged either directly to the muffler 36 or directly to the exhaust port 37.

Fig. 2 is a block diagram illustrating a monitoring system 40 according to a preferred embodiment of the invention for an oxygen concentrator such as the concentrator 10 of Fig. 1. The pressure sensor 26 and the sensors 28 are shown connected to supply operational data to the concentrator control microprocessor 32. In response to this data, the microprocessor 32 operates the motor 18 and the cycling valve 24 (Fig. 1) and also measures the valve cycle time. The sensors 28 are illustrated as a single unit which includes an oxygen concentration sensor 41, an oxygen flow sensor 42 and an oxygen temperature sensor 43 which are connected to a controller and memory 44 in addition to the microprocessor 32. Preferably, the sensors 28 are combined into a single device, such as the sensor described and illustrated in United States patent 5,247,826, the disclosure of which is incorporated herein. The sensor 28 may include transducers and a microprocessor which calculates gas concentration, gas flow rate and gas temperature from information generated by the transducers. The microprocessor may be included in the controller and memory 44.

As described above, the operation of the concentrator 10 is controlled by the microprocessor 32. The microprocessor 32 controls the compressor motor 18, the valve 24 and generates messages and alarms. It continuously monitors the pressure in the accumulator tank 27 as well as the cycle times. Based upon parameters stored in non-volatile memory in the controller and memory 44, the microprocessor 32 will alarm if a system malfunction is determined by 1) the pressure being either too low or too high, or 2) the gas separation cycle time for each sieve bed 11 and 12 being either too long or too short, or 3) oxygen concentration falling below a stored set point, or 4) a microprocessor timing error or 5) a non-volatile memory malfunction. The microprocessor 32 provides a signal representing the valve states to the controller and memory 44 and digital codes representing the status of the oxygen concentrator 10.

The controller and memory 44 is the overseer of the oxygen concentrator 10. It continuously monitors the communication from the microprocessor 32 for alarm messages and checks the valve switching times. At a rate of twice per second, the controller and memory 44 measures the oxygen concentration coming out of the concentrator 10 as well as the temperature and the flow rate of the concentrated oxygen. This information is sent twice per second to a communications port 45 in a standard serial data format. At the conclusion of the data stream, the controller and memory 44 monitors the communications port 45 for commands from an external device. Oxygen concentrator operational data stored in the controller and memory 44 also may include the serial number of the oxygen concentrator 10, hours of operation, patient identification and prescription details, and service information such as a record of past services performed and a schedule of when future service will be needed.

One or more of three different communications methods and devices may be used for communicating with the



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oxygen concentrator 10. A dealer who sells, leases or services the oxygen concentrator 10 may have a microcomputer 46 having a port 47 which may be connected directly to the communications port 45 when initially setting up the oxygen concentrator 10 prior to delivery to the patient and when the concentrator 10 is in the shop for servicing. The dealer's microcomputer 46 may be used to keep a master record on each oxygen concentrator sold, leased or serviced and to update the patient and service data stored in the controller and memory 44.

As an alternate method for transferring data between the dealer's microcomputer 46 and the oxygen concentrator 10, the oxygen concentrator 10 may include a communicator 48 which connects the communications port 45 to a telephone line. The communicator 48 includes a microprocessor, a non-volatile memory, a real time clock for time and date stamping the stored data, a modem and a battery. The dealer's microcomputer 46 also is provided with a modem (not shown) connected to a telephone line to permit communications between the oxygen concentrator 10 and the microcomputer 46. If a communicator 48 is provided in the oxygen concentrator, it may be programmed to automatically connect to the dealer's computer 46 either at preprogrammed times to download maintenance and usage data or whenever one or more alarms are generated to send data identifying the oxygen concentrator and the nature of the alarm. The communicator 48 also may inform the dealer when routine service based on operating time is needed. The dealer then will know immediately when service is needed and will know the urgency of the needed service. The communicator 48 will reduce the service costs for the oxygen concentrator, since service will be performed only when needed and the technician will know the nature of the needed service prior to making a service call. In the past, it was necessary for the servicing technician to make regularly scheduled service calls based on an estimated usage of the oxygen concentrator. This often resulted in service calls before they were needed. If desired, each time the oxygen concentrator is turned on and off may be stored and periodically transmitted either to the dealer's computer or to the patient's physician's computer. From this information, the physician can determine patient compliance with the prescription and can confirm that the oxygen concentrator was set to administer the prescribed oxygen flow. If the patient is not responding properly to the prescribed therapy, the physician will know either that the prescription needs revising or that the patient is not complying with the prescription.

Alternately, a portable, hand held data display and memory unit 49 may be connected to the communications port 45 by a service technician during a service call. The hand held unit 49 may download data from a number of oxygen concentrators as the service technician makes service calls during a day and subsequently the data is downloaded from the hand held unit 49 into the dealer's microcomputer 46 at the end of the day to update the dealer's records. When data is downloaded into the dealer's computer, the data may be stored in file formats that are compatible with popular spread sheet programs, word processor programs and/or data base programs. These programs can be used for generating reports, trend analysis, or patient/unit records. Further, the hand held unit 49 may be used by the technician to display data such as alarm messages, the service record and other information relating to the oxygen concentrator 10. For example, the service technician may be informed that a particular filter needs to be replaced. Once the filter is replaced, the service technician may update the data stored in the controller and memory 44, using a keyboard on the hand held unit 49, to show that the filter has been replaced. The keyboard also may be used to modify the information stored in the oxygen concentrator, such as the service record, or the patient prescription.

Referring now to Fig. 3, a flow chart 52 illustrates operation of the microprocessor 32 in the oxygen concentrator 10 of Fig. 1 for switching the molecular sieve bed selection valve 24 and for generating messages relating to the operation of the molecular sieve beds. Initially, the flow chart 52 is entered at a power up terminal 53. A selftest cycle is automatically run, the compressor motor 18 is started, the valve 24 is turned on and a valve cycle timer is started at a block 54. The valve cycle timer is a clock in the microprocessor 32 which measures the elapse time between changing the position of the valve 24. Hence, the cycle timer measures the gas separation operating cycle time for each of the molecular sieve beds 11 and 12. A "normal operation" message then is generated at a block 55. The pressure drop across the operating molecular sieve bed 11 or 12, as measured by the sensor 26, is then read at a block 56. Pressure data is supplied from the block 56 to four blocks 57-60. The controller and memory 44 (Fig. 2) stores several pressures which are used to control operation of the oxygen concentrator 10. A minimum acceptable oxygen pressure is stored, a shift pressure threshold for the valve 24 is stored and a maximum acceptable oxygen pressure is stored. The block 57 establishes an output if the measured oxygen pressure is less than the stored minimum pressure. The block 58 establishes an output if the measured oxygen pressure is less than the shift pressure but greater than the minimum pressure. The block 59 establishes an output if the measured oxygen pressure is greater than the stored maximum pressure. The block 60 establishes an output if the measured oxygen pressure is greater than the stored shift pressure but less than the stored maximum pressure.

During normal operation of the oxygen concentrator 10 with the valve 24 on, the measured pressure will be less than the shift pressure but greater than the minimum pressure until it is time to regenerate the sieve bed 11. The block 58 will apply an output to a decision block 61 which asks if the valve cycle timer has exceeded a stored maximum cycle time. If not, the microprocessor returns to the block 32 and again measures the pressure at the block 56. Once the pressure exceeds the shift pressure while remaining below the stored maximum pressure, the block 60 applies an output to a decision block 62 which asks if the timer is less than the minimum cycle time. If the cycle timer is not less



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than the minimum, a block 63 shifts the valve 24, restarts the cycle timer and returns to the block 56 for again measuring the pressure. If the cycle timer was less than the minimum cycle time at the block 62, a decision block 64 asks if the valve 24 is on or off. If the valve 24 is on, a "minimum cycle time bed 1" message is generated at a block 65 and if the valve 24 is off, a "minimum cycle time bed 2 " message is generated at a block 76. In either case, an alarm is turned on at a block 66, the compressor motor 18 and the valve 24 are turned off at a block 67 and the program quits at a terminal 68.

As indicated above, if the concentrated oxygen pressure is determined to be less than the shift pressure but greater than the minimum pressure, the block 61 asks if the valve cycle timer has exceeded its maximum time. If the maximum cycle time has been exceeded, a block 69 asks if the valve 24 is on. If the valve 24 is on, the block 70 sends a "maximum cycle time bed 1" message, an alarm is turned on at the block 66, the compressor and valve are turned off at the block 67 and the program quits at the terminal 68. If the valve 24 was determined to be off at the decision block 69, a block 71 sends a "maximum cycle time bed 2" message, an alarm is turned on at the block 66, the compressor motor 18 and the valve 24 are turned off at the block 67 and the program quits at the terminal 68.

If the measured oxygen pressure was determined at the block 57 to be less than the stored minimum pressure, a block 72 sends a "minimum pressure" message and an alarm. However, the oxygen concentrator 10 is not shut off in response to only a minimum pressure message, since the measured pressure will be below the minimum whenever the oxygen concentrator 10 is initially turned on and may briefly drop below the minimum immediately after the valve 24 is shifted. From the block 72, the program advances to the decision block 61 which compares the cycle timer with the stored maximum cycle time. From the block 62, the program will either return to the pressure measurement block 56 or will proceed to establish a "maximum cycle time" message at one of the blocks 70 or 71.

If the measured pressure is determined to exceed the stored maximum pressure at the block 59, a decision block 73 asks if the valve 24 is on. If so, a block 74 sends a "maximum pressure bed 1" message. If the valve 24 is off, a block 75 sends a "maximum pressure bed 2" message. After a message is sent at either block 74 or block 75, an alarm is turned on at the block 66, the compressor motor 18 and the valve 24 are turned off at the block 67 and the program quits at the terminal 68.

The various messages generated by the program illustrated in the flow chart 52 are stored in the controller and memory 44 of Fig. 2. If a communicator 48 is provided, the communicator 48 may be programmed to dial the telephone number for the dealer's microcomputer 46 and to transmit both data identifying the patient and oxygen concentrator which requires service and the message established by the microprocessor 32. If a portable data display and memory unit 49 is used by a service technician, the messages may be displayed and/or the messages and other data relating to the oxygen concentrator 10 may be downloaded into the memory for transfer at a later time to the dealer's microcomputer 46.

Figs. 4a and 4b are a program flow chart 80 which illustrate the process for storing and transferring data. On power up, the cycle is began at a terminal 81. At a block 82, the microprocessor in the controller and memory 44 is initialized and self tests are run. The process then goes to a half second loop timer 83 which includes a timer for automatically recycling through the flow chart 80 every one half second. From the loop timer 83, the oxygen concentration percentage, the concentrated oxygen flow rate and the concentrated oxygen temperature are measured at a block 84. The operating status of the concentrator then is read at a block 85 if any new message is present. A decision block 86 asks if the valve status has changed since the last program loop cycle. If not, the program jumps through a terminal 87 to "A" in Fig. 4b. If the valve status has changed, a decision block 88 asks if the valve 24 was "on". If the valve was "on", the "on" time is saved as "Cycle Time On" at a block 89, the valve timer is reset at a block 90 and the program jumps via the terminal 87 to "A" in Fig. 4b. If the block 88 determines that the valve was "off', a block 91 saves the "off" time as "Cycle Time Off", the valve timer is reset at the block 90 and the program jumps via the terminal 87 to "A" in Fig. 4b.

From the terminal 87 in Fig. 4b, the alarm set point for the oxygen concentration is read at block 92. A decision block 93 then asks if the concentration is less than the set point. If so, a low concentration alarm is turned of at a block 94 and the data is sent to the serial port (45 in Fig. 2) at a block 95. If the oxygen concentration was not less than the set point at the block 93, the low oxygen concentration alarm is turned off at a block 96 and the data is sent to the serial port at the block 95. After the data is sent to the serial port, a decision block 97 asks if there are any commands. If not, the program returns through a loop terminal 98 to the loop timer 83 in Fig. 4a. If there were commands at the decision block 97, a decision block 99 asks if the command was to write data to the non volatile memory. If so, the data is written at a block 100, verified at a block 101 and the program returns through the loop terminal 98 to the loop timer 83 in Fig. 4a. If decision block 99 determines that the command was not to write data, a decision block 102 asks if data was to be read from the non volatile memory. If not, the program returns to the cycle timer 83 through the loop terminal 98. If it was determined at the block 102 that data was to be read, the data is read at a block 103 and sent to the serial port at a block 104. The program then returns through the loop 98 to the cycle timer 83. Data will be read whenever requested by a device connected to the communications port 45, such as by the portable data display and memory unit 49.

Fig. 5 is a plan view showing a portable data display and memory unit 49. Preferably, the unit 49 is of a sufficiently



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small size to be easily held in the hand of the service technician during use. The unit 49 includes an internal non volatile memory (not shown) to which data may be written and read. The memory may be, for example, an EEPROM. The unit 49 has a housing 108 from which a serial data cord 109 extends. The cord 109 terminates at a connector 110 which is adapted to connect to the serial communications port 45 on the oxygen concentrator monitoring system 40 of Fig. 2. A digital data display 111 is located on the housing 108 along with a keyboard 112 for entering data and for requesting the display of data. Many of the keys on the keyboard 112 may have multiple functions. For example, a key 113 is labeled "ABC 1 ID" When the unit 49 is first turned on with a key 114, the operator is asked to enter his or her initials to record the service technician's identification. If the first initial of the technician is an "A", the key 113 is pushed once. If the first initial is a "B", the key 113 is pushed twice, or if it is a "C" it is pushed three times. After the first initial is displayed, left and right arrow keys 126 and 127 may be pushed to move the cursor for entering and/or changing the remaining initials. An "Enter" key 116 then is pushed to save the initial and the remaining two initials are similarly entered.

Once the initials are entered, the display 111 indicates "Task Completed". If a "Save" key 115 is pushed, the operator is asked to enter the accumulated hours of operation of the oxygen concentrator as indicated on an hour meter. If the "Enter" key 116 is then pushed, all data relating to operation of the oxygen concentrator, including the entered operating hours, and the date and time are stored in a non volatile memory in the controller and memory 44. The key 113 may be used to display the patient identification and, if necessary, the patient identification may then be changed. A key 117 may be used to select "Rx" for displaying and modifying the patient prescription. Both a rest flow rate and, if desires, an activity flow rate may be entered. A key 118 may be used to display and reprogram the serial number of the oxygen concentrator 10. The serial number will be changed only when the controller and memory 44 has been replaced. A key 119 is pushed to display the oxygen concentration, a key 120 is pushed to display the oxygen flow rate in liters per minute and a key 121 is pushed to display the oxygen temperature. A key 122 displays the cycle time for the molecular sieve bed 11 I and a key 123 displays the cycle time for the molecular sieve bed 12. A key 124 displays the status of the oxygen concentrator 10 and causes any alarm messages to be displayed. A key 125 displays a record for the oxygen concentrator including a maintenance check list. The technician may move down through the check list and enter a record of any maintenance performed and times when maintenance will be required. The oxygen concentrator record will be stored in the internal memory in the unit 49. An exemplary record for an oxygen concentrator is shown in Table I. Preferably, the memory in the portable unit 49 is sufficiently large to store records for all oxygen concentrators serviced by a technician during at least one day and may cover a longer period such as one week. Periodically, the information stored in the unit 49 is downloaded into the dealer's computer 46. This information is used for maintaining a service record on each oxygen concentrator and for scheduling future maintenance. By having an accurate record on each oxygen concentrator, the dealer can efficiently provide service only when needed, thus reducing the cost for supplying oxygen to the patient.

TABLE I

| Concentrator Data: | |
|---|---|
| Serial No. | M12345DS |
| Hours of Operation: | 1534 |
| Concentration | 94.35 (%) |
| Flow Rate: | 4.1 (LPM) |
| Temperature: | 78.3 (°F) |
| Cycle Time 1: | 10.9 (sec) |
| Cycle Time 2: | 10.8 (sec) |
| Error Message: | Unit Running OK |
| Audible Alarm | OK |
| Visual Alarm | OK |
| Check LPM | 3.9 |
| O2 Analyze | 94.2 (%) |
| Check LPM | 5.0 |
| O2 Minimum | 93.8 (%) |
| Exterior Filter | Replaced |
| Long Life Intake Filter | Yes |
| Intake Filter | OK |
| Check Battery | OK |
| Cabinet Clean | Cleaned |
| Cannula/Mask | Provided 2 |



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TABLE I (continued)

| | |
|---|---|
| Concentrator Data: | |
| O2 Tubing | Replaced 1 |
| Humidifier | Removed 1 |
| O2 Port Fitting | OK |
| O2 Tank | Provided |
| Total Tank Qty | 3 |
| O2 Tank Size | D |
| O2 Conserver | Yes |
| O2 Cart Bag | No |
| No Smoke Sign | Provided 2 |
| Provider Info | Provided 1 |
| Instruction Guide | OK |
| Preposition Unit | No |
| Unit Repaired | No |
| Unit Replaced | No |
| Patient Usage: | |
| Rx LPM Resting | 4.0 |
| Rx Hours/Day | 10 |
| Rx Humidification | No |
| Usage Check | 358 hours/34 days = 10.5 hours/day |

Figs. 6a to 6e form a flow chart 130 illustrating the preferred operation of the portable data display and memory unit 49. Referring first to Fig. 6a, the unit 49 is first connected either to the serial port 45 of the oxygen concentrator 10 or to a serial port on the dealer's microcomputer 46. The flow chart 130 is initiated at a START UP terminal 131, as will be discussed in greater detail below. The display 111 (Fig. 5) is turned on at a block 132. The unit has a number of preset functions. Exemplary functions are listed below in Table II. After the display is initially turned on, the function is set to 1 at a block 133 and the display 111 is set to indicate "Checking System" while the operation of the unit 49 is automatically checked. The battery is then checked at a block 134 and a "good battery? query is asked at a decision block 135. If the battery if not good, the function is set to 2 and the display 111 is set to indicate "Low Battery" at a block 136. If the battery is determined to be good, the function is set to 3 and the display 111 is set to indicate "Enter Initials" at a block 137. In either case, the program proceeds to a MAIN terminal 138 which connects to a block 139 where interrupts are cleared and the keyboard 112 is initialized. The program then goes to a SLEEP terminal 140 and waits for the next command either from the keyboard or through the serial cable 109 from an oxygen concentrator or a microcomputer. The portion of the program in Fig. 6a also has an input terminal 141 called SHUT DOWN. Whenever a SHUT DOWN command is received, the display and memory are turned off and the power is reduced at a block 142. The program then proceeds through the block 139 to the SLEEP terminal 140. If the program goes to the SLEEP terminal 140 via the MAIN terminal 138, the display 11 and the memory in the unit 49 will remain on, while if the SLEEP terminal 140 is reached via the SHUT DOWN terminal 141, they are turned off.

The unit 49 enters the SLEEP mode to conserve power. It will remain in the SLEEP mode until it receives an interrupt. If the memory and display unit 49 remains on, the pressing of any key on the keyboard 112 will generate an interrupt which will cause the program to enter a WAKE terminal 145 in Fig. 6b. If the SLEEP mode was entered via the SHUT DOWN terminal 141, the power ON key 114 must be pushed. A timer (not shown) also will periodically wake the unit 49 from the power on SLEEP mode. At a decision block 146, it is determined if the unit 49 woke because of a timer interrupt. If not, a decision block 147 asks if a key was pressed. If so, the program jumps to a KEY FUNCTION terminal 148 in Fig. 6c. If a key was not pressed, the program jumps to the main terminal 138 in Fig. 6a.

TABLE II

| Function | Description |
|---|---|
| 0 | Power Down Mode |
| 1 | Checking System |
| 2 | Low Battery Display |
| 3 | Input Operators Initials |



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TABLE II (continued)

| Function | Description |
|---|---|
| 4 | Task Completed/Next Function Mode |
| 5 | Display Unit Serial Number |
| 6 | Display Oxygen Concentration |
| 7 | Display Flow Rate |
| 8 | Display Temperature In °F. |
| 9 | Display Cycle Time For Bed 11 |
| 10 | Display Cycle Time For Bed 12 |
| 11 | Display Patient ID |
| 12 | Display Prescription Flow Rate |
| 13 | Display Prescription Hours/Day |
| 14 | Display Prescription Humidification |
| 15 | Display Concentrator Status Message |
| 16 | Input Hours Of Operation |
| 17 | Store Data |

If there was a timer interrupt at the decision block 146, the "seconds" counter is updated at a block 149 and the unit prepares to update the display function at a block 150. If a display function is present and it is, for example, Function 6, the program proceeds through a block 151 to a block 152 where the current oxygen concentration is read from a connected oxygen concentrator and is displayed. The program then jumps to the MAIN terminal 138 in Fig. 6a. If, for example, the display function 7 is present, the program proceeds from the block 150 through a block 153 to a block 154 where the flow rate data is read from a connected oxygen concentrator and is displayed. The program then jumps to the MAIN terminal 138 in Fig. 6a. Similar blocks (not shown) are provided for the other display functions. If the function is not a display function, the program passes through a block 155 and jumps to the MAIN terminal 138 in Fig. 6a.

If a key was pressed at the decision block 147, the program jumps to the KEY FUNCTION terminal 148 in Fig. 6c. The program then looks at the current function at a block 158. If the function is set to 0 for the power down sleep mode, the program passes through a block 159. The program will respond at a block 160 only to the "ON" key 114 and jump to the START UP terminal 131 in Fig. 6a. If the function was set to 1 for checking the system, the program proceeds from the block 158 through a block 161 to a block 162. The keys are ignored until data is received from a READ loop 163 in Fig. 6d. When data is received, the function is set to 3 and the program then jumps to the MAIN terminal 138 in Fig. 6a. Similar blocks (not shown) are provided for each of the remaining functions other than those which are marked on the keypad 112 (Fig. 5).

It will be noted in Fig. 5 that in addition to other markings, the key 113 is marked with the function "ID" (patient ID), the key 118 is marked with the function "Rx" (prescription), the key 118 is marked with the function "SN" (concentrator serial number), the key 119 is marked with the function "02" (oxygen concentration), the key 120 is marked with the function "LPM" (liters per minute oxygen flow rate), the key 121 is marked with "Temp" (oxygen temperature °F), the key 122 is marked with the function "CT1" (cycle time for sieve bed 11), the key 123 is marked with the function "CT2" (cycle time for the sieve bed 12), the key 124 is marked with the function "Status" (alarm messages), and the key 115 is marked with "SAVE". Referring again to Fig. 6c and to Table II, if the function was set to any of Functions 4, 6, 8, 10, 15 or 17, the program passes through blocks 164 and 165. If, for example, the "1" key or key 113 is pressed, the program passes through a block 166 to a block 167 where the function is set to 11 and the display is set to show "Patient ID. The program then jumps to the MAIN terminal 138. Or, if the "9" key or key 124 is pushed, the program passes through a block 168 and the function is set to 15 and the display is set to show "Unit Status XXXXX" at a block 169. The program then jumps to the MAIN terminal 138. Similar blocks (not shown) are provided for the other function keys on the keyboard 112. If the power ON/OFF key 114 was pushed, the program passes through a block 170 and jumps to the SHUT DOWN terminal 141 in Fig. 6a.

If data was called for from the READ loop 163, the program enters a READ terminal 173 in Fig. 6d. A decision block 174 then asks if the power off key 114 was pushed. If so, the program jumps to the SHUT DOWN terminal 141. If not, a decision block 175 asks if there has been any data received in the last 8 minutes. If not, the program again jumps to the SHUT DOWN terminal 141 to extend the battery life. If data has been received in the previous 8 minutes, a decision block 176 asks if it was received in the last 0.6 seconds. If not, a block 177 causes the display to indicate "Check Connection" and the program returns to the block 174. It should be noted that the oxygen concentrator program updates data and sends the data to the serial port 45 every 0.5 seconds. The dealer's microprocessor will have a similar or a faster program cycle time. If data was received within the previous 0.6 seconds, the program then asks at



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a decision block 178 if the data was from the dealer's microcomputer. If so, the program jumps to Fig. 6e via a PC terminal 179. If not, the data from the oxygen concentrator is stored is a random access memory at a block 180 and the program jumps to the MAIN terminal 138.

TABLE III

| Command | Description |
|---|---|
| M | Load Message Into Memory From PC |
| N | Read Message From Memory To PC |
| R | Read Byte From Memory To PC |
| W | Write Byte To Memory From PC |
| S | Set Time In Second Counter |
| T | Read Time To PC |
| P | Power Down |
| D | Down Load Data From Memory To PC |
| ? | Send Error Message To PC |

Fig. 6e shows a program loop 182 for communicating with the dealer's microcomputer 46. From the PC terminal 179, input command messages are read at a block 183 and any functions based on commands from the dealer's microcomputer 46 are performed at a block 184. Table III shows exemplary commands from the microcomputer 46. From the block 184, the program passes through a PC Command block 185 If, for example, the microcomputer 46 gives an "N" command to load a message, the program passes through a block 186 to a block 187 where the display message is loaded from the dealer's microcomputer 46 into an EEPROM memory in the unit 49. The transmission then is terminated with a carriage return at a block 188 and the program returns to the block 183. This loop is repeated for additional commands other than a power down command. Thus, if the next command is a "D" or down load command, the program passes from the block 185 through a block 189 to a block 190 where a block of data stored in the EEPROM memory in the unit 49 is down loaded to the microprocessor 46. Additional blocks (not shown) for the other commands are provided, as needed. If the command at the block 185 is a "P" or power down command, the program passes through a block 191 and jumps to the SHUT DOWN terminal 141 in Fig. 6a.

Having the capability to modify the operation of the oxygen concentrator provides several benefits not previously available. The capability of programming operating limits for the oxygen concentrator allows the manufacturer to use the same circuitry for different capacity oxygen concentrators. In the past, it was necessary to use different circuitry for controlling different capacity concentrators. Also, operation of the oxygen concentrator is easily modified for different markets. For example, the oxygen concentrator may be designed for operation from different voltage sources available in different countries. Changing the voltage level and frequency can affect the speed of the compressor motor which will affect the operating pressures. This in turn can change the cycle times for the molecular sieve beds. It is a simple matter to reprogram the alarm limits for the molecular sieve bed cycle times based upon the available power source. In the past, any alarm limits had to be set very broad to prevent false alarms caused by environmental changes. The cycle times could be affected, for example, by altitude and other factors. The alarm limits now can be set more closely to the actual normal operation of the oxygen concentrator for each installation.

It also should be appreciated that multiple alarm limits may be easily provided. A first alarm message may be generated when a first limit is exceeded to indicate that non urgent service should be scheduled. In this event, the oxygen concentrator is not shut down. A second alarm limit may generate an urgent message and shut down operation of the oxygen concentrator when broader limits are exceeded. The program operation for such multiple alarm limits can be easily understood by referring to Fig. 3. When the pressure was less than a minimum limit, an alarm message was established at block 72 and the oxygen concentrator continued to operate. A narrow limit range for the sieve bed cycle times and/or the maximum pressure may be set in a similar way to establish alarm messages without shutting down the concentrator.

It will be appreciated that various modifications and changes may be made to the above described preferred embodiment of a monitoring system for pressure swing oxygen concentrators without departing from the spirit and the scope of the following claims.

## Claims

1. A method for monitoring the operation of an oxygen concentrator comprising the steps of:



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a) storing in a memory in said oxygen concentrator data on the operation of said oxygen concentrator;
b) attaching a portable memory and display unit to said oxygen concentrator;
c) transferring oxygen concentrator operation data from said oxygen concentrator memory to a memory in said portable memory and display unit;
d) selectively displaying operation data from said oxygen concentrator memory on said attached portable memory and display unit; and
e) selectively transferring oxygen concentrator operation data from said portable memory and display unit memory to a computer located remote from said oxygen concentrator.

**2.** A method for monitoring the operation of an oxygen concentrator, as set forth in claim 1, and wherein said oxygen concentrator is monitored by measuring the gas separation cycle time for each of two molecular sieve beds which are alternately switched between a gas separation cycle and a gas purge cycle in response to measured pressure and comparing said monitored cycle times for each molecular sieve bed with stored minimum and maximum cycle times.

**3.** A method for monitoring the operation of an oxygen concentrator, as set forth in claim 2, and further including the step of establishing a cycle time alarm message when the measured time of a gas separation cycle is outside a predetermined time range.

**4.** A method for monitoring the operation of an oxygen concentrator, as set forth in claim 3, and further including the steps of storing in a memory a predetermined minimum gas separation cycle time, and wherein said cycle time alarm message is established in response to a comparison of the measured gas separation cycle time and the stored minimum gas separation cycle time.

**5.** A method for monitoring the operation of an oxygen concentrator, as set forth in claim 3, and further including the steps of storing in a memory a minimum gas pressure, a sieve bed cycle gas pressure and a maximum gas pressure, wherein switching of said sieve bed from a gas separation cycle to a gas purge cycle is in response to a comparison of the measured gas pressure and the stored sieve bed cycle gas pressure, and further including the steps of establishing a maximum pressure alarm message when the measured gas pressure exceeds the stored maximum gas pressure and establishing a minimum pressure alarm message when the measured gas pressure falls below the stored minimum gas pressure.

**6.** A method for operating an oxygen concentrator, as set forth in claim 5, and further including the step of stopping operation of said oxygen concentrator in response to at least one of a cycle time alarm message and a maximum pressure alarm message.

**7.** A method for operating an oxygen concentrator, as set forth in claim 5, and further including the steps of transferring any alarm message from the oxygen concentrator to a computer located remote from said oxygen concentrator.

**8.** A method for operating an oxygen concentrator, as set forth in claim 7, and wherein said alarm message is transferred to said computer by storing said computer by writing said alarm message to said memory in said portable memory and display unit, and writing said stored alarm message from said portable memory and display unit into a memory in said computer.

**9.** A method for operating an oxygen concentrator of the type having a gas separation sieve bed which is alternately operated in gas separation cycle and a gas purge cycle, said method comprising the steps of:

a) switching operation of said sieve bed from a gas separation cycle to a gas purge cycle in response to a measured gas pressure;
b) measuring the time of the gas separation cycle for said sieve bed; and
c) establishing a cycle time alarm message when the time of said gas separation cycle is outside a predetermined cycle time range.

**10.** A method for operating an oxygen concentrator, as set forth in claim 9, and further including the steps of storing in a memory a predetermined minimum gas separation cycle time, and wherein said cycle time alarm message is established in response to a comparison of the measured gas separation cycle time and the stored minimum gas separation cycle time.



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11. A method for operating an oxygen concentrator, as set forth in claim 10, and further including the steps of storing in a memory a predetermined maximum gas separation cycle time, and wherein said cycle time alarm message is established in response to a comparison of the measured gas separation cycle time and the stored minimum and maximum gas separation cycle times.

12. A method for operating an oxygen concentrator, as set forth in claim 11, and further including the steps of storing in a memory a minimum gas pressure, a sieve bed cycle gas pressure and a maximum gas pressure, wherein switching of said sieve bed from a gas separation cycle to a gas purge cycle is in response to a comparison of the measured gas pressure and the stored sieve bed cycle gas pressure, and further including the steps of establishing a maximum pressure alarm message when the measured gas pressure exceeds the stored maximum gas pressure and establishing a minimum pressure alarm message when the measured gas pressure falls below the stored minimum gas pressure.

13. A method for operating an oxygen concentrator, as set forth in claim 12, and further including the step of stopping operation of said oxygen concentrator in response to at least one of a cycle time alarm message and a maximum pressure alarm message.

14. A method for operating an oxygen concentrator, as set forth in claim 12, and further including the steps of transferring any alarm message from the oxygen concentrator to a computer located remote from said oxygen concentrator.

15. A method for operating an oxygen concentrator, as set forth in claim 14, and wherein said alarm message is transferred to said computer by programming said oxygen concentrator to connect to said remote computer in response to an alarm and to transfer information identifying said oxygen concentrator and the alarm to said remote computer.

16. A method for operating an oxygen concentrator, as set forth in claim 13, and wherein said alarm message is transferred to said computer by storing said computer by writing said alarm message to a portable memory, and writing said stored memory from said portable memory into a memory in said computer.

17. A monitoring system for an oxygen concentrator, comprising means in said oxygen concentrator for generating and storing data indicative of the operation of said oxygen concentrator, a portable data storage and display unit adapted to connect to said oxygen concentrator, said storage and display unit including means for selectively indicating operation data from said oxygen concentrator and memory means for storing operation data from said oxygen concentrator, a microcomputer located remote from said oxygen concentrator and having a port adapted to connect to said portable data storage and display unit, and means for downloading oxygen concentrator data from said portable data storage and display unit into said microcomputer.

18. A monitoring system for an oxygen concentrator, as set forth in claim 17, and wherein said memory means in said portable memory and display unit is adapted to store operation data from a plurality of oxygen concentrators, and wherein said means for downloading oxygen concentrator data downloads operation data for a plurality of oxygen concentrators from said portable data storage and display unit to said microcomputer.



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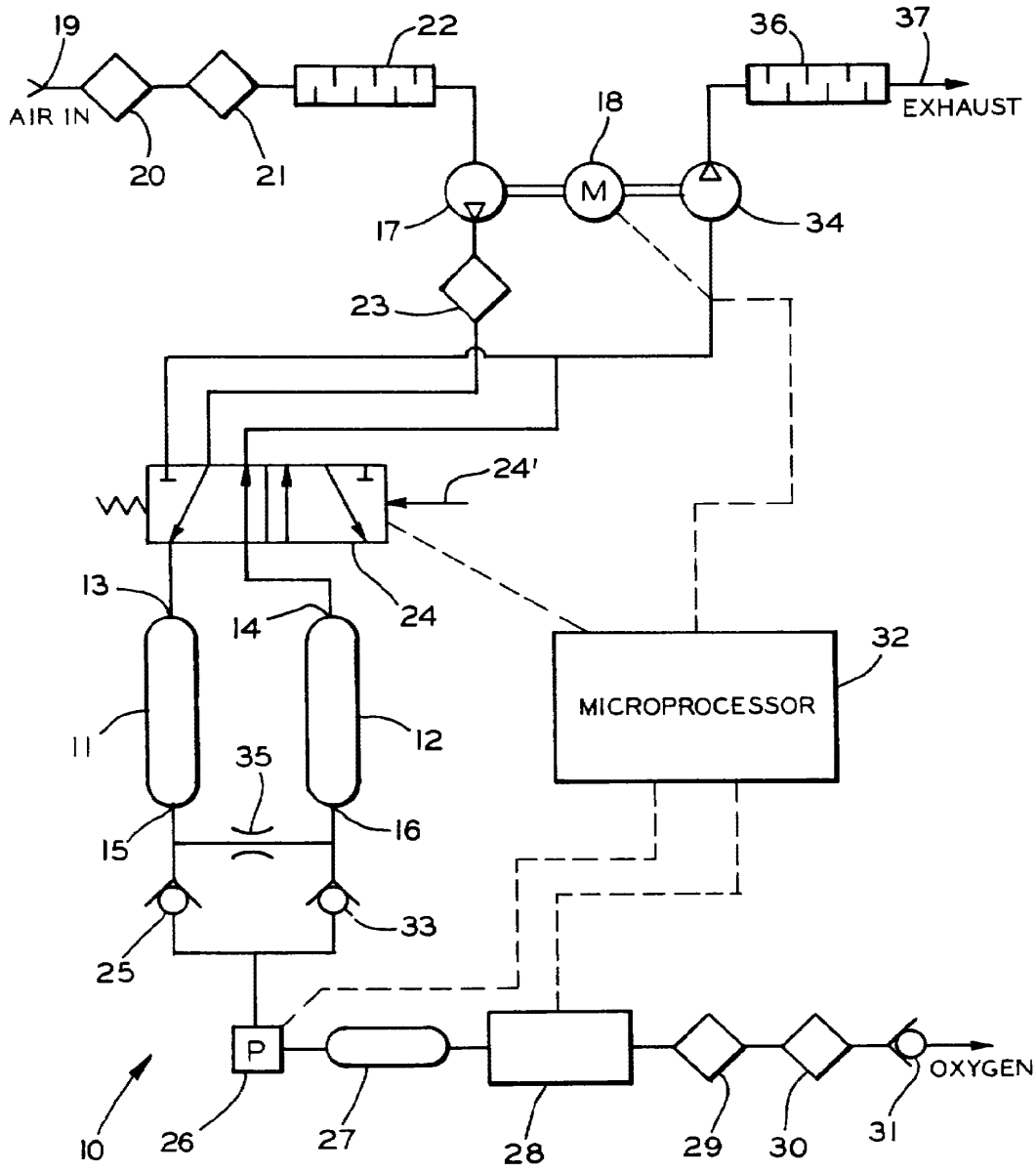



FIG. 1



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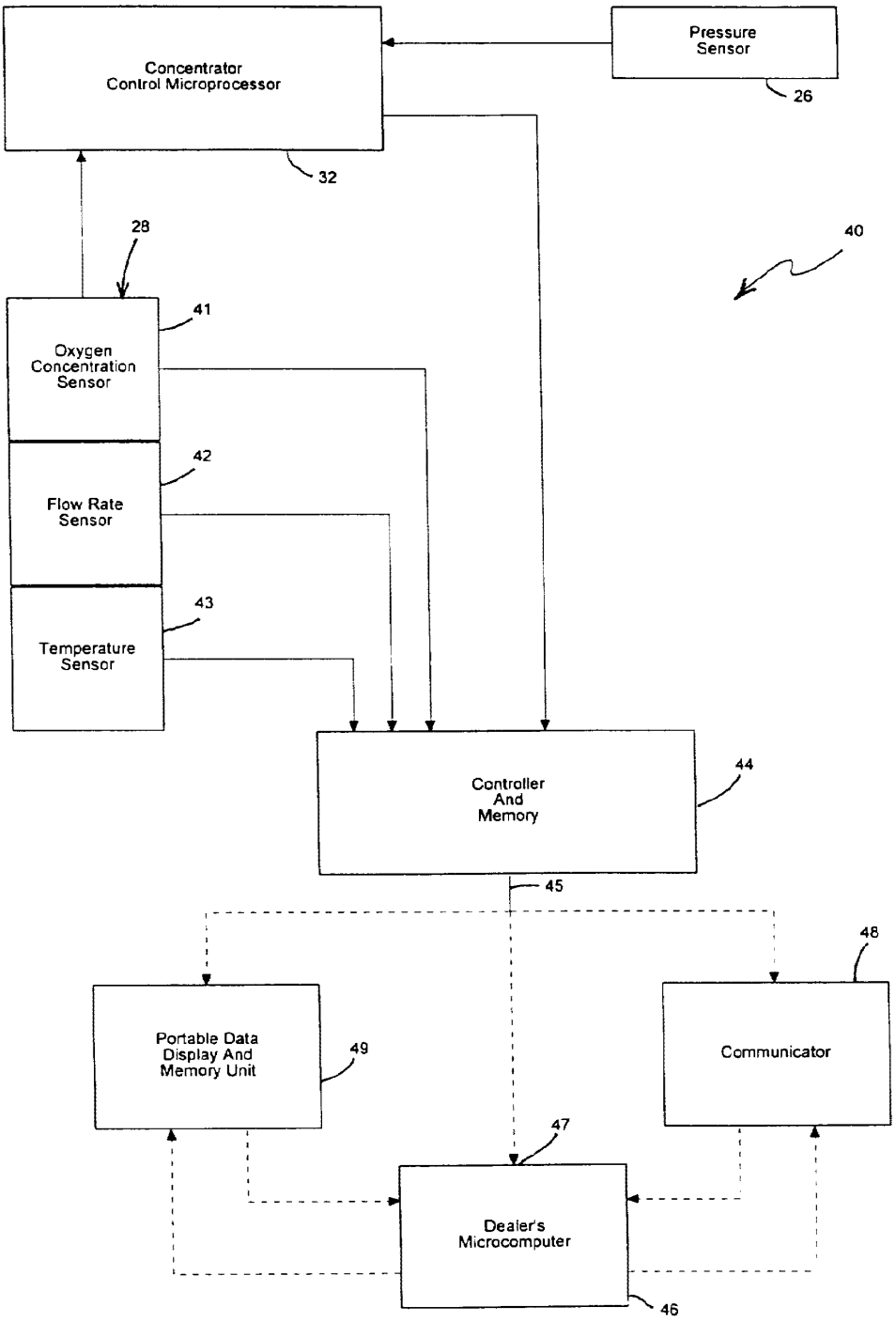



Fig. 2



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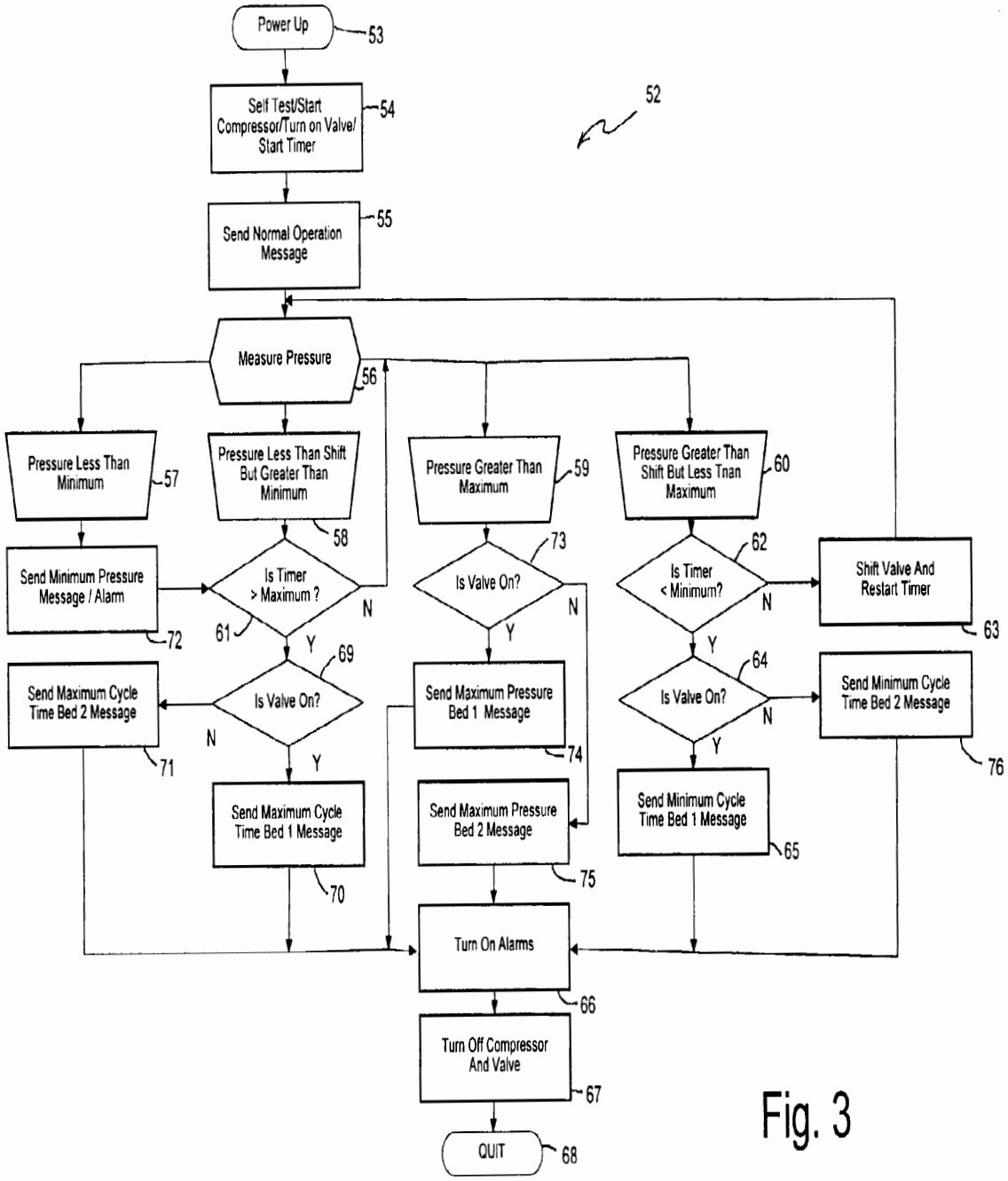

Power Up
53
Self Test/Start Compressor/Turn on Valve/ Start Timer
54
52
Send Normal Operation Message
55
Measure Pressure
56
Pressure Less Than Minimum
57
Pressure Less Than Shift But Greater Than Minimum
58
Pressure Greater Than Maximum
59
Pressure Greater Than Shift But Less Tnan Maximum
60
Send Minimum Pressure Message / Alarm
72
Is Timer > Maximum ?
61
N
Y
Is Valve On?
73
N
Y
Is Timer < Minimum?
62
N
Y
Shift Valve And Restart Timer
63
Send Maximum Cycle Time Bed 2 Message
71
Is Valve On?
69
N
Y
Send Maximum Pressure Bed 1 Message
74
Is Valve On?
64
N
Y
Send Minimum Cycle Time Bed 2 Message
76
Send Maximum Cycle Time Bed 1 Message
70
Send Maximum Pressure Bed 2 Message
75
Send Minimum Cycle Time Bed 1 Message
65
Turn On Alarms
66
Turn Off Compressor And Valve
67
QUIT
68

Fig. 3

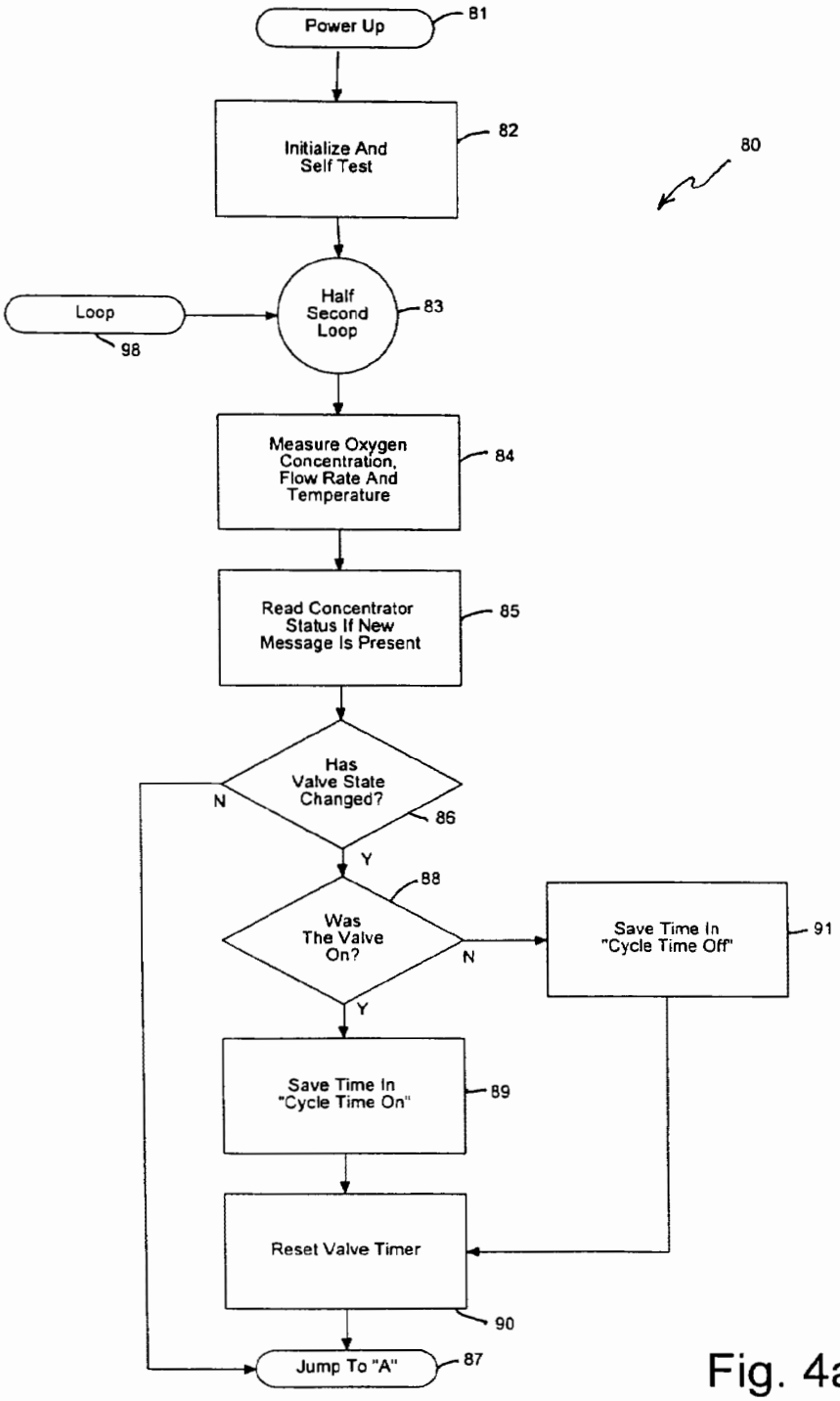



Fig. 4a



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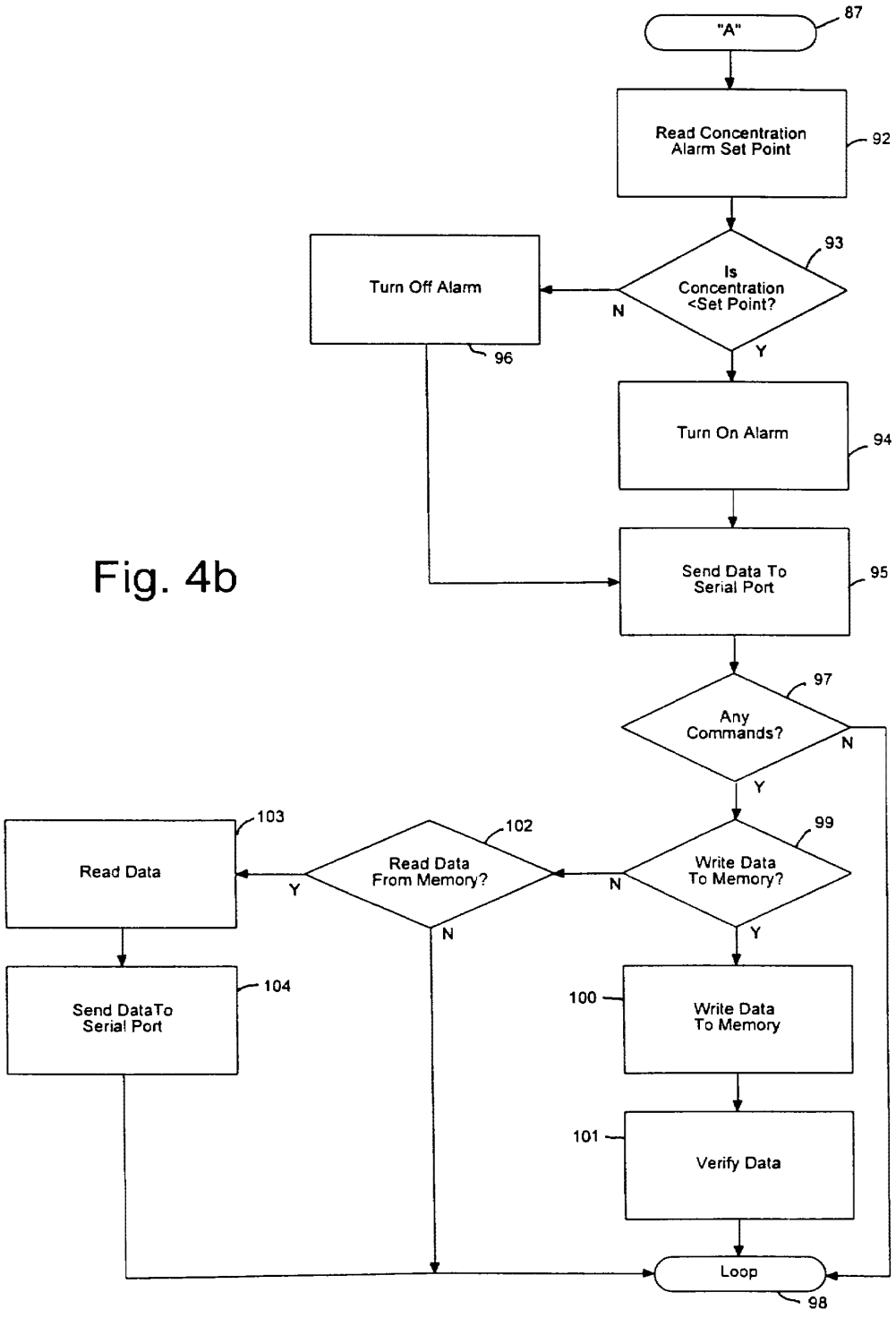



Fig. 4b



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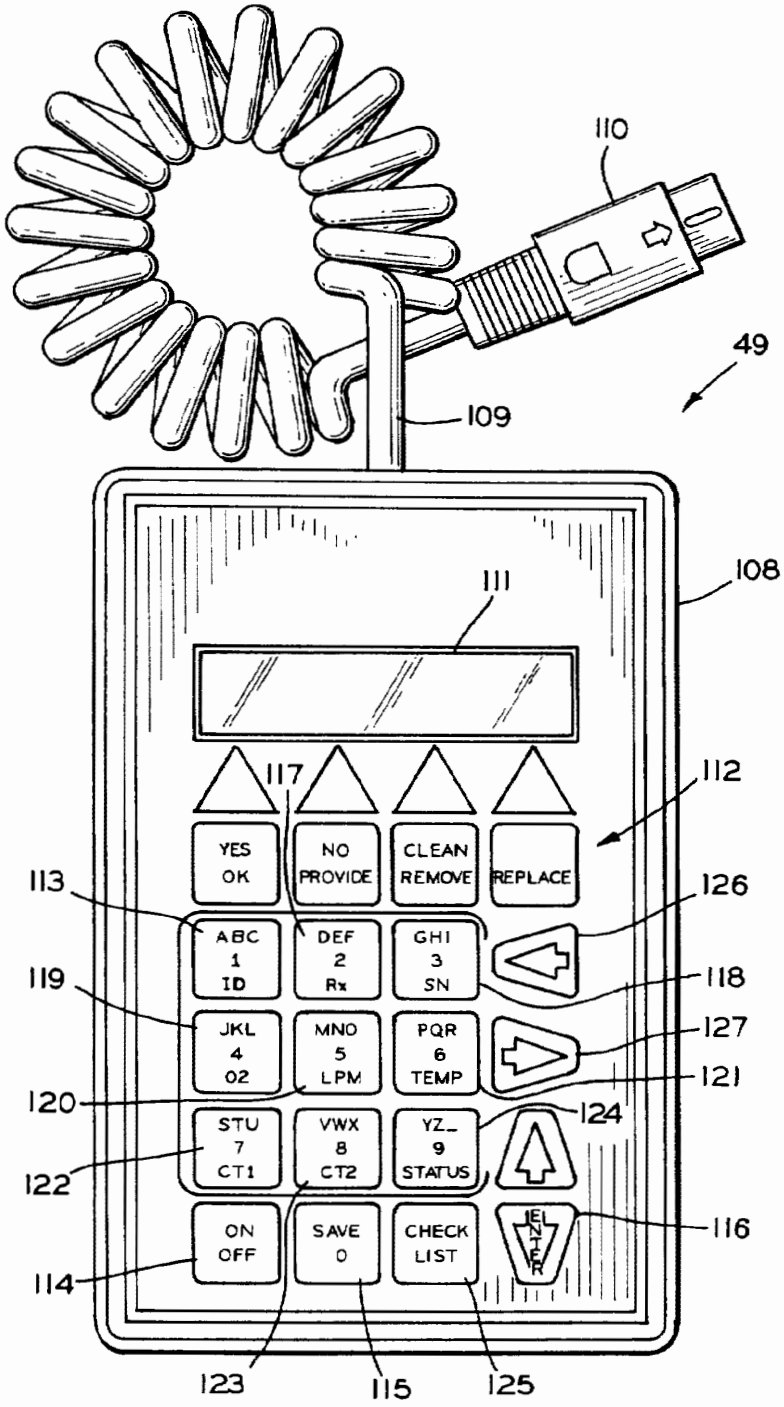



FIG. 5



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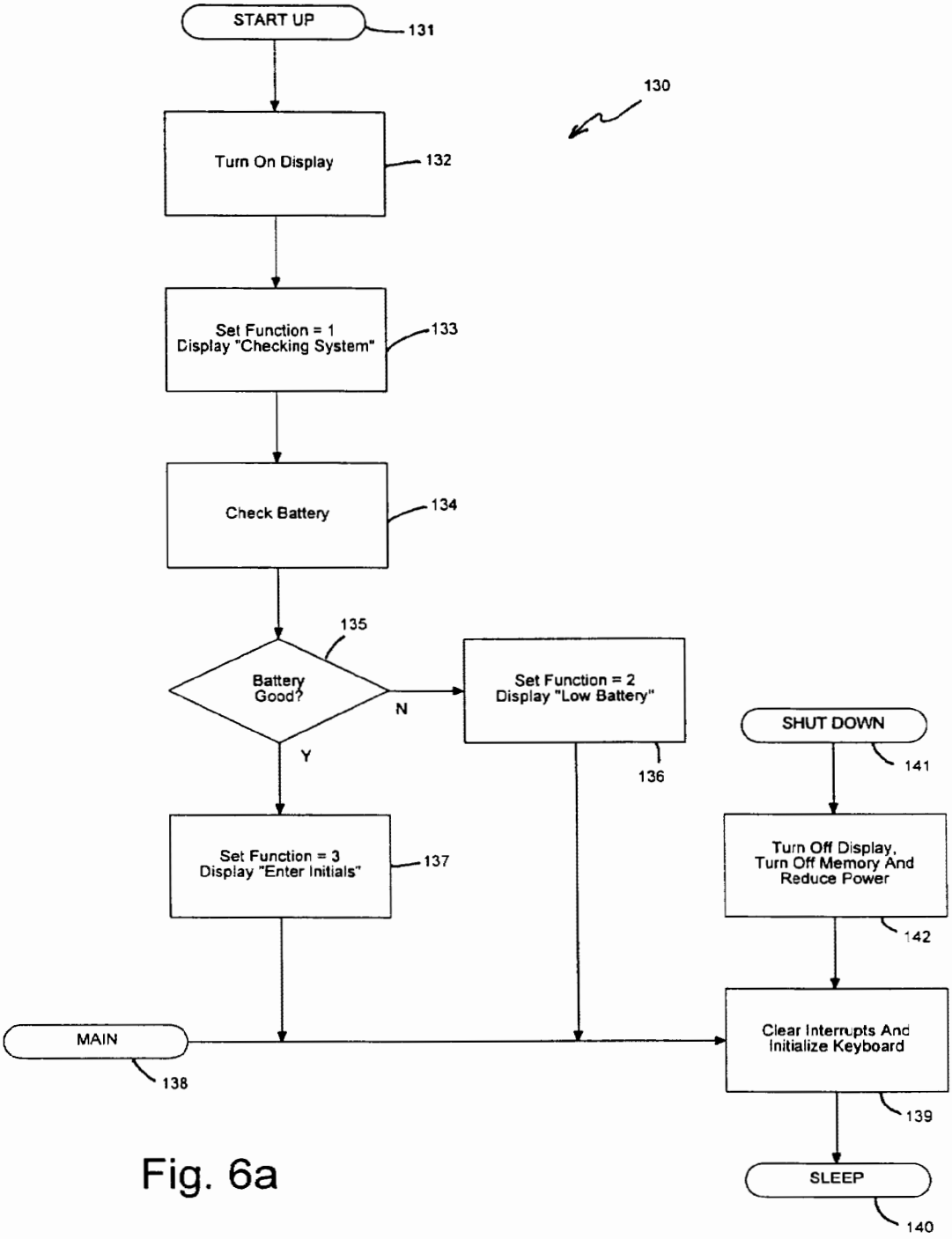



Fig. 6a



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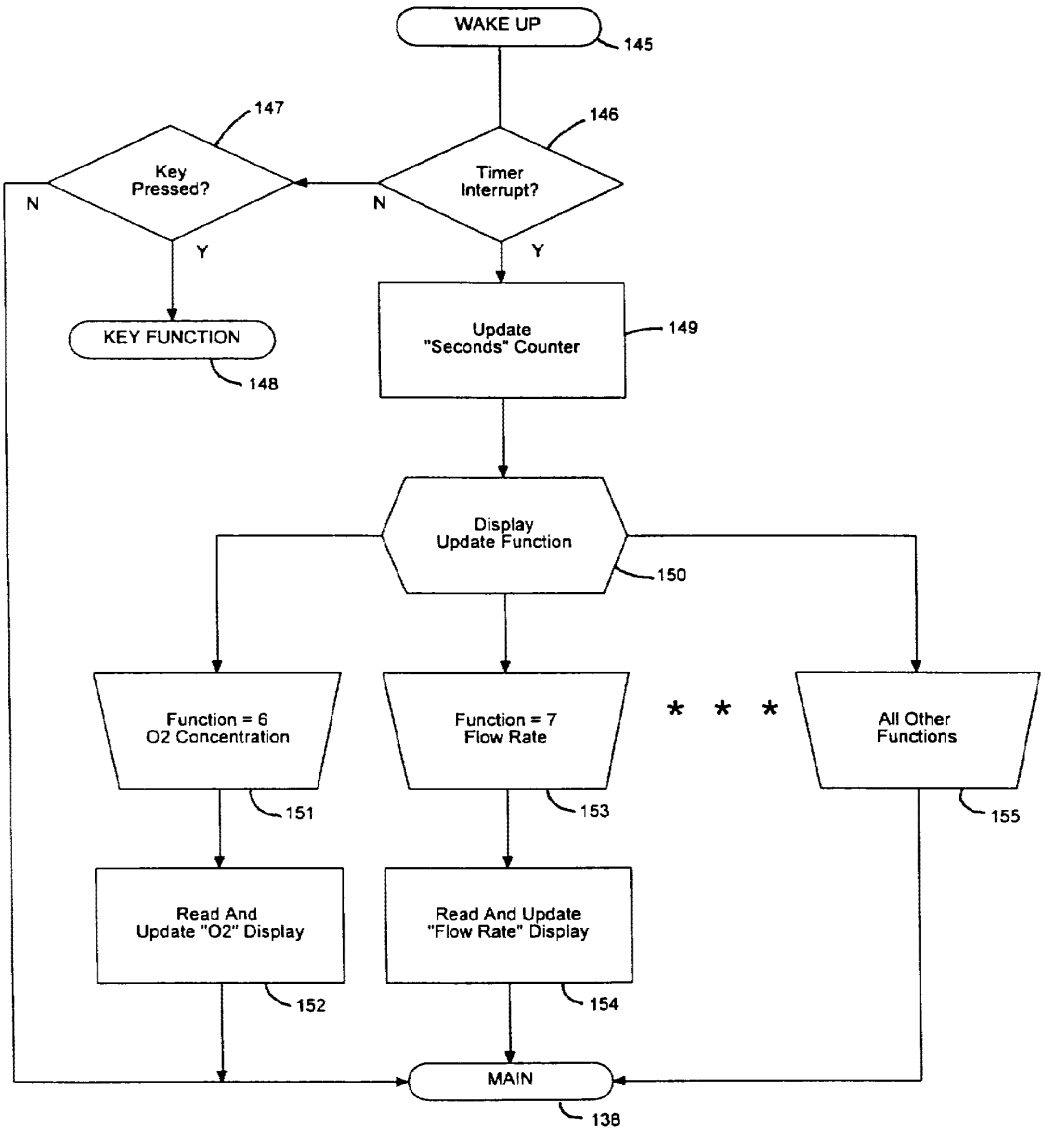



Fig. 6b



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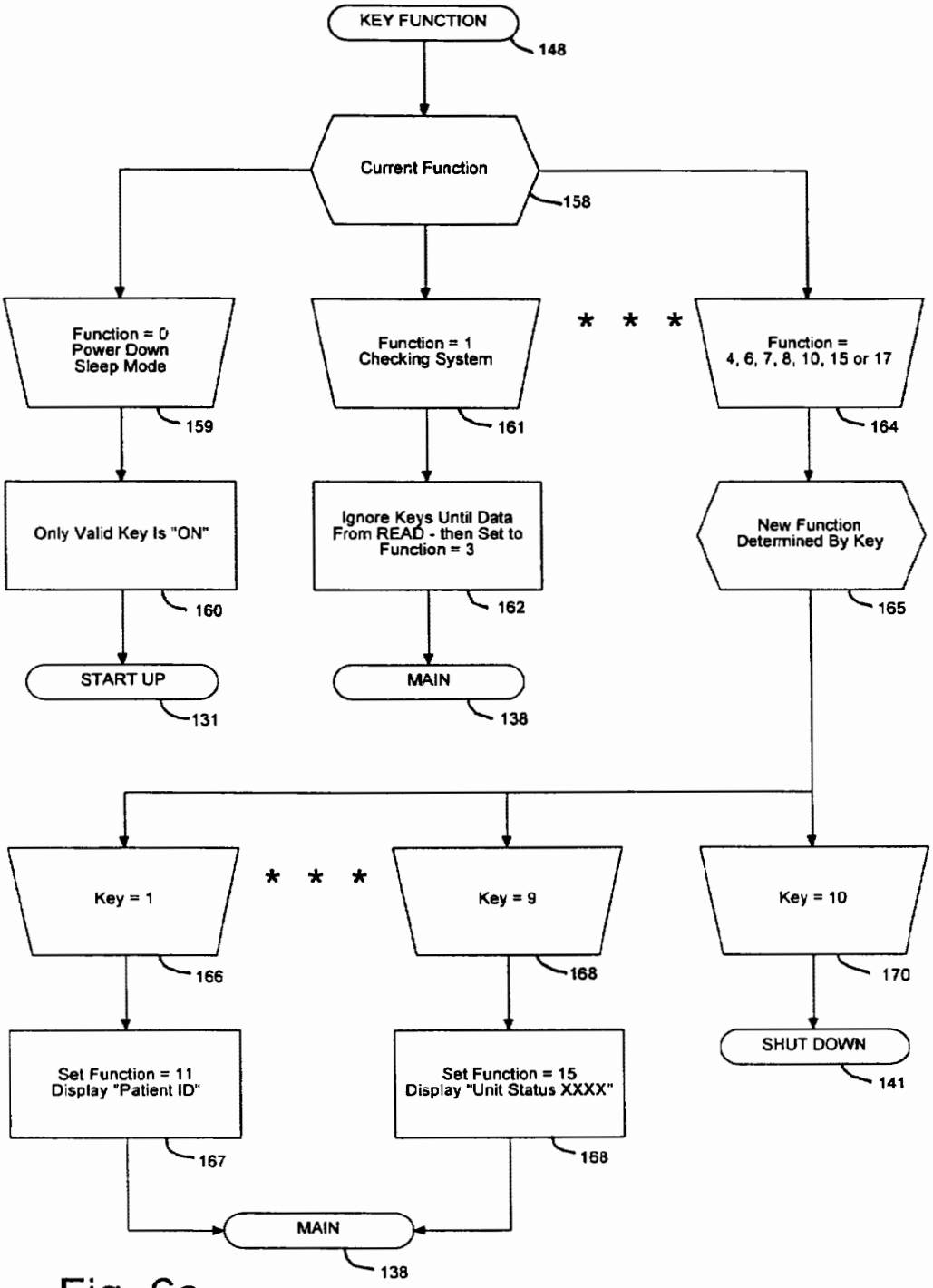



Fig. 6c



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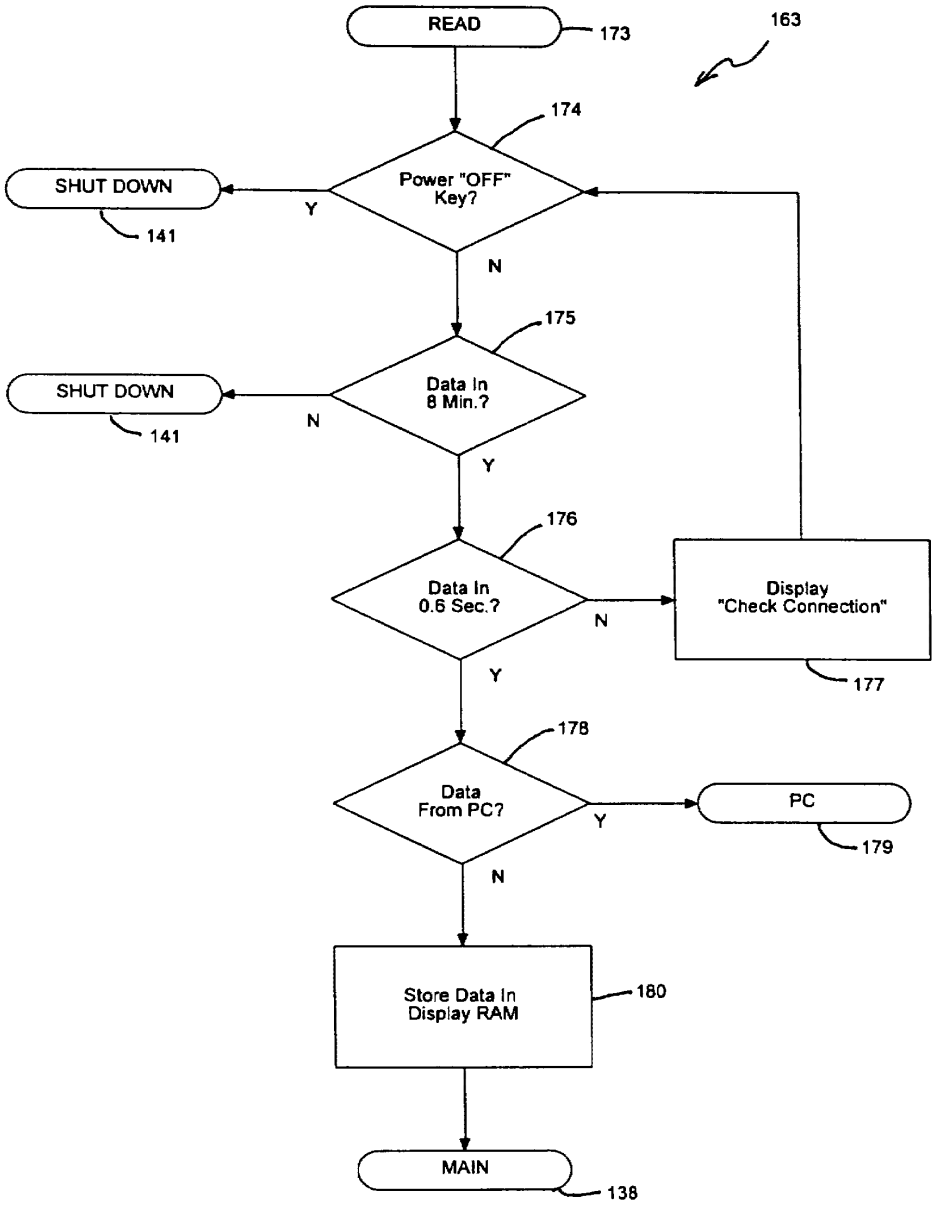



Fig. 6d



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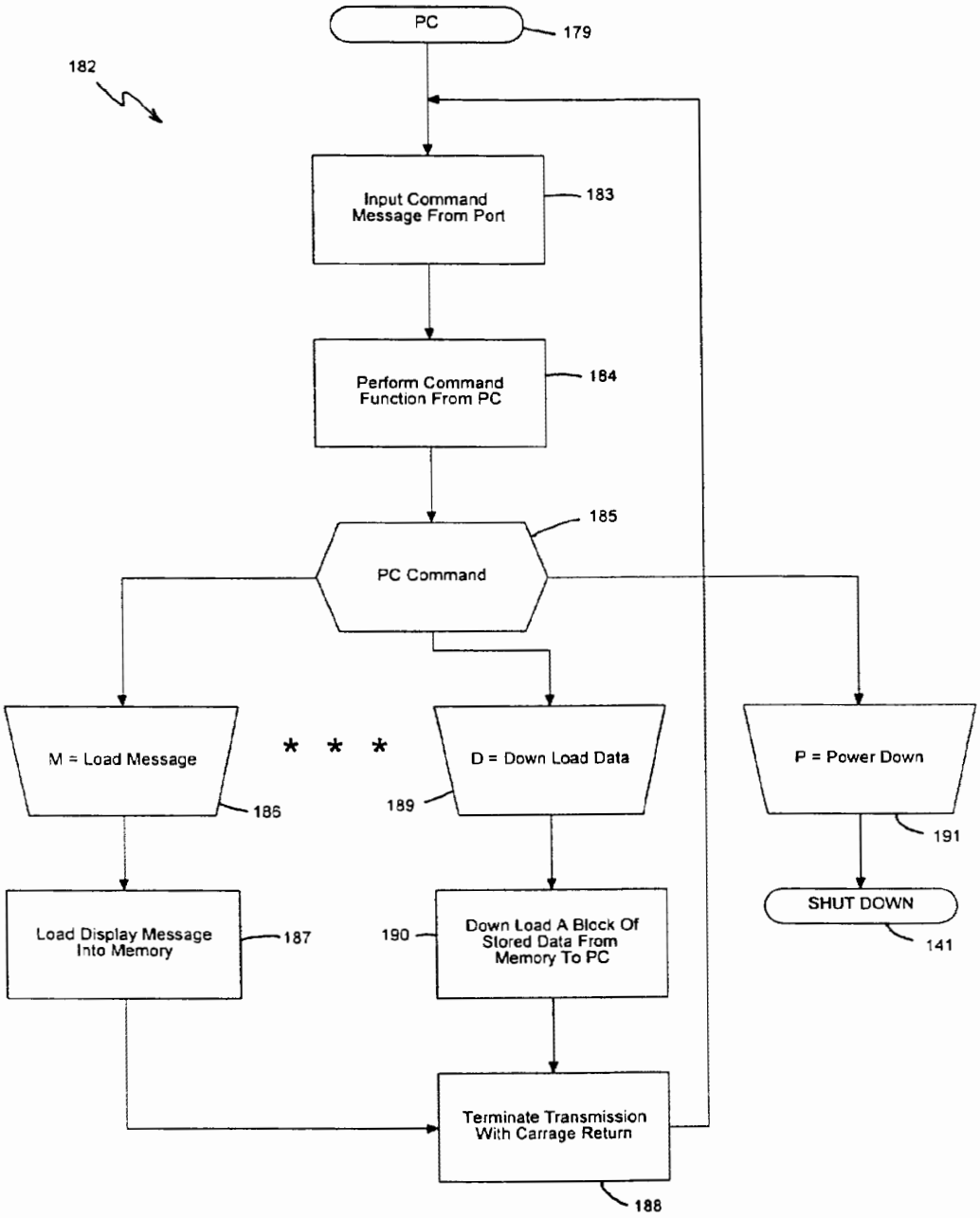



Fig. 6e



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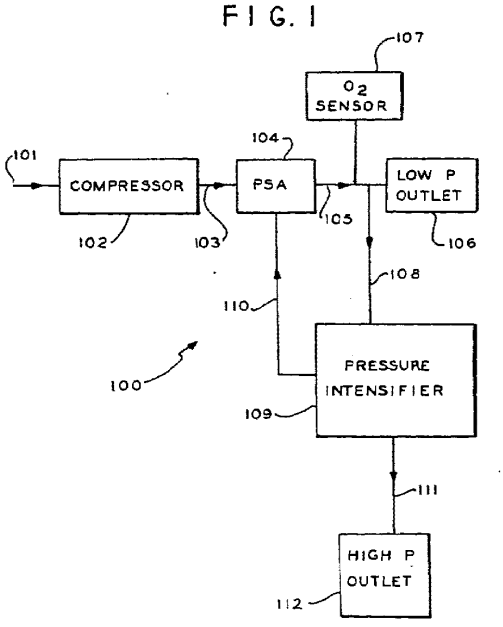
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## (54) Oxygen concentrator

(57) An apparatus for providing oxygen-enriched air at a first pressure and at a second pressure, the second pressure being greater than the first pressure. The apparatus comprises, in combination, a pressure swing adsorption system (104) and a pressure intensifier (109). The pressure swing adsorption system (104) for enriching the oxygen content of air has a pressure of at least the first pressure. The pressure swing adsorption system (104) is adapted to provide oxygen-enriched air to a first outlet (106) at the first pressure and to provide oxygen-enriched air to a pressure intensifier (109) at the first pressure. The pressure intensifier (109) pressurizes the oxygen-enriched air and provides the oxygen-enriched air to a second outlet (112) at the second pressure.

FIG. 1




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## Description

### Field of the Invention

The present invention provides an oxygen concentrator and cylinder recharger and in particular provides a home health care oxygen concentrator and a cylinder recharger that is capable of simultaneously or independently providing oxygen-enriched air at a low pressure and at a high pressure. Such provision of oxygen-enriched air at two different pressures enables the apparatus to be utilized for the provision of oxygen-enriched air at a pressure suitable for use by a patient while at the same time providing oxygen-enriched air at a pressure suitable for recharging of a cylinder, thereby providing the patient with the option to be mobile.

### Background of the Invention

There are a number of instances where the provision of oxygen-enriched air, sometimes referred to as oxygen, is required to be provided at a low pressure. One particular situation where such low pressure oxygen-enriched air is required is in the health care field, both in the health care field within various institutions as well as within a patient's home. In such situations, it may be necessary to provide a continuous flow of low pressure oxygen-enriched air to a patient on an on-going basis. While such patients may be able to withstand the absence of the flow of oxygen-enriched air for short periods of time, such patients frequently are unable to withstand the absence of such a flow of oxygen-enriched air for longer periods of time without suffering major health problems. In particular, such patients are normally not able to move from one location to another without oxygen-enriched air.

Systems for the provision of a flow of low pressure oxygen-enriched air include distillation processes, adsorption separation processes and membrane separation processes. One such process is a so-called pressure swing adsorption (PSA) process which has the advantage of being able to provide oxygen-enriched air in a short period of time after the supply of a suitable feed gas e.g. compressed air, to the apparatus. An example of a pressure swing adsorption process is described in U.S. Patent 4,948,391 of Y. Noguchi, which issued August 14, 1990.

A typical pressure swing adsorption process applied to the provision of oxygen-enriched air could comprise the following steps:

a) introducing air under pressure into a cylinder having an adsorption column of an adsorbent which selectively adsorbs nitrogen,
b) continuing the introduction of the air into the cylinder such that a zone of the adsorbed gas (nitrogen) moves forward through the cylinder,
c) collecting the desired gas (oxygen) that passes from the cylinder,
d) terminating the introduction of air to the cylinder,
e) terminating the collection of oxygen from the cylinder,
f) discharging a portion of the pressurized gas remaining in the cylinder, and
g) returning a portion of the oxygen back into the cylinder to flow through the column in the opposite direction to desorb the gas (nitrogen) adsorbed on the adsorbent and to purge the desorbed gas (nitrogen) from the cylinder.

Steps (a)-(e) effect the separation of nitrogen from the air so as to provide oxygen-enriched air, whereas steps (f) and (g) effect regeneration of the column used in the process. This permits continuous and long-term use of the pressure swing adsorption process to provide oxygen-enriched air.

One of the disadvantages of known processes for the supply of oxygen-enriched air at low pressure is that such processes lack the flexibility to allow the patient to be located at a fixed location but also to be able to move from that location with relative ease with an assurance that the source of oxygen-enriched air is sufficient. For instance, the patient may wish or need to be able to move from a bed or other similar location, e.g. to use toilet facilities, prepare meals, go to another room or change locations for other reasons. Such mobility with existing processes generally requires a patient to be disconnected from a first source of low pressure oxygen-enriched air and to be re-connected to a second system for provision of such air and which is mobile e.g. a cylinder of oxygen-enriched air. The patient would want an assurance that the source of oxygen-enriched air is sufficient, which would be of particular concern if the patient wished to be mobile for an extended period of time e.g. a day or weekend, without having to re-fill the mobile, or ambulatory, cylinder at a dealer or distributor.

### Summary of the Invention

Apparatus has now been found that is capable of simultaneously providing low pressure oxygen-enriched air for use by a patient as well as high pressure oxygen-enriched air for the filling of a pressure cylinder.

Accordingly, one aspect of the present invention provides an apparatus for providing oxygen-enriched air comprising, in combination:

a low pressure oxygen enriched air separation system for enriching the oxygen content of inlet air, said air separation system adapted to provide oxygen-enriched air to a first outlet at a first pressure, said air separation system adapted to provide oxygen-enriched air to a second outlet at approximately said first pressure;
a pressure intensifier connected to said second outlet of the air separation system to pressurize said



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oxygen-enriched air and to provide said oxygen-enriched air to an outlet of the pressure intensifier at a second pressure; and

wherein said air separation system is capable of simultaneously providing oxygen-enriched air to said first outlet and said second outlet such that a flow of oxygen-enriched air can be supplied to said first outlet while said pressure intensifier is pressurizing said oxygen-enriched air to said second pressure.

In another aspect, the present invention provides a method for providing oxygen-enriched air at a first pressure and at a second pressure, said second pressure being greater than the first pressure, said method comprising:

feeding air to an air separation system;

providing oxygen-enriched air from said air separation system to a first outlet at said first pressure and controlling the flow of oxygen-enriched air at said first outlet;

providing oxygen-enriched air from said air separation system to a pressure intensifier at approximately said first pressure, increasing the pressure of said oxygen-enriched air to said second pressure using said pressure intensifier; and

wherein said air separation system is capable of simultaneously providing oxygen-enriched air to said first outlet and said second outlet such that a flow of oxygen-enriched air can be supplied to said first outlet while said pressure intensifier is pressurizing said oxygen-enriched air to said second pressure.

In a further aspect, the present invention provides apparatus for providing oxygen-enriched air comprising, in combination:

an air compressor, a low pressure oxygen-enriched air separation system and a pressure intensifier;

said air compressor being adapted to provide compressed air to said air separation system;

said air separation system being adapted to provide oxygen-enriched air to a first outlet at a first pressure, said first outlet having a controller to control the flow of oxygen-enriched air at a first outlet;

said air separation system further providing oxygen-enriched air to the pressure intensifier at said first pressure, said pressure intensifier being adapted to provide oxygen-enriched air at said second pressure;

wherein said air separation system is capable of simultaneously providing oxygen-enriched air to said first outlet and said second outlet such that a flow of oxygen-enriched air can be supplied to said first inlet while said pressure intensifier is pressurizing said oxygen-enriched air to said second pressure.

In embodiments of the apparatus and method of the present invention, the air separation system is a pressure swing absorption system, and the oxygen-enriched air from the pressure swing adsorption system is provided either simultaneously or independently to said pressure intensifier.

In another embodiment, the apparatus further comprises an air compressor for providing air to said pressure swing adsorption system, said air compressor having an inlet and exhaust air sound suppression system.

## Brief Description of the Drawings

The present invention is illustrated by the embodiments shown in the drawings in which:

Figure 1 is a schematic representation in block format of apparatus of the invention;

Figure 2 is a schematic representation of the apparatus of Figure 1;

Figure 3 is a schematic representation of a fill port adaptor body;

Figure 4 is a schematic representation of a plan view of the fill port adaptor body;

Figure 5 is a schematic representation of a cross section of the fill port adaptor body through lines A-A of Figure 4;

Figure 6 is a schematic representation of a cross section of the fill port adaptor body through lines D-D of Figure 4;

Figure 7 is a schematic representation of a cross section of a part of the fill port adaptor of Figure 6, through B-B;

Figure 8 is a schematic representation of a perspective view of a flow controller body;

Figure 9 is a schematic representation of an end view of the flow controller body of Figure 8;

Figure 10 is a schematic representation of a cross section of the flow controller body of Figure 9 through line A-A;

Figure 11A is a schematic representation of a compressor system; and

Figure 11B is a schematic representation of a compressor sound suppression system, through line B-B of Figure 11A.

## Detailed Description of the Invention

The low pressure oxygen-enriched air separation system may be an oxygen generator, a distiller, an air separator or a membrane separator, but it will be described herein with reference to the preferred embodiment in which it is a pressure swing absorption system.

Fig. 1 shows apparatus of the invention, generally indicated by 100, in block format. Ambient air 101 is connected to compressor 102. Outlet 103 from compressor 102 is connected to pressure swing adsorber (PSA) 104 and pressure intensifier 109 (see Fig. 2). Outlet 105 from pressure swing adsorber 104 is connected to oxygen



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sensor 107, low pressure outlet 106, and through tubing 108, to pressure intensifier 109. Pressure intensifier 109 is connected by tubing 111 to high pressure outlet 112, as well as being interconnected by tubing 110 to pressure swing adsorber 104.

In operation, ambient air enters air inlet 101 and is pressurized in compressor 102. The pressurized air is fed to pressure swing adsorber 104, as well as being fed to pressure intensifier 109 when the latter is energized (see Fig. 2). The oxygen-enriched air from pressure swing adsorber 104 is monitored by oxygen sensor 107, as well as fed to low pressure outlet 106 for use by a patient. In addition, oxygen-enriched air is fed to pressure intensifier 109. Pressurized oxygen-enriched air is fed from pressure intensifier 109 to high pressure outlet 112 where it may be used to re-charge a cylinder of oxygen-enriched air.

Fig. 2 shows an embodiment of an apparatus of the invention, generally indicated by 200, in greater detail. Apparatus 200 has inlet 101 connected in turn to particulate filter 201, tuned resonator 202 and to the inlet to compressor 203. Compressor 203 is driven by motor 204, and has relief valve 205. The outlet from compressor 203 is connected to a heat exchanger, generally indicated by 206, which has heat exchanger section 207 and cooling fan 208. Outlet tubing 209 from heat exchanger 207 is connected to two solenoid valves, 210 and 211, which are in a parallel relationship.

Solenoid valves 210 and 211 are connected to a tuned resonator 212. Each of solenoid valves 210 and 211 is connected to a sieve bed, 213 and 214 respectively, which are also in a parallel relationship. Sieve beds 213 and 214 are molecular sieve beds. Each of the outlets of sieve bed 213 and 214 is connected to a check valve, 215 and 216 respectively. In addition, sieve bed 213 and 214 are interconnected through orifice 217, the interconnection being located between the outlet of each of sieve bed 213 and 214 and each of check valves 215 and 216.

The outlets from each of check valves 215 and 216 are joined together, and connected to oxygen plenum 220. Oxygen plenum 220 is connected through orifice 221 to oxygen sensor 222 and filter 223, which is vented to the atmosphere. Oxygen plenum 220 is also connected to low pressure switch 237. The outlet of oxygen plenum 220 is connected in turn to pressure regulator 231, through orifice 232, filter 233, check valve 234, flow meter 235 to patient outlet 236. Patient outlet 236 is a low pressure outlet i.e. the pressure would normally be only a few inches of water, as will be understood by persons skilled in the art.

The outlet of the oxygen plenum 220 is also connected to a pressure intensifier generally indicated by 240. Pressure intensifier 240 has a first stage cylinder 241 and a second stage cylinder 242, with drive air cylinder 243 located therebetween. The opposed sides of drive cylinder 243 are connected through solenoid valves 244 and 245 to tubing between solenoid valve 210 and sieve bed 213, and tubing between solenoid valve 211 and sieve bed 214, respectively, of the pressure swing adsorber. Check valve 247 is located between oxygen plenum 220 and first stage cylinder 241. First stage cylinder 241 is also connected through check valve 248 and check valve 250 to second stage cylinder 242. Rupture disc 246 is attached to first stage cylinder 241 and rupture disc 251 is attached to second stage cylinder 242. Filters 252 and 253 are connected to first stage cylinder 241 and second stage cylinder 242, respectively, and vented to the atmosphere.

Check valve 249 is connected to filter 260, pressure switch 261 and pressure connection 262, which is the high pressure connection.

In an example of a typical operation of the embodiment of Figure 2, air enters air compressor 203 through inlet 101, particulate filter 201 and tuned resonator 202. Tuned resonator 202 is for sound suppression of compressor 203. The air is compressed to the operating pressure e.g. a pressure ranging from 20 to 30 psig. The compressed air obtained is routed through a fan cooled tube and fin heat exchanger 206 which cools the air before it enters the molecular sieve beds 213 and 214. The cooling of the air from the compressor optimizes performance of the molecular sieve beds as well as cooling the oxygen-enriched air obtained from the beds before it reaches the patient. Air flow into and out of the beds is controlled by two solenoid valves (valve 210 and valve 211) which allow compressed air to either enter the bed or to be vented from the bed to atmosphere.

A normal three phase cycle is (i) a first filling phase to fill bed 213 and exhaust bed 214, (ii) an intermediate overlap phase and (iii) a second filling phase to fill bed 214 and exhaust bed 213.

In phase (i), solenoid valve 210 allows compressed gas to enter bed 213 while valve 211 is venting gas from bed 214 to atmosphere. The molecular sieve in bed 213 adsorbs water vapour and nitrogen from the compressed air, allowing oxygen and argon to pass through the sieve. A portion of the gas is used as product gas which is delivered to the patient as low pressure oxygen-enriched air. The remainder of the oxygen-enriched air passes through an orifice, 217, and purges nitrogen and water vapour out of bed 214 as it is vented to atmosphere through solenoid valve 211. At the end of this phase, which in a typical embodiment of the invention lasts approximately 8 seconds, the pressure in bed 213 is typically approximately 30 psig and in bed 214 it is typically slightly above zero psig. At this time, bed 213 is almost saturated with nitrogen and continued flow through this bed without purging would result in nitrogen breakthrough and loss of purity in the product gas.

In overlap phase (ii), there is an overlap of approximately 1.6 seconds in which both solenoid valves 210 and 211 are switched to allow flow into both molecular sieve beds. This allows the air from sieve bed 213, initially at approximately 30 psig, as discussed above, to back flow through solenoid valve 210 into bed 214 which



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is initially at a low pressure. The crossflow of air in addition to the air from compressor provides a quick charge of bed 214, thereby reducing the compressor flow requirements and improving the efficiency of the system. At the end of the overlap cycle, the pressure in both beds is typically approximately 20 psig.

At the end of the overlap phase i.e. in phase (iii), solenoid valve 210 is switched to the vent position allowing bed 213 to vent to atmosphere until the pressure is only slightly above zero. Venting the bed to atmosphere plus back filling with product gas from bed 214 purges nitrogen and water vapour from bed 213 in preparation for the next cycle. At the same time bed 214 is providing product gas to the patient and purging bed 213. At the end of this phase, the entire cycle is repeated.

The oxygen-enriched air from the sieve beds passes through check valves 215 and 216 into the product storage (oxygen plenum 220). From oxygen plenum 220, the oxygen-enriched air passes into pressure regulator 231, through flow control orifice 232, HEPA filter 233 and check valve 234 to flow meter 235. Regulator 231 reduces the pressure from, for example, 30 psig in the oxygen storage plenum 220 to a constant pressure e.g. 6 psig, at the inlet of flow meter 235. Flow meter 235 is adjustable by the patient to provide the prescribed flow rate of oxygen at very low pressure.

A small amount of gas from the plenum e.g. typically less than 250 cc per minute, is continuously monitored by the oxygen sensor to ensure that the oxygen purity is above a predetermined value e.g. 90%. If the purity is below the predetermined or threshold value, a microprocessor energizes a warning light to alert the patient that an equipment malfunction has occurred and to prevent cycling of the pressure intensifier.

Pressure intensifier 240 may be standard two-stage device with a drive air cylinder and first and second stage product gas cylinders. The intensifier is turned on by opening solenoid valve 244 and 245 and which are either both open or both closed. A typical pressure intensifier cycle is as follows:

1. In the first step of the cycle, drive air enters drive air cylinder 243 through solenoid valve 244 beginning at the start of the overlap cycle of the molecular sieve beds discussed above, which occurs just before bed 213 starts charging, and continuing through the bed 213 charging cycle. This causes the piston to shuttle, drawing product gas into first stage cylinder 241 through check valve 247 and compressing the oxygen-enriched air in the second stage cylinder 242 which exits through check valve 249.
2. The drive air cylinder supply is common to the sieve bed supply and controlled by the sieve bed solenoid valves. Thus, the air in drive air cylinder 243 also helps precharge the sieve beds during the overlap phase which further increases the efficiency of the system. In this step during the overlap phase, the air in the drive air cylinder 243 back flows through solenoid valve 244, backflows through solenoid valve 210 and back into bed 214 through solenoid valve 211.
3. When the bed solenoid valves cycle, ambient air enters drive air cylinder 243 through solenoid valve 245 causing the piston to shuttle compressing the oxygen enriched air in the first stage cylinder 241. This gas then passes through check valves 248 and 250 and enters the second stage cylinder. The drive air, entering through solenoid valve 245 in this side of the drive air cylinder 243 aids in precharging bed 213 during the overlap cycle, as previously described.

After leaving the pressure intensifier, the gas passes through a filter 260 and the high pressure coupling 262 before entering a pressure vessel (not shown). The pressure intensifier will continue to operate until the pressure is high enough to activate a pressure switch 261 in the product line which signals the controller to close solenoid valves 244 and 245.

An example of apparatus for monitoring gaseous oxygen concentration is shown in U.S. Patent 5,402,665 of R.F. Hart et al, which issued April 4, 1995. An example of a pressure swing adsorber system having a pneumatically driven booster compressor is shown in U.S. Patent 5,354,361 of K.M. Coffield, which issued October 11, 1994.

In embodiments of the present invention, the first pressure is in the range of 5-10 psi and the second pressure is in the range of up to 3000 psi. The apparatus of the present invention is particularly adapted to be capable of providing both first and second pressures simultaneously.

The oxygen content of the oxygen-enriched air may be varied over a wide range but is preferably at least 85% by volume. In preferred embodiments, the oxygen content is at least 90% by volume, especially in the range of 92-94% by volume.

The oxygen concentrator described herein utilizes a pressure intensifier to raise the pressure of a portion of the oxygen-enriched air to a suitable pressure e.g. 2000 psig, for storage in pressure vessels e.g. a cylinder, for use by ambulatory patients. It will be understood that when the cylinder is empty, the pressure intensifier will provide oxygen-enriched air at a relatively low pressure, for instance, about the pressure in the storage plenum e.g. 30 psig, but that this pressure will rise as the cylinder fills e.g. to the aforementioned 2000 psig.

While the oxygen concentrator may be operated so as to produce oxygen-enriched air having a content of, for example, 93% oxygen, and thereby meet certain regulations relating to the provision of oxygen-enriched air for patients, such oxygen-enriched air may not meet the specifications for a medical-grade oxygen. Thus cylinders with adaptors meeting regulations for medical-



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grade oxygen may not be able to be used with the oxygen concentrator. Nonetheless, a unique integrated high pressure fitting and flow controller is preferred, that allows the patient to easily and safely fill their cylinders with oxygen-enriched air e.g. 93% oxygen, from the concentrator. Such a high pressure fitting and flow controller is shown in Figures 3-11.

Figure 3 shows a perspective view of a fill port adaptor body, generally indicated by 300. Fill port adaptor body 300 has a housing 301 with integral cradle 302, handle 303, locking clip 304 and adaptor housing 305. Cradle 302 of housing 301 is adapted to accept the flow controller body described herein below. Cradle 302 has fill port adaptor 306 therein, which is further shown in Figure 5. Fill port adaptor 306 is connected to handle 303, and is movable into cradle 302 using handle 303. Adaptor 307 extends from housing 301 from the opposed side thereof to fill port adaptor 306, and is interconnected therewith as shown in Figure 5.

Locking clip 304 is intended to retain the flow controller body within cradle 302, as described below. Latching of the flow controller body into cradle 302 occurs automatically when the flow controller body of a cylinder is placed in the cradle. However, an operator must manually move locking clip 304 into a release position to remove the cylinder. Similarly, handle 303 must be moved before the flow controller body of a cylinder may be placed in cradle 302 or removed therefrom, to reduce risk of damage to fill port adaptor 306.

Adaptor housing 305 is open on its upper face, as illustrated, and has housing window 308 on the opposed side thereof. The end of adaptor housing 305 opposed to cradle 302 has housing slot 309 therein.

Figure 4 is a plan view of fill port adaptor port 300. Handle 303 is located on housing 301, and is adjacent to cradle 302. Locking clip 304 is shown as extending into cradle 302. Cradle 302 has adaptor housing 305 extending therefrom and in an aligned relationship. Adaptor housing 305 has housing window 308 therein, with housing slot 309 in the end thereof. Figure 4 shows that adaptor 307 is connected to gear rack 401 which extends into housing 301.

Figure 5 shows a cross section of fill port adaptor body 300 through line A-A. Locking clip 304 is pivoted about locking clip pivot 501. Locking clip spring 504 located within housing 301 biases locking clip base 503 in an outwards direction so that locking clip lip 502 is biased into to cradle 302 to lock the flow controller body therein.

A fill port adaptor, generally indicated by 505 in Figure 5, is on the opposed side of cradle 302 to locking clip 304.

Fill port adaptor extends from adaptor 307 to adaptor pin 511. Adaptor 307 is connected to adaptor channel 506 which is located within adaptor channel housing 507 which in turn is located within adaptor housing 512. Gear rack 401, shown in Fig. 4 and Fig. 6 is machined into adaptor housing 512. As discussed herein, adaptor housing 512 is movable with respect to housing 301. Adaptor channel 506 connects with spring chamber 513 in which is located adaptor spring 508 and check valve base 509. Check valve base 509 extends through check valve seat 510 and terminates in adaptor pin 511. In the retracted position as shown in Fig. 5, adaptor pin 511 is within housing 301, but in the engaged position the adaptor pin extends into cradle 302.

Figure 6 shows a cross section of the adaptor body shown in Figure 4, through D-D. Handle 303 is located on housing 301. Shaft 605 extends downward from handle 303 and connects handle 303 to pinion 601; pinion 601 is more clearly seen in Figure 7, and is attached to shaft 605 by screw 604. Rotation of handle 303 causes rotation of pinion 601. Handle 303 has, on its lower side, handle projection 608, which is shown as being located in indent 609 in housing 301. Indent 609, and a similar indent that is not shown, serve to position and retain handle 303 in predetermined locations.

In a preferred embodiment, indent 609 has an adjustable screw therein (not shown), such screw being tapered or having a shoulder thereon. Projection 608 contacts the adjustable screw, on said taper or shoulder. Adjustment of the position of the screw adjusts the location of the taper or shoulder, and hence radially adjusts the position at which handle 303 is positioned and retained. This permits precise adjustment of the positioning of the fill port adaptor.

Figure 7 shows the rack and pinion, generally indicated by 600. Rack and pinion 600 has pinion 601 with pinion cogs 602. Pinion 601 rotates about pinion shaft 603, which extends down from handle 303, forming part thereof, as discussed above. Pinion 601 is located within pinion housing 606 which is part of housing 301. Pinion cogs 602 intermesh with ratchet slots 607 of ratchet 401. Ratchet 401 forms part of adaptor channel housing 507 illustrated in Figure 5.

Figure 8 illustrates the flow controller body, generally indicated by 700. Flow controller body 700 has pressure gauge 701, rupture disc housing 709, outlet fitting 706, and flow restrictor 704. Outlet fitting 706 is adapted to receive a flexible hose. The body is adapted to be threaded onto a high pressure cylinder by threaded port 705, flow restrictor 704 being located within threaded port 705. Flow controller body 700 has an integral cradle housing 702 which is shaped and dimensioned to fit precisely in the cradle 302 (see Figure 3). A flow control (ON/OFF) knob 703 with mechanical alignment/interlock bar 708 is attached to flow controller body 700 at the end opposed to the threaded port 705. Fill port adaptor inlet 707 is located in integral cradle housing 702.

Figure 9 shows an end view of flow controller body 700. Flow controller body 700 has flow restrictor 704 in the end thereof. Pressure gauge 701 and outlet port 706 are shown at an angle.

Figure 10 shows a cross section of flow controller body 700 through line A-A of Figure 9. Adaptor inlet 707 has inlet valve 800. Inlet valve 800 has inlet valve stem



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801 located within inlet valve chamber 802, but it will be noted that as illustrated inlet valve stem 801 does not extend fully to the end of inlet valve chamber 802. Inlet valve stem 801 has inlet valve spring 803 which biases valve end 804 towards valve seat 805 thereby biasing the valve toward a closed position.

In operation, flow controller body 700 is attached to a cylinder, not shown, through threaded cylinder port 705. Flow controller body 700 would normally be located on a cylinder, and remain thereon although it can be removed if desired. The cylinder with flow controller body 700 thereon is placed into fill port adaptor body 300 which forms part of the oxygen concentrator. Cradle housing 702 is adapted to fit within cradle 302 of fill port adaptor body 300. Alignment/interlock bar 708 on flow controller body 700 fits into housing slot 309 on adaptor housing 305. Flow controller knob 703 must be in the OFF position in order to insert alignment/interlock bar 708 into housing slot 309. This locates the rupture disk opposite housing window 308, but also locates inlet valve 800 opposite fill port adaptor 306. In order to insert flow controller body 700 into cradle 302, it is necessary to move locking clip 304 away from fill port adaptor 300, but after flow controller body 700 has been inserted into cradle 302, locking clip 304 biases back and locks flow controller body 700 in position.

Handle 303 is then tilted, rotated and locked in position, with handle projection 608 of handle 303 engaging in indent 609 in housing 301, at which time adaptor 306 moves inwards into cradle 302. In doing so, fill port adaptor 306 is inserted into adaptor inlet 707, with adaptor pin 511 contacting valve end 804. This moves valve end 804 away from valve seat 805, thereby opening the valve. However the amount of movement of valve end 804 is limited, thereby also causing check valve 509 to open. This forms a channel for the flow of oxygen-enriched air from the oxygen concentrator through fill port adaptor body 300, flow controller body 700, and out of cylinder port 704 into the cylinder. The cylinder cannot be removed without moving handle 303 such that fill port adaptor 306 becomes retracted. This disconnects the fill port adaptor so that the oxygen concentrator is not connected to the cylinder, and closes both the cylinder and the oxygen concentrator check valves 804 and 509, to prevent the flow or leakage of gas therefrom. In particular, the check valve in the fill port adaptor, 509, prevents gas from oxygen plenum 220 from escaping to atmosphere through check valves 247-250 in the pressure intensifier when the pressure intensifier is not in operation. A single movement of handle 303 opens or closes the channels for flow of high pressure air to the cylinder.

It will be appreciated that the flow controller body permits the use of cylinders of various sizes to be filled using the concentrator described herein.

It is to be understood that the oxygen concentrator described herein may be operated in a mode in which oxygen-enriched air is only supplied to a patient. Alternatively, the oxygen concentrator may be operated in a mode in which oxygen-enriched air is only supplied for the re-charging or filling of a cylinder. However, the oxygen concentrator also may be operated in a dual mode, supplying oxygen-enriched air to a patient and simultaneously supplying oxygen-enriched air for re-charging or filling of a cylinder.

Figure 11A shows a compressor system, generally indicated by 820. Compressor system 820 has compressor 821 mounted on base 822. Base 822 also has an integral sound suppressor 823 for suppressing noise from both air drawn into the compressor and air exhausted from the concentrator. The integral sound suppressor assists in reducing the size and complexity of the compressor system and permits the suppressors to be mounted in the base, thus reducing the manufacturing steps required in the fabrication of the oxygen concentrator. Sound suppressor 823 is more clearly shown in cross-section, through B-B, in Figure 11B.

Figure 11B illustrates air compressor sound suppressor, generally indicated by 900. Sound suppressor 900 has compressor base 901 into which two outer silencer tubes 902 and 903 have been moulded, utilizing the natural contour of the air compressor. End caps 904 and 905 along with inner silencer tubes 906 and 907 are attached to each end of the compressor base silencer tubes 902 and 903. Compressor port 910 is adapted to be connected to an air compressor.

Operation of the sound suppressor can be described as follows:

As air is drawn into an air compressor, valve noise and air noise is generated. In more general terms, noise is generated when compressed air is exhausted to atmosphere in any pneumatic device.

In air compressor sound suppressor 900, compressor port 910 is connected to an air compressor and air is drawn into silencer tube chamber 908, which is moulded into the compressor base 901, via inlet port 911 in the end cap 904. The air travels through inner silencer tube 906 and into the inlet silencer chamber 908. Noise coming from the air compressor is muffled, because as sound travels from the air compressor and into compressor port 910, the noise is absorbed and deflected through outlet silencer chamber 909 by the outer silencer tube 902 and the inner silencer tube 906 before exiting through outlet 911. By varying the diameter and length of outer silencer tube 902 and inner silencer tub 906, sound compressor 900 can be tuned to minimize the unwanted noise from the compressor.

The concentrator exhaust silencer operates in a similar manner as the inlet silencer except exhaust air noise is minimized. Air enters the silencer tube 903 through exhaust inlet 913 in end cap 905. Noise coming from the exhaust air is muffled, because as air travels through inlet exhaust inlet 913, the exhaust air noise is absorbed and deflected by the inner silencer tube 907 and outer silencer tube 903 before exiting through outlet 912. By varying the diameter and length of the outer si-



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lencer tube 903 and inner silencer tube 907, sound suppressor 900 can be tuned to minimize the unwanted exhaust air noise.

It is understood that the air compressor may be mechanically or an electrically driven compressor, or any other mechanism that compresses air for introduction into the oxygen enrichment portion of the apparatus.

The apparatus described herein may be used to fill cylinders of a variety of different sizes and configurations by providing suitable adaptors to the oxygen fill port adaptor, including use of adaptors to fill stationary cylinder which would not otherwise fit into the fill port. For example, a flow controller body may be adapted to fit in the fill port adaptor 300 and to be connected via a high pressure hose to a flow controller body 700 on a cylinder which is not, itself, contained with the fill port adaptor. The substitute flow controller body may be used to otherwise mimic the functions of a flow controller body when installed in the fill port adaptor to permit gas from the oxygen concentrator to be used to replenish a large (for example, stationary) cylinder which would not otherwise fit within the fill port adaptor body 300. Lastly, it is be noted that outlet port 706 may be used to connect via tubing (not shown) the cylinder to which the flow controller body is attached to the patient so that a patient may breathe oxygen enriched air contained in the cylinder by adjusting the flow rate of oxygen enriched air by means of the flow control knob 703. Pressure gauge 701 may be used by the patient to monitor the gas remaining in the cylinder.

## Claims

1. An apparatus for providing oxygen-enriched air comprising, in combination:

   a low pressure oxygen enriched air separation system for enriching the oxygen content of inlet air, said air separation system adapted to provide oxygen-enriched air to a first outlet at a first pressure, said air separation system adapted to provide oxygen-enriched air to a second outlet at approximately said first pressure;
   a pressure intensifier connected to said second outlet of the air separation system to pressurize said oxygen-enriched air and to provide said oxygen-enriched air to an outlet of the pressure intensifier at a second pressure; and
   wherein said air separation system is capable of simultaneously providing oxygen-enriched air to said first outlet and said second outlet such that a flow of oxygen-enriched air can be supplied to said first outlet while said pressure intensifier is pressurizing said oxygen-enriched air to said second pressure.

2. The apparatus of Claim 1 further comprising a compressor, said compressor being adapted to provide compressed air to said air separation system.

3. The apparatus of Claim 1 or Claim 2 further comprising a compressor, said compressor being adapted to provide compressed air to said pressure intensifier.

4. The apparatus of any one of Claims 1-3 in which the first pressure is provided to a flow meter and is suitable for patient respiration and said second pressure is suitable for filling high pressure cylinders or storage plenums.

5. The apparatus of any one of Claims 1-4 in which the first pressure is up to about 10 psi and the second pressure is up to about 3000 psi.

6. The apparatus of Claim 2 in which the air separation system and the pressure intensifier are interconnected and are adapted to cycle cooperatively.

7. The apparatus of any one of Claims 1-6 wherein the outlet to the pressure intensifier is adapted to accept an ambulatory pressure vessel using a securing mechanism.

8. The apparatus of any one of Claims 1-7 further comprising an oxygen concentration monitoring system to prevent cycling of the pressure intensifier if the oxygen concentration of the oxygen-enriched air falls below a predetermined value.

9. The apparatus of any one of Claims 1-8 in which the flow rate of the oxygen-enriched air to the first outlet may be adjusted independent of the pressure intensifier.

10. The apparatus of any one of Claims 1-9 which further includes a rupture disc in the pressure intensifier to prevent the pressure exceeding a predetermined value.

11. The apparatus of Claim 2 having a sound suppression system to suppress inlet noise to said compressor and exhaust noise from said air separation system.

12. The apparatus of Claim 11 in which the sound suppression system is located in a base of said compressor, said sound suppression system comprising inlet and exhaust air silencer tubes integrally incorporated into said base, said tubes being tuned to minimize noise associated with intake of air into the compressor and exhaustion of air from the concentrator.

13. The apparatus of Claim 12 in which said tubes have



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a variable length and a variable diameter, said tubes being tuned to suppress said noise by varying the diameter and length of said tubes.

**14.** The apparatus of any one of Claims 1-13, wherein said low pressure oxygen-enriched air separation system is an oxygen generator.

**15.** The apparatus of Claim 14, wherein said oxygen generator is a pressure swing adsorption system.

**16.** The apparatus of any one of Claims 1-13, wherein said air separation system is one of a distiller, an air separator and a membrane separator.

**17.** The apparatus of any one of Claims 1-13, wherein said pressure intensifier includes:

a first stage cylinder, a second stage cylinder and a drive air cylinder located therebetween.

**18.** A method for providing oxygen-enriched air at a first pressure and at a second pressure, said second pressure being greater than the first pressure, said method comprising:

feeding air to an air separation system;
providing oxygen-enriched air from said air separation system to a first outlet at said first pressure and controlling the flow of oxygen-enriched air at said first outlet;
providing oxygen-enriched air from said air separation system to a pressure intensifier at approximately said first pressure, increasing the pressure of said oxygen-enriched air to said second pressure using said pressure intensifier; and
wherein said air separation system is capable of simultaneously providing oxygen-enriched air to said first outlet and said second outlet such that a flow of oxygen-enriched air can be supplied to said first outlet while said pressure intensifier is pressurizing said oxygen-enriched air to said second pressure.

**19.** The method of Claim 18 in which said air separation system and said pressure intensifier are interconnected such that cycling of the air separation system and said pressure intensifier are coordinated.

**20.** The method of Claim 18 or Claim 19 in which the oxygen-enriched air has an oxygen content of at least 90% by volume.

**21.** The method of any one of Claims 18-20, wherein said low pressure oxygen-enriched air separation system is an oxygen generator.

**22.** The method of Claim 21, wherein said oxygen generator is a pressure swing adsorption system.

**23.** Apparatus for providing oxygen-enriched air comprising, in combination:

an air compressor, a low pressure oxygen-enriched air separation system and a pressure intensifier;
said air compressor being adapted to provide compressed air to said air separation system;
said air separation system being adapted to provide oxygen-enriched air to a first outlet at a first pressure, said first outlet having a controller to control the flow of oxygen-enriched air at a first outlet;
said air separation system further providing oxygen-enriched air to the pressure intensifier at said first pressure, said pressure intensifier being adapted to provide oxygen-enriched air at said second pressure;
wherein said air separation system is capable of simultaneously providing oxygen-enriched air to said first outlet and said second outlet such that a flow of oxygen-enriched air can be supplied to said first inlet while said pressure intensifier is pressurizing said oxygen-enriched air to said second pressure.

**24.** The apparatus of Claim 23 in which said air separation system and said pressure intensifier being interconnected such that cycling of the air separation system and said pressure intensifier are coordinated.

**25.** The apparatus of Claim 23 or Claim 24 in which said compressor is adapted to provide compressed air to said pressure intensifier.

**26.** The apparatus of any one of Claims 23-25 in which the first pressure is provided to a flow meter and is suitable for patient respiration and said second pressure is suitable for filling high pressure cylinders or storage plenums.

**27.** The apparatus of any one of Claims 23-26 in which the first pressure is up to about 10 psi and the second pressure is up to about 3000 psi.

**28.** The apparatus of any one of Claims 23-27 adapted to accept an ambulatory pressure vessel to said second outlet using a securing mechanism.

**29.** The apparatus of any one of Claims 23-28 in which there is an oxygen concentration monitoring system to prevent cycling of the pressure intensifier if the oxygen concentration of the oxygen-enriched air falls below a predetermined value.



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**30.** The apparatus of any one of Claims 23-29 in which the flow rate of the oxygen-enriched air to the first outlet may be adjusted independent of the pressure intensifier.

**31.** The apparatus of any one of Claim 23-30 which further includes a rupture disc in the pressure intensifier to prevent the pressure exceeding a predetermined value.

**32.** The apparatus of any one of Claims 23-31 having a sound suppression system to suppress inlet noise to said air compressor and exhaust noise from said concentrator.

**33.** The apparatus of Claim 32 in which the sound suppression system located in a base of said compressor, said sound suppression system comprising inlet and exhaust air silencer tubes integrally incorporated into said base, said tubes being tuned to minimize noise associated with intake of air into the compressor and exhaustion of air from the air separation system.

**34.** The apparatus of Claim 33 in which said tubes have a variable length and a variable diameter, said tubes being tuned to suppress said noise by varying the diameter and length of said tubes.

**35.** The apparatus of Claim 28 further comprising an ON/OFF flow controller adapted to prevent flow of oxygen-enriched air through said second outlet when said second outlet is disconnected from said ambulatory pressure vessel.

**36.** The apparatus of any one of Claims 23-35, wherein said low pressure oxygen-enriched air separation system is an oxygen generator.

**37.** The apparatus of Claim 36, wherein said oxygen generator is a pressure swing adsorption system.

**38.** The apparatus of any one of Claims 23-35, wherein said air separation system is one of a distiller, an air separator and a membrane system.

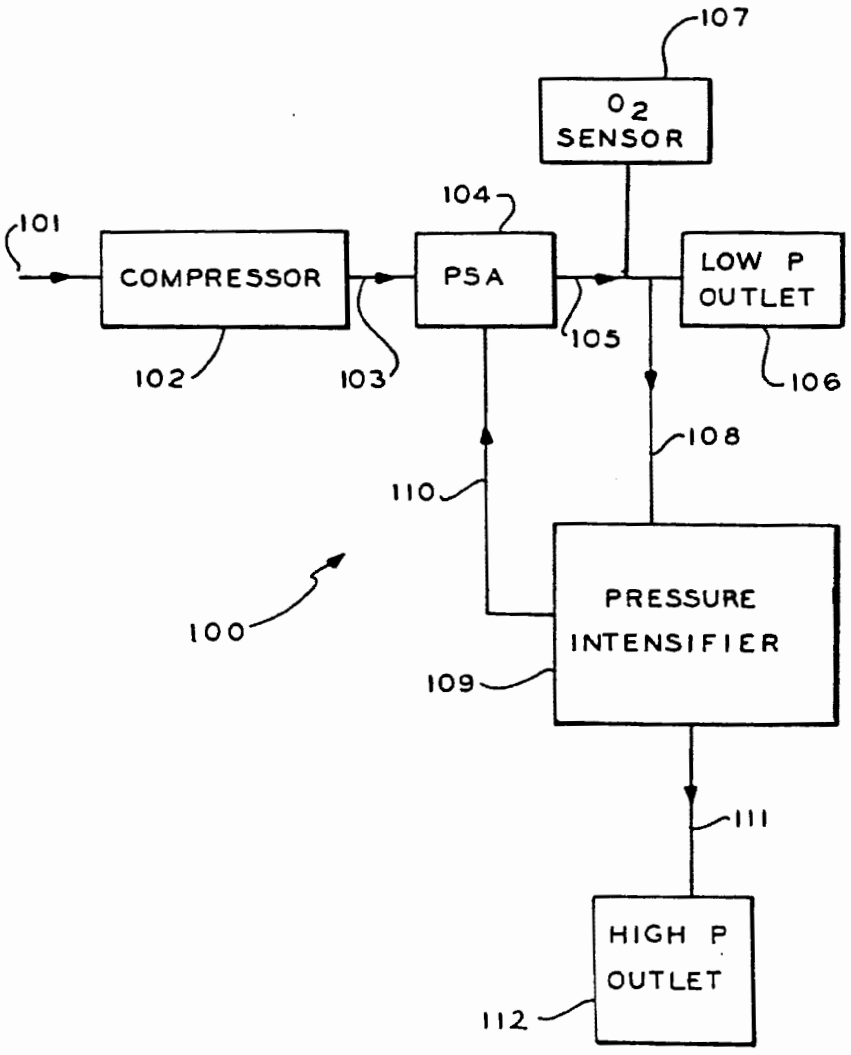
**39.** The apparatus of any one of Claims 23-35, wherein said pressure intensifier includes:

a first stage cylinder, a second stage cylinder and a drive air cylinder located therebetween.



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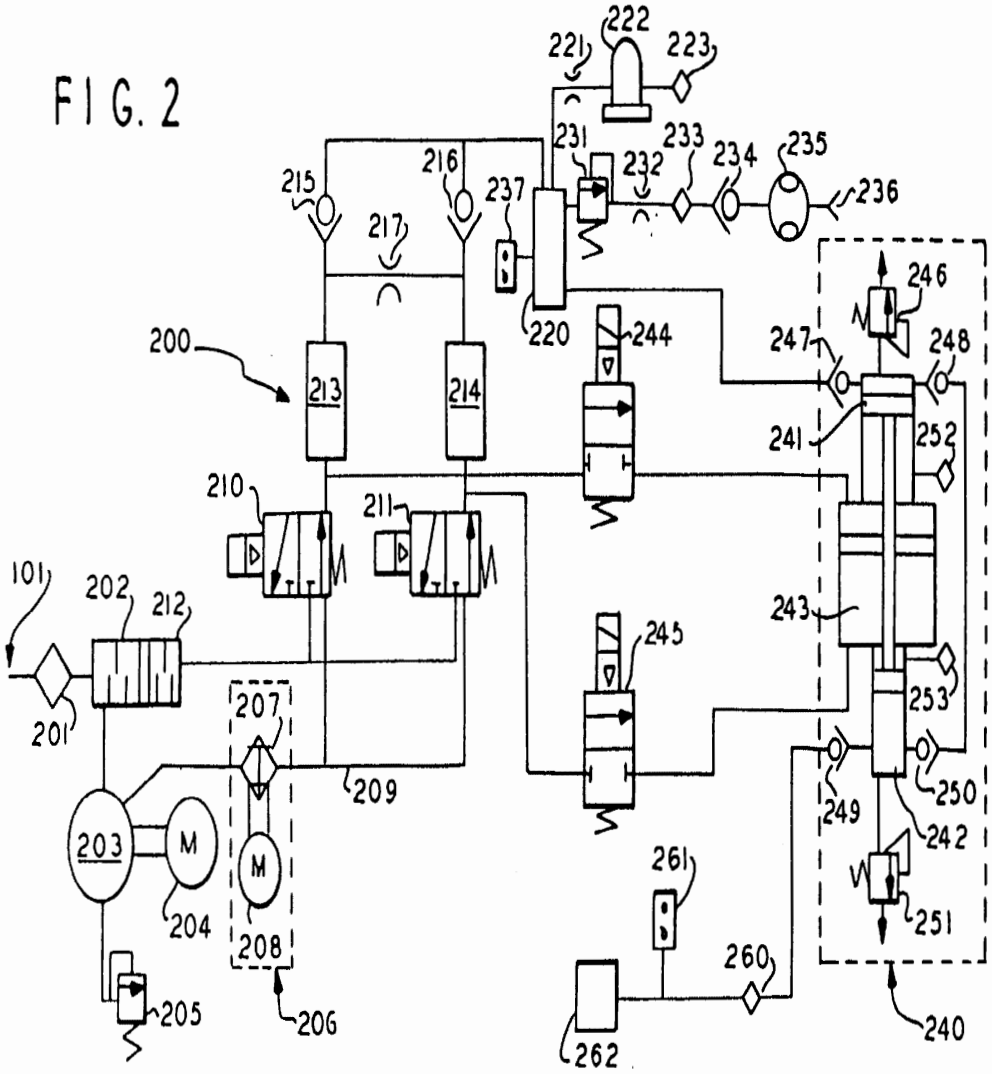
FIG. 1






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FIG. 2

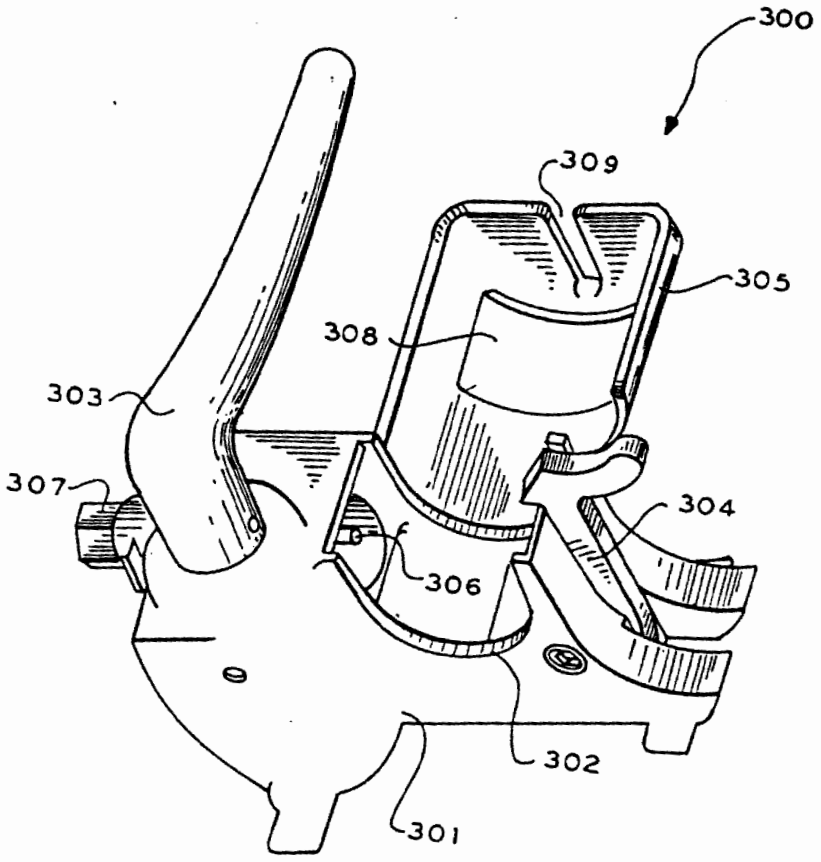





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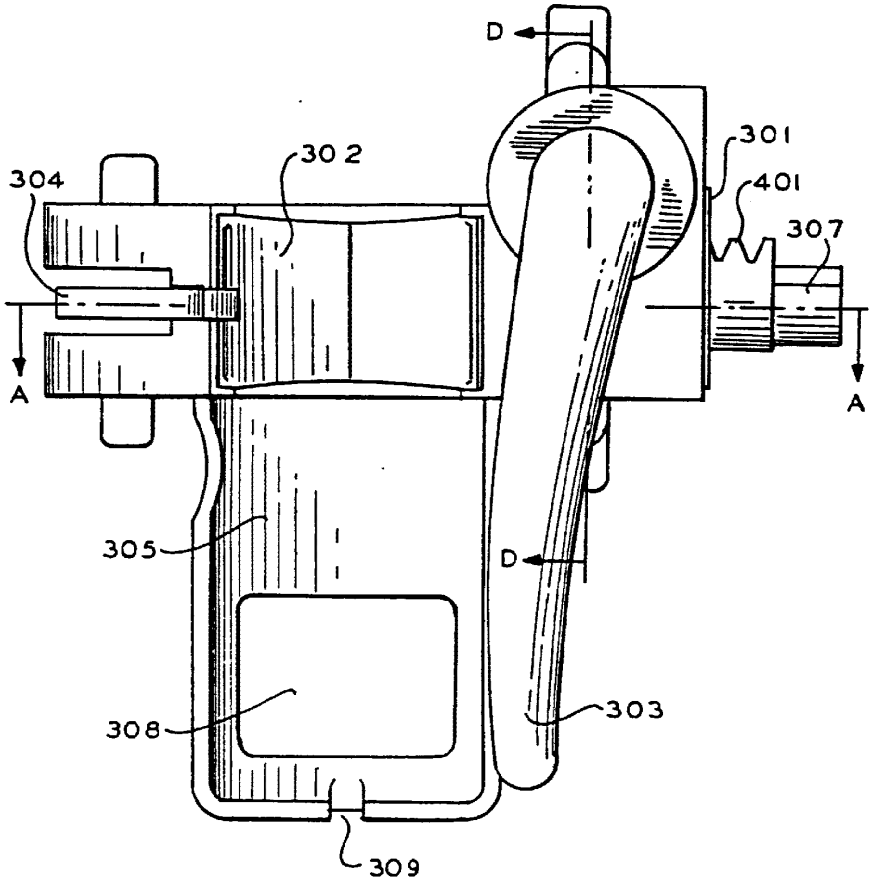
FIG. 3






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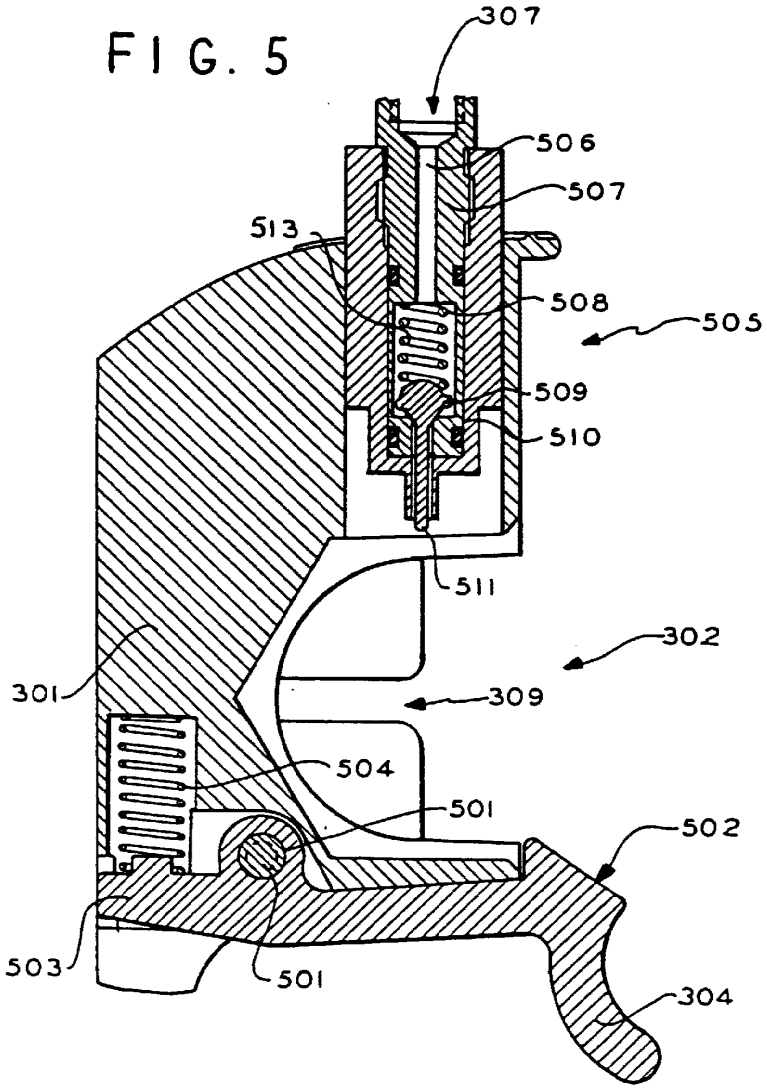
FIG. 4






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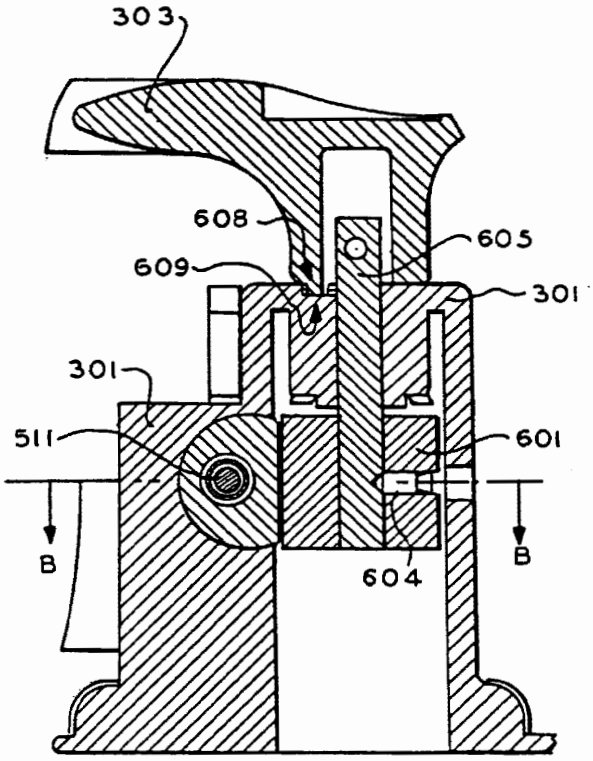
FIG. 5






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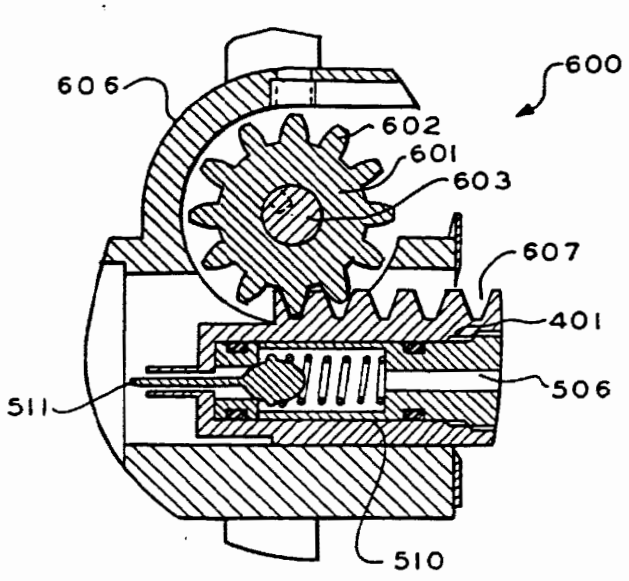
FIG. 6






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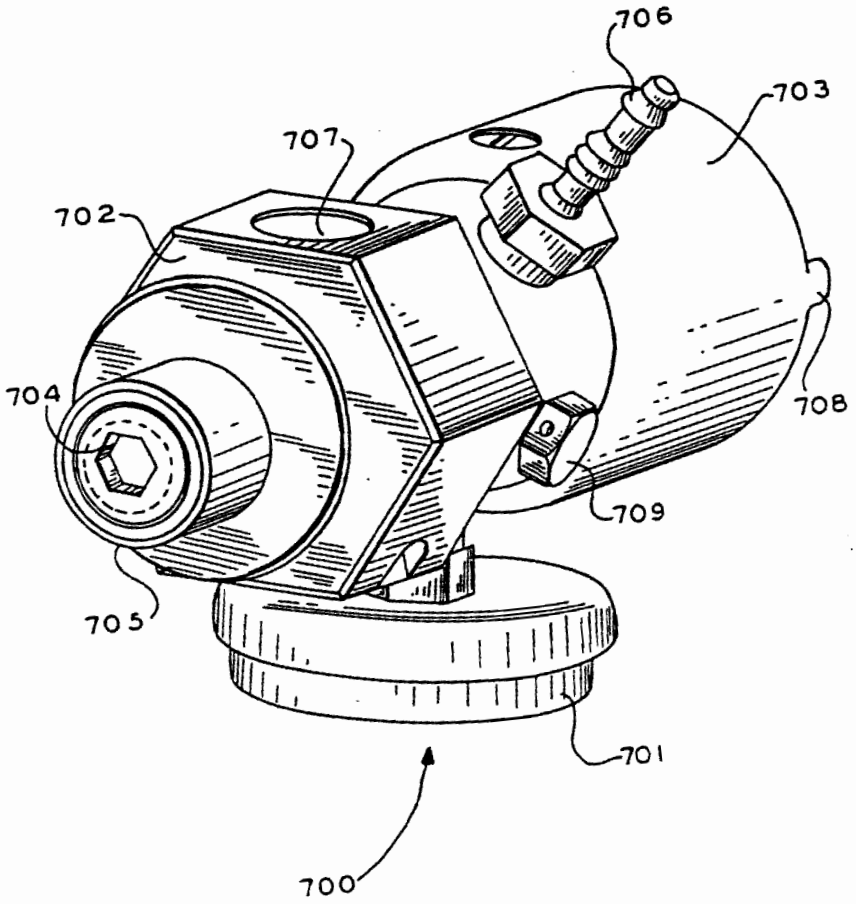
FIG. 7






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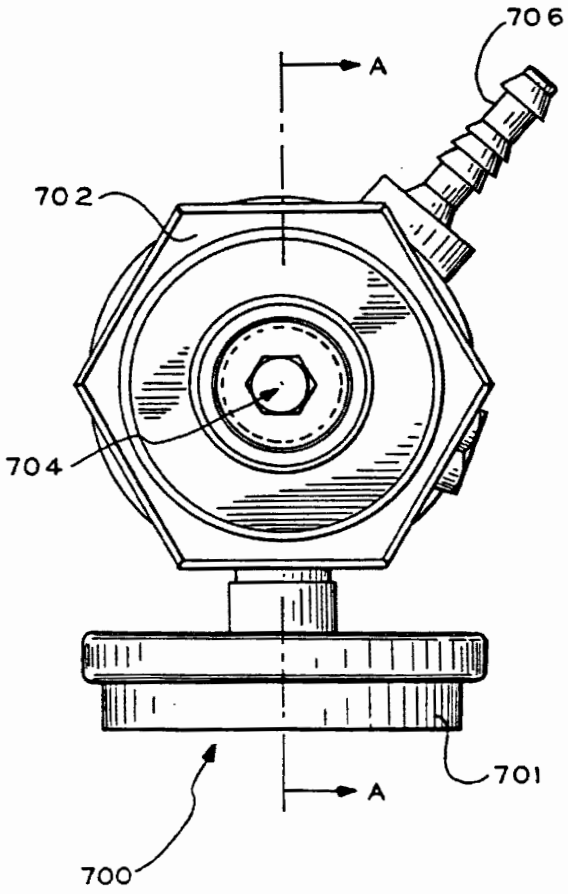
FIG. 8






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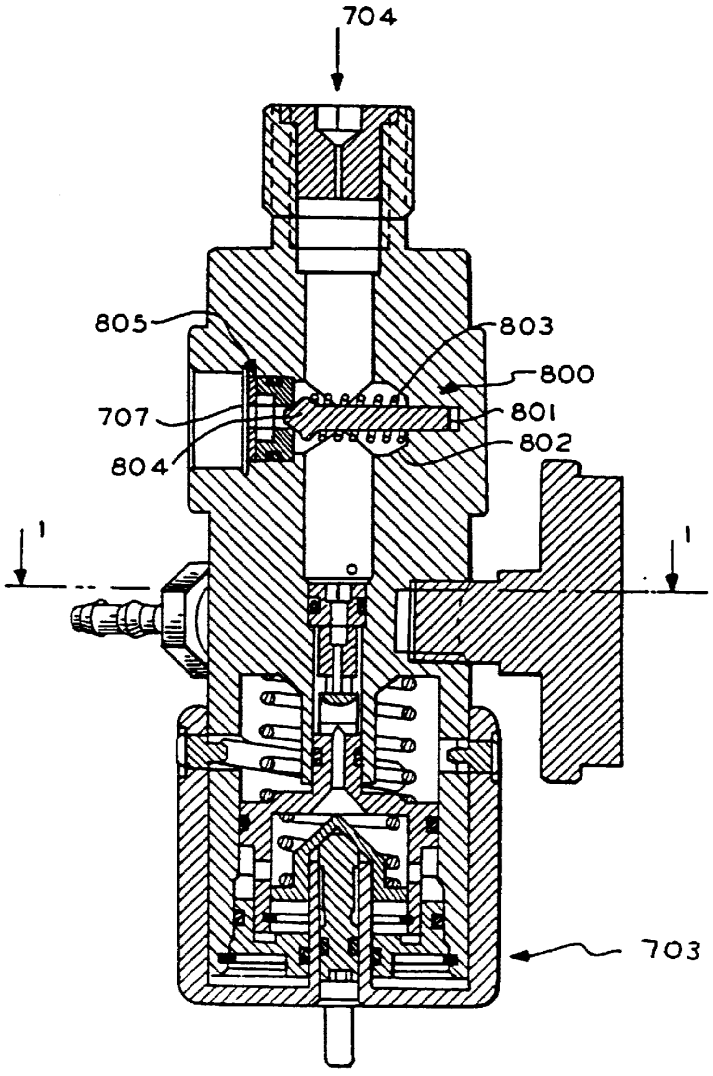
FIG. 9






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FIG. 10






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FIG. 11A

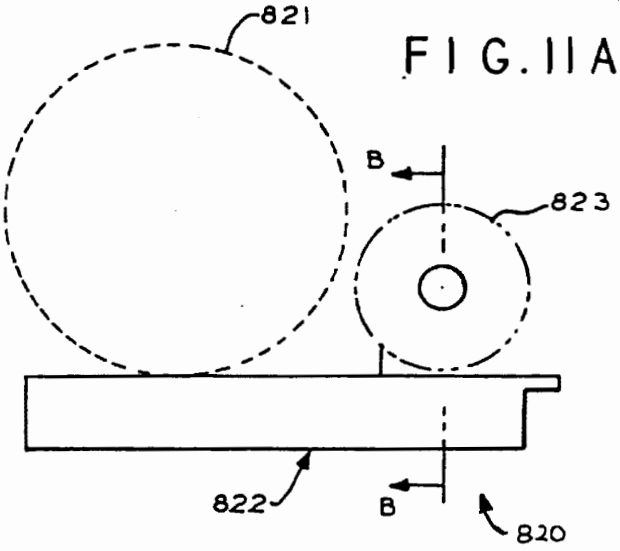
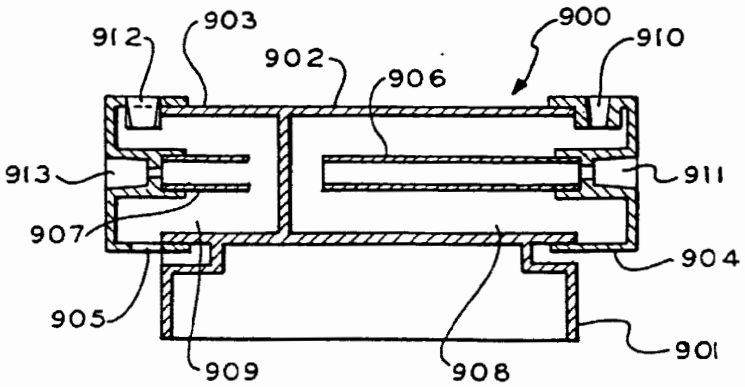



FIG. 11B






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## (54) Pressure swing adsorption process and apparatus

(57) A process and apparatus for separating the components of a gas mixture in a pair of adsorption vessels using a single gas compressor/ pump to move gas into and out of the adsorption vessels A and B. The cycle is such that the gas compressor/pump P is in continuous operation. The adsorption cycle is non-symmetrical in that the series of steps carried out in one of the adsorbers is not the same as the series of steps carried out in the other adsorber. The apparatus includes an intermediate gas storage container or reservoir C which is used to temporarily store gas removed from the non-adsorbed gas outlet end of the adsorption vessels A and B so that it can be used to partially pressurize the adsorption vessels A and B upon completion of the adsorbent regeneration step of the adsorption cycle.

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## Description

This invention relates to a process and apparatus for the separation of the components of a gas mixture, and more particularly to the separation of gas components by a novel pressure swing adsorption process. The invention is especially well adapted to the separation of the components of air at pressures near atmospheric pressure.

Pressure swing adsorption (PSA) processes are becoming increasingly more popular for the separation of gas mixtures containing two or more components at least one component of which is more strongly adsorbed by a selected adsorbent at elevated pressures than is at least one other component. The most basic processes comprise a cycle carried out in closed elongate vessels which contain a selected adsorbent and which have a feed gas inlet and a nonadsorbed product gas outlet. The cycle includes an adsorption step, during which a gas mixture is passed through one or more of the closed vessels in a cocurrent direction (from the feed gas inlet end towards the nonadsorbed product gas outlet end) at an elevated pressure, thereby causing the more strongly adsorbed component to be selectively adsorbed and the less strongly adsorbed to be discharged from the adsorption vessel as nonadsorbed product gas; and an adsorbent regeneration step, during which process gas in the adsorption vessel is countercurrently (in the direction opposite to the cocurrent direction) removed therefrom, thereby reducing the pressure in the adsorption vessel and causing the more strongly adsorbed component to be desorbed from the adsorbent. The desorbed gas is discharged through the inlet end of the vessel and discarded as a waste gas or collected as a second product gas.

Over the years since the discovery of the basic adsorption cycle, various steps have been added to the cycle to improve the yield or purity of the gas product(s) of the process. Thus, to conserve energy and improve yield, bed equalization steps have been added to cycles in which two or more adsorption vessels are operated in parallel and out of phase, such that one or more vessels is in the adsorption mode while one or more other vessels are in the bed regeneration mode. Bed equalization comprises transferring gas from a vessel of the system which has just completed its adsorption step and contains gas at an elevated pressure to another vessel of the system which has just completed its bed regeneration step and is at a low pressure. A product backfill step has likewise been added to enhance the purity of the nonadsorbed product gas. During the product backfill step nonadsorbed product gas is passed countercurrently through the adsorption vessel, which causes strongly adsorbed gas to be forced toward the feed gas inlet end of the vessel. This minimizes or eliminates adulteration of the nonadsorbed product gas during the early stages of the following adsorption step of the cycle.

US-A-5,122,164, discloses a two bed PSA process having a cycle which features a purge step, in addition to a bed equalization step with optional bed evacuation and a product backfill step. The disclosure of this patent is incorporated herein by reference.

US-A-5,536,299, discloses a PSA process for gas separation in which the steps of cycle overlap the succeeding steps of the cycle. It is asserted that the disclosed cycle results in increased capacity and reduced power consumption.

US-A-5,565,018, shows the production of oxygen from air using segregated external gas storage tanks.

The above processes include steps in which the feed gas compressor and the vacuum pump are not operated; accordingly these equipment units must be shut down or idled when they are not in use.

Efforts are continuously being made to improve the efficiency of PSA cycles by increasing the yield or enhancing the purity of the desired product or by reducing energy consumption during the process. Thus, US-A-5,370,728, discloses a single adsorption vessel system which uses a single gas compressor to both charge feed gas into the adsorption vessel and to evacuate the vessel during bed regeneration.

It would be highly desirable to have a plural bed process which operates more cost effectively and more efficiently. The invention disclosed herein accomplishes these objectives.

The present invention derives energy savings benefit from the use of a temporary storage tank to receive partially purified gas cocurrently discharged from the adsorption vessels of the system at pressures intermediate the nonadsorbed gas product pressure and the final evacuation pressure, and makes use of the gas contained in the temporary storage tank as a purge gas and as a partial repressurization gas for the adsorption vessels. In the case of an adsorption system comprising pairs of adsorption vessels, capital cost economy and additional energy economy are realized by the use of a single gas compressor/pump to both pressurize and evacuate the adsorption vessels of the system in a cycle which permits the compressor/pump to be in continuous service during the adsorption cycle.

According to the present invention there is provided a process for producing first component-enriched gas from a feed gas containing first and second components in a system comprising at least one gas reservoir and at least two adsorption zones (in regions), each of the adsorption zones containing an adsorbent which selectively adsorbs the second component, comprising repeatedly performing the steps:

(i) producing first component-enriched gas in at least one adsorption zone of the system by cocurrently introducing feed gas thereinto and cocurrently removing first component-enriched gas therefrom, while partially depressurizing at least one other adsorption zone of the system by cocurrently removing gas therefrom and storing the removed gas in the gas reservoir;



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(ii) partially depressurizing at least one adsorption zone by cocurrently transferring gas therefrom to the gas reservoir, while countercurrently removing second component-enriched gas from at least one other adsorption zone;

(iii) purging at least one adsorption zone of the system by transferring gas cocurrently out of at least one other adsorption zone and countercurrently into the adsorption zone(s) being purged in this step (iii) while countercurrently withdrawing gas from the at least one adsorption zone(s) being purged;

(iv) countercurrently removing second component-enriched gas from at least one adsorption zone, while partially pressurizing at least one other adsorption zone by countercurrently transferring gas thereinto from the gas reservoir;

(v) purging at least one adsorption zone of the system other than the adsorption zone(s) purged in step (iii) by countercurrently transferring gas thereinto from the gas reservoir and countercurrently removing second component-enriched gas from the at least one adsorption zone being purged in this step (v), while further pressurizing the adsorption zone(s) partially pressurized in step (iv) by cocurrently introducing feed gas thereinto;

(vi) partially pressurizing at least one adsorption zone of the system by countercurrently transferring gas thereinto from the gas reservoir, while producing first component-enriched gas by cocurrently introducing feed gas into at least one other adsorption zone other than the adsorption zone(s) in which first component-enriched gas was produced in step (i), and cocurrently removing first component-enriched gas therefrom; and

(vii) further pressurizing the adsorption zone(s) partially pressurized in step (vi) by cocurrently introducing feed gas thereinto, while continuing to produce first component-enriched gas in the adsorption zone(s) in which first component-enriched gas was produced in step (vi) by cocurrently introducing feed gas thereinto and cocurrently removing first component-enriched gas therefrom.

In a preferred aspect of the process according to the invention, the process further comprises, between steps (i) and (ii), the additional step of continuing to produce cocurrently produce first component-enriched product gas in the at least one adsorption zone referred to in step (i), while countercurrently removing second component-enriched gas from the at least one other adsorption zone referred to in step (i).

In a more preferred aspect of the process according to the invention, the process additionally comprises, between steps (iv) and (v), the additional step of countercurrently removing second component-enriched gas from the at least one adsorption zone referred to in step (iv), while partially pressurizing the at least one other adsorption zone referred to in step (iv) by countercurrently introducing first component-enriched product gas thereinto, and between steps (vi) and (vii) the additional step of partially pressurizing the at least one adsorption zone referred to in step (vi) by countercurrently transferring first component-enriched product gas thereinto, while continuing to produce first component-enriched product gas in the at least one other adsorption zone referred to in step (vi) and cocurrently remove first component-enriched gas therefrom.

The process of the invention can be carried out in one or more pairs of adsorption zones or in three or more adsorption zones operated serially.

A preferred embodiment of the invention comprises carrying out the process of the invention in a system comprising at least one gas reservoir and one or more pairs of first and second adsorption zones, and with respect to each such pair of first and second adsorption zones, the steps of the process are:

(i) producing first component-enriched product gas by cocurrently introducing feed gas into the first adsorption zone and cocurrently removing first component-enriched gas therefrom, while partially depressurizing the second adsorption zone by cocurrently transferring gas therefrom into storage in the gas reservoir or at least one of the gas reservoirs;

(ii) partially depressurizing the first adsorption zone by cocurrently transferring gas therefrom to said gas reservoir storage, while countercurrently removing second component-enriched gas from the second adsorption zone;

(iii) purging the second adsorption zone by transferring gas cocurrently out of the first adsorption zone and countercurrently into the second adsorption zone while countercurrently removing second component-enriched gas therefrom;

(iv) countercurrently removing second component-enriched gas from the first adsorption zone, while partially pressurizing the second adsorption zone by countercurrently transferring gas thereinto from said gas reservoir stage;



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(v) purging the first adsorption zone by countercurrently transferring gas thereinto from said gas reservoir storage and countercurrently removing gas from the first adsorption zone, while further pressurizing the second adsorption zone by cocurrently introducing feed gas thereinto;

(vi) partially pressurizing the first adsorption zone by countercurrently transferring gas thereinto from said gas reservoir storage, while producing first component-enriched product gas by cocurrently introducing feed gas into the second adsorption zone and cocurrently removing first component-enriched gas therefrom; and

(vii) further pressurizing the first adsorption zone by cocurrently introducing feed gas thereinto, while continuing to produce first component-enriched product gas by cocurrently introducing feed gas into the second adsorption zone and cocurrently removing first component-enriched gas therefrom.

Preferably, if there are one or more pairs of adsorption zones, the process further comprises, between steps (i) and (ii) the additional step of producing first component-enriched product gas by cocurrently introducing feed gas into the first adsorption zone and cocurrently removing first component-enriched gas therefrom, while countercurrently removing second component-enriched gas from the second adsorption zone. In a most preferred aspect, the process additionally comprises between steps (iv) and (v) the additional step of countercurrently removing second component-enriched gas from the first adsorption zone, while partially pressurizing the second adsorption zone by countercurrently transferring first component-enriched product gas thereinto, and between steps (vi) and (vii) the additional step of partially pressurizing the first adsorption zone by countercurrently transferring first component-enriched product gas thereinto, while producing first component-enriched product gas by cocurrently introducing feed gas into the second adsorption zone and cocurrently removing first component-enriched gas therefrom.

In the embodiment carried out in pairs of adsorption vessels, the feed gas is generally introduced into the second adsorption zone in step (v) and into the first adsorption zone in step (vii) at a first predetermined pressure, and the feed gas is generally introduced into the first adsorption zone in step (i) and into the second adsorption zone in steps (vi) and (vii) at a pressure which increases from the first predetermined pressure to a second predetermined pressure. The first predetermined pressure is preferably in the range of about 0.6 to about 2 bara, the second predetermined pressure is preferably in the range of about 1 to about 5 bara, and the pressure in the second adsorption zone during at least part of steps (ii) and (iii) and in the first adsorption zone during at least part of steps (iv) and (v) is preferably in the range of about 0.1 to about 1 bara. The first predetermined pressure is more preferably in the range of about 0.8 to about 1.2 bara, the second predetermined pressure is more preferably in the range of about 1 to about 2 bara, and the pressure in the second adsorption zone during at least part of steps (ii) and (iii) and in the first adsorption zone during at least part of steps (iv) and (v) is more preferably in the range of about 0.2 to about 0.8 bara.

In another preferred aspect of using pairs of adsorption zones, all steps of the process are carried out using a single gas pumping means. In one version of this aspect, the gas pumping means is used to introduce feed gas into the first adsorption zone during step (i) and into the second adsorption zone during steps (vi) and (vii) and is used to remove second component-enriched gas from the second adsorption zone during steps (ii) and (iii) and from the first adsorption zone during steps (iv) and (v). In another version of this aspect, the gas pumping means is used to remove first component-enriched gas from the first adsorption zone during step (i) and from the second adsorption zone during steps (vi) and (vii), and is used to remove second component-enriched gas from the second adsorption zone during steps (ii) and (iii) and from the first adsorption zone during steps (iv) and (v).

In any embodiment of the invention each adsorption zone may contain different adsorbents. Also, in any embodiment, the feed gas is preferably air, and when the feed gas is air, the second component is preferably nitrogen.

In another preferred aspect of the invention, the system in which the process is carried out has one pair of adsorption zones and one pair of gas reservoirs. In this aspect, the gas transferred cocurrently out of the second adsorption zone during step (i) is charged into a first gas reservoir; the gas transferred cocurrently out of the first adsorption zone during step (ii) is charged into a second gas reservoir; the gas countercurrently transferred to the second adsorption zone during step (iv) is removed from one of the first and second gas reservoirs; and the gas countercurrently transferred to the first adsorption zone during steps (v) and (vi) is removed from the other of the first and second gas reservoirs.

The invention also includes an apparatus embodiment which comprises a system for adsorptively fractionating a feed gas comprising:

(a) a pair of adsorption zones each having an inlet and outlet and each containing an adsorbent which selectively adsorbs the same component or components of the feed gas;
(b) a gas storage container;
(c) gas pumping means having an inlet and an outlet;
(d) adsorption zone exhaust gas conduit means connecting the inlet of each adsorption zone to the gas pumping



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means inlet, the adsorption zone exhaust gas conduit means including valve means to cut off communication between either or both of the adsorption zone inlets and the gas pumping means inlet;

(e) first feed gas conduit means with valve means connecting a feed gas source to the gas pumping means inlet;

(f) second feed gas conduit means with valve means connecting the feed gas source to each adsorption zone inlet;

(g) adsorption zone feed conduit means connecting the outlet of the gas pumping means to the inlet of each adsorption zone, the adsorption zone feed conduit means including valve means to cut off communication between the gas pumping means outlet and either or both of the adsorption zone inlets;

(h) exhaust gas discharge conduit means with valve means connecting the gas pumping means outlet to an exhaust gas discharge point;

(i) product gas conduit means connecting the outlet of each adsorption zone to a product gas discharge point, the product gas conduit means including valve means to cut off communication between either or both of the adsorption zone outlets and the product gas discharge point;

(j) cross-connection conduit means with valve means connecting the outlets of the adsorption zones; and

(k) storage conduit means connecting the outlet of each adsorption zone to the gas storage container, the gas storage conduit means including valve means to cut off communication between either or both of the adsorption zone outlets and the gas storage container.

In a preferred aspect, the apparatus further comprises a second product gas conduit means connecting the outlet of each adsorption zone to the gas pumping means inlet, the second product gas conduit means including valve means to cut off communication between either or both of the adsorption zone inlets and the gas pumping means inlet; and product gas discharge conduit means connecting the gas pumping means outlet to the product gas discharge point, the exhaust gas discharge conduit including valve means. In another preferred aspect, the feed gas is ambient air and the adsorption zones contain adsorbent which selectively adsorbs nitrogen from air.

In a preferred embodiment, the first and second adsorption zones contain different adsorbents.

In another preferred embodiment, the system is provided with first and second gas storage containers; first intermediate storage conduit means connecting the outlet of the first adsorption zone to the first intermediate gas storage container, the first intermediate gas storage means conduit means including valve means to cut off communication between either or both of the adsorption zone outlets and the first intermediate gas storage container; and second intermediate storage conduit means connecting the outlet of the second adsorption zone to the second intermediate gas storage container.

The process and system according to the invention will now be described by way of example with reference to the accompanying drawings, in which:

Figure 1 is a schematic representation of an adsorption system that can be used in one embodiment of the invention;

Figures 2A - 2J are schematic representations of the steps of a preferred adsorption cycle carried out in the adsorption system of Figure. 1 using a single compressor/pump to charge feed gas into the adsorption vessels and to withdraw sorbed gas from these vessels; and

Figure 3A - 3J are schematic representations of the steps of a preferred adsorption cycle carried out in the adsorption system of Figure. 1 using a single compressor/pump to withdraw nonadsorbed product gas from the adsorption vessels and to withdraw sorbed gas from these vessels.

Figures 4A - 4J are schematic representations of the steps of a preferred adsorption cycle carried out in the adsorption system of Figure 1 using a single compressor/pump to charge feed gas into the adsorption vessels and to withdraw sorbed gas from these vessels and using a pair of tanks to hold intermediate storage gas;

The same reference numerals and letters are used to represent the same or similar parts in the various drawings. Auxiliary equipment, including compressors, heat exchangers and valves, not necessary for an understanding of the invention, have been omitted from the drawings to simplify discussion of the invention.

Referring to Figure 1, there is illustrated therein an adsorption system comprising a pair of adsorption vessels, A and B, arranged in parallel, gas storage container C and gas compressor/pump P, and piping with valves for transporting the various gas streams through the system. Vessels A and B may be the same size or vessel B may be smaller or larger than vessel A. Both of vessels A and B are packed with an adsorbent which selectively adsorbs one or more gas components of the feed gas to the system more readily than it adsorbs one or more other components of the feed gas. Vessels A and B may be packed with the same adsorbent or they may be packed with different adsorbents.

Gas storage tank C is a conventional gas storage vessel; it may be packed with an adsorbent, if desired.



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Pump P may be any gas compressor, blower or, pump, that is capable of pumping gas into vessels and removing gas from the vessels at the various pressures encountered in the process. Gas pump A preferably operates efficiently at both superatmospheric and subatmospheric pressures since, in preferred embodiments of the invention, it will be used to introduce feed gas into the system at atmospheric or above-atmospheric pressures as well as evacuate vessels A and B during adsorbent regeneration.

In the system of Figure 1, gas feed line 2, fitted with valve 4, connects a source of feed gas to the inlet end of pump P. Waste gas discharge line 6, provided with valve 8, connects the outlet end of pump P to a waste gas discharge point. Upstream of valve 8, line 10, provided with valve 12, connects line 6 to adsorption vessel inlet manifold 14. Manifold 14 is connected to adsorption vessel inlet lines 16 and 18 via valves 20 and 22. Lines 24 and 26, provided with valves 28 and 30, respectively, join the nonadsorbed gas outlets of vessels A and B to nonadsorbed product gas discharge line 32. Cross-connection line 34 joins lines 24 and 26 via valves 36 and 38, respectively. Intermediate storage tank supply line 40, provided with valve 42 connects tank C to line 34 at a point between valves 36 and 38. Lines 16 and 18 connect the inlet ends of vessels A and B to evacuation manifold 44 via valves 46 and 48, respectively. Manifold 44 is connected to feed line 2 via evacuation line 50, fitted with valve 52. Pump bypass waste gas line 54, provided with valve 56, connects manifold 44 to waste gas discharge line 6 downstream of valve 8. Pump bypass feed gas line 58 connects line 2 to line 10 via valve 60 and to line 54, upstream of valve 56, via valve 62.

The process of the invention will be described as it applies to the separation of air to produce an oxygen-enriched nonadsorbed gas product in the system illustrated in Figure 1 according to the cycle illustrated in Figures 2A - 2J. The steps illustrated in Figures 2B, 2F and 2I are optional, but they will be included in the described cycle since they are included in the most efficient aspect of the process of the invention. In this embodiment, vessels A and B are packed with a nitrogen-selective adsorbent, such as type A or type X zeolite. The vessels may contain a first bed of desiccant, such as activated alumina or silica gel to remove moisture from the air. Carbon dioxide may also be removed from the air by the desiccant, if it is activated alumina; otherwise, it will generally be adsorbed in the initial portion of the main adsorbent, since it is more strongly adsorbed by the above adsorbents than is nitrogen. Pump P is in service during each step of the cycle. The pressures and step durations stated below are merely exemplary for operation of the described adsorption system to separate air into oxygen-enriched and oxygen-depleted components by the disclosed process. It should be understood, however, that these values are not to be construed as imitative.

During step 1 (Figure 2A), valves 4,12, 20, 38 and 42 are initially open and all other valves are closed. Ambient air at atmospheric pressure is drawn into the system through line 2 by pump P and charged into vessel A, thereby causing the pressure to increase in vessel A. When vessel A is pressurized to the desired adsorption pressure, generally in the range of about 1 to about 1.6 bara, valve 28 is opened and oxygen-enriched gas product passes out of the system through lines 24 and 32. The oxygen product gas is generally stored in a storage vessel, from which product is drawn as needed. During this step, vessel B, which has just completed an oxygen-enriched gas production step, undergoes its first depressurization step, reducing the pressure in vessel B to about 1 to about 2 bara. Also, during this step void space gas is cocurrently discharged from vessel B to intermediate storage vessel C. The duration of this step is generally in the range of about 3 to about 10 seconds.

At the completion of step 1, optional step 2 (Figure 2B) is started, during which valves 38 and 42 are closed and valves 48 and 56 are opened. In this step the cocurrent production of oxygen-enriched gas is continued in vessel A at the desired adsorption pressure while vessel B undergoes its second depressurization step, during which gas contained in vessel B is countercurrently vented to the atmosphere through lines 18, 54 and 6, thereby further reducing the pressure in vessel B to about 1 to about 1.8 bara. This atmospheric vent step permits pump P to continue to be used to force feed air into vessel A. The duration of this step is generally in the range of 0 to about 10 seconds.

When the step 2 is completed, step 3 (Figure 2C) is initiated, during which valves 8, 36, 42, 48 and 52 are open and all other valves are closed. Vessel A now undergoes its first depressurization step with the cocurrent discharge of void space gas from this vessel to tank C through lines 24, 34 and 40, reducing the pressure in vessel A to about 1 to about 2 bara. Meanwhile, vessel B undergoes its third depressurization to a pressure in the range of about 1.2 to about 0.2 bara, by countercurrent evacuation of gas through lines 18, 50, 2 and 6, using pump P. The duration of this step is generally in the range of about 3 to about 15 seconds.

The next step of the process is step 4 (Figure 2D). For this step, valves 8, 36, 38, 48 and 52 are open and all other valves are closed. Vessel A now undergoes it second depressurization to a pressure of about 0.8 to about 1.8 bara, and vessel B undergoes purge-evacuation by flowing gas cocurrently out of vessel A and countercurrently into and through vessel B, while gas continues to be withdrawn countercurrently from vessel B and discharged to the environment until the pressure in vessel B is reduced to about 0.2 to about 1.0 bara. This step completes the regeneration of adsorbent contained in vessel B. The duration of this step is generally in the range of about 4 to about 15 seconds.

The next step of the process is step 5 (illustrated in Fig 2E). During this step vessel A undergoes its first countercurrent evacuation of the cycle, to a pressure of about 0.2 bara to about 1.2 bara, using pump P, and vessel B undergoes its first partial pressurization step by flowing gas from storage vessel C countercurrently into vessel B until the pressure in vessel B is in the range of about 0.3 to about 1.0 bara. Only valves 8, 42, 38, 46 and 52 are opened during



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this step. Gas is evacuated from vessel A through lines 16, 50 and 2 by means of pump P, and is discharged to the environment through line 6. The duration of this step is generally in the range of about 3 to about 15 seconds.

The next step of the process, step 6 (Figure 2F), is optional. During this step, when employed, valves 8, 30, 46 and 52 are open and all other valves are closed. Vessel A continues to be countercurrently evacuated by pump P through lines 16, 50, 2 and 6, to a pressure of about 0.2 to about 1.0 bara, while vessel B undergoes its second partial pressurization to a pressure of about 0.4 to about 1.0 bara, by the countercurrent flow of oxygen-enriched product gas through lines 32 and 26 and into vessel B. The duration of this step is generally in the range of 0 to about 15 seconds.

During the next step of the process, step 7 (Figure 2G), only valves 8, 22, 36, 42, 46, 52 and 60 are open. Vessel A is purged by the flow of gas from tank C and countercurrently into vessel A accompanied by the countercurrent evacuation of gas from vessel by pump P through lines 16, 50, 2 and 6 to a final pressure of about 0.2 to about 1.0 bara, while vessel B receives its third partial pressurization of the process by the introduction of ambient air at atmospheric pressure cocurrently into this vessel through lines 2, 58, the tail end of line 10, and lines 14 and 18. The duration of this step is generally in the range of about 4 to about 15 seconds.

The eighth step of the cycle is shown in Figure 2H. During this step vessel A receives its first partial repressurization of the cycle, while vessel B undergoes its final repressurization and initiation of production of oxygen-enriched gas product. Generally, for this step, only valves 4, 12, 22, 36 and 42 are initially open, and vessel A is pressurized to a pressure of about 0.3 to about 1.0 bara by the countercurrent flow of gas from storage vessel C through lines 40, 34 and 24, while the pressure in vessel B is raised to the desired adsorption pressure (1 to about 1.6 bara) by cocurrently pumping ambient air thereinto through lines 2, 10, 14 and 18 by means of pump P. When the pressure in vessel B reaches the desired adsorption pressure, valve 30 is opened and oxygen-enriched product gas flows to product storage through line 32. Vessel A continues to receive gas from tank C. The step generally lasts for a period in the range of about 3 to about 15 seconds.

Step 9, illustrated in Figure 2I, is the third optional step of the process. During this step, vessel A is further pressurized by flowing oxygen-enriched gas thereinto, while vessel B is in production service. Valves 4, 12, 22, 28 and 30 are the only valves open during this step. Oxygen-enriched product gas flows countercurrently into vessel A through line 24 until the pressure in vessel A reaches about 0.4 to about 1.0 bara, while feed air continues to be charged cocurrently into vessel B at the desired adsorption pressure using pump P. The duration of this step is generally in the range of 0 to about 10 seconds.

The tenth and last step of the cycle is the step illustrated in Figure 2J. During this step, vessel A is further pressurized with ambient air while vessel B continues to be used to produce oxygen-enriched product gas at the desired adsorption pressure. For this step, valves 4, 12, 22, 30, 46 and 62 are open and all other valves are closed. Feed air at ambient pressure flows cocurrently into vessel A through lines 2, 58, 44 and 16, while feed air is charged currently into vessel B at the desired adsorption pressure (about 1.0 to about 2 bara) through lines 2, 6, 10, 14 and 18 and oxygen-enriched gas is discharged cocurrently from vessel B and sent to product storage through lines 26 and 32. The duration of this step is generally in the range of about 4 to about 10 seconds.

The above process has a number of unusual features. First, the process is not symmetrical, i.e. the set of steps conducted in vessel A is not identical to the set of steps carried out in vessel B. Partly because of this, nonadsorbed product gas is not continuously produced. In the process illustrated in Figures 2A - 2J, nonadsorbed product gas is produced only in the steps 1, 2, 8, 9 and 10. Secondly, the steps of the process are tailored to provide a highly energy-efficient cycle. Thus, in steps 7 and 10 ambient air is introduced into the adsorption vessels without using pump P, and in step 2, vessel B is vented without the aid of pump P. This permits pump P to be used for other operations during these steps. A third feature of importance is the continuous use of pump P during the process. Because pump P is used in each step of the process it is never idled. This provides a considerable energy savings. In this respect it is noteworthy that pump P is used to pump feed gas into the adsorption vessels during steps 1, 2, 8, 9 and 10, and it is used to evacuate the adsorption vessels during the steps 3, 4, 5, 6 and 7.

The process carried out in steps 1 to 10 of the Figure 3 series of steps is identical to that carried out in steps 1 to 10 of Figure 2, except that in the Figure 3 system, pump P is used to withdraw oxygen-enriched gas from adsorber A during steps 1 and 2 and from adsorber B during steps 8, 9 and 10, while in steps 1, 2, 8, 9 and 10 of the Figure 2 series of steps, pump P is used to charge fresh feed into the respective adsorbers. The result is the same except that a lower pressure will be maintained in vessel A during steps 1 and 2 and in vessel B during steps 8, 9 and 10 than will be experienced in these adsorbers during the corresponding steps of the Figure 2 series of steps.

Figure 4 illustrates a system similar to the system of Figure 2, except that two intermediate storage tanks are used in place of the single tank used in the Figure 2 system. The system of Figure 4 is particularly useful when the adsorption system is comprised of two adsorbers, such as the system illustrated in Figure 1, and when it is desired to introduce the gas into the storage tank in such a manner that little or no mixing occurs. This occurs, for example, when the gas is introduced into the storage tank under plug flow conditions, such as when the storage tank is baffled with walled partitions that are parallel to the direction of flow of the gas into and out of the storage tank. The advantage of plug flow operation during transfer of gas to and from the storage tank is that the gas that is first removed from the nonadsorbed outlet end



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of the adsorption vessels will be most pure in the nonadsorbed gas, and this gas will be returned to the adsorption vessels last. Accordingly, the gas most pure in nonadsorbed gas will always be closest to the nonadsorbed gas outlet end of the adsorption vessels; thus the nonadsorbed product gas removed from the adsorption vessels during the production step will always be of the highest purity with respect to the nonadsorbed gas component.

In the embodiment shown in Figure 4, tank C is used for the flow of gas to and from vessel A and tank D is used for the flow of gas to and from vessel B. It is not necessary, however, that the gas being returned to the adsorption vessels be returned to the vessel from which it was taken. For example, gas stored in tank C from vessel A in step 3 (Figure 4C) can be returned to vessel B in the step 5 (Figure 4E), and the gas stored in vessel D in step 1 (Figure 4A) can be returned to vessel A in steps 7 and 8 (Figures 4G and 4H).

It will be appreciated that it is within the scope of the present invention to utilize conventional equipment to monitor and automatically regulate the flow of gases within the system so that it can be fully automated to run continuously in an efficient manner.

The invention is further illustrated by the following example in which, unless otherwise indicated, parts, percentages and ratios are on a volume basis.

**EXAMPLE 1**

This example illustrates use of the process of the invention to separate air into an oxygen-enriched gas product and an oxygen-depleted gas product using the sequence of steps shown in Figure 2. The example was carried out using two identical cylindrical adsorption vessels 3 inches in diameter and 33 inches long. The adsorption vessels were packed with type X zeolite which had a silicon-to-aluminum atomic ratio of about 1 and which was exchanged with lithium and rare earth ions (described in detail in US-A-5,464,467). The system was operated to produce an oxygen-enriched gas product containing 90% by volume oxygen. The step durations, in seconds, and the step end pressures, in bar, absolute (bara), are stated in the Table. Steps 1 to 10 correspond to the steps illustrated Figures 2A to 2J, respectively.

TABLE

| Step | Duration, sec | End Pressure Bed A, bara | End Pressure Bed B, bara |
|---|---|---|---|
| 1 | 6 | 1.30 | 1.22 |
| 2 | 4 | 1.54 | 1.05 |
| 3 | 10 | 1.23 | 0.58 |
| 4 | 9 | 1.00 | 0.55 |
| 5 | 7 | 0.61 | 0.72 |
| 6 | 3 | 0.56 | 0.84 |
| 7 | 9 | 0.57 | 1.00 |
| 8 | 3 | 0.75 | 1.17 |
| 9 | 3 | 0.81 | 1.38 |
| 10 | 6 | 1.00 | 1.57 |

The specific product obtained in the above experiment was 43 $Nm^3/Hr/m^3$ (normal cubic meters of nonadsorbed product gas per hour per cubic meter of adsorbent). The yield (defined as the total number of moles of oxygen contained in the nonadsorbed product gas divided by the total number of moles of oxygen contained in the feed gas) was 58%. The calculated power consumption was 0.334 $Kwh/Nm^3O_2$ (kilowatt hours per normal cubic meter of oxygen-enriched gas produced).

**EXAMPLE 2**

The procedure of Example 1 was repeated using the conventional 10-step cycle illustrated in Figure 4 of US-A-5,122,164 with a 60 sec. cycle and a pressure swing between 1.5 bara and 0.49 bara. The specific product was 43 $Nm^3Hr/m^3$, the yield was 58% and the calculated power consumption was 0.376 $Kwh/Nm^3O_2$.

A comparison of Examples 1 and 2 shows that the cycle illustrated in Figure 2 provides substantially the same yield



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at a considerable savings in power consumption relative to the yield and power consumption obtained using the ten-step cycle described in US-A-5,122,164.

Although the invention has been described with particular reference to specific equipment arrangements and to specific experiments, these features are merely exemplary of the invention and variations are contemplated. For example, the order of certain of the steps can be reversed. Thus, the steps illustrated in Figures 2F, 3F and 4F can precede the steps illustrated in Figures 2E, 3E and 4E, respectively or they can follow the steps shown in Figures 2J, 3J and 4J, respectively, and the steps illustrated in Figures 2I, 3I and 4I can precede the steps illustrated in Figures 2H, 3H and 4H, respectively or they can follow the steps shown in Figures 2J, 3J and 4J, respectively. Furthermore, the activity taking place in one or both adsorbers during one step may overlap with the activity taking place in one or both adsorbers during the following step. For example, the partial pressurization of vessel A with gas stored in tank C, shown in Figures 2H, 3H and 4H, may partially coincide with the vessel A product backfill step shown in Figures 2I, 3I and 4I, respectively.

**Claims**

1. A process for producing first component-enriched gas from a feed gas containing first and second components in a system comprising at least one gas reservoir and at least two adsorption zones, each adsorbent zone containing an adsorbent which selectively adsorbs said second component, comprising repeatedly performing the steps:

   (i) producing first component-enriched gas in at least one of said adsorption zones by cocurrently introducing feed gas thereinto and cocurrently removing first component-enriched gas therefrom, while partially depressurizing at least another of said adsorption zones by cocurrently transferring gas therefrom and into said gas reservoir;

   (ii) partially depressurizing at least one of said adsorption zones by cocurrently transferring gas therefrom and into said gas reservoir, while countercurrently removing second component-enriched gas from at least another of said adsorption zones;

   (iii) purging at least one of said adsorption zones by transferring gas cocurrently out of at least another adsorption zone and countercurrently into said one of said adsorption zones of this step (iii) while countercurrently withdrawing gas therefrom;

   (iv) countercurrently removing second component-enriched gas from at least one of said adsorption zones, while partially pressurizing at least another of said adsorption zones by countercurrently transferring gas thereinto from said gas reservoir;

   (v) purging at least one of said adsorption zones other than the said adsorption zone purged in step (iii) by countercurrently transferring gas thereinto from said gas reservoir and countercurrently removing second component-enriched gas from said one of said adsorption zones of this step (v), while further pressurizing the said adsorption zone partially pressurized in step (iv) by cocurrently introducing feed gas thereinto;

   (vi) partially pressurizing at least one of said adsorption zones by countercurrently transferring gas thereinto from said gas reservoir, while producing first component-enriched gas by cocurrently introducing feed gas into at least another of said adsorption zones other than said one adsorption zone of step (i) and cocurrently removing first component-enriched gas therefrom; and

   (vii) further pressurizing the said adsorption zone partially pressurized in step (vi) by cocurrently introducing feed gas thereinto, while continuing to cocurrently introduce feed gas into said another of said adsorption zones referred to in step (vi) and cocurrently removing first component-enriched gas therefrom.

2. A process according to claim 1, wherein said system comprises at least one gas reservoir and one or more pairs of first and second adsorption zones, and, with respect to the or each pair of first and second adsorption zones, the steps are:

   (i) producing first component-enriched product gas by cocurrently introducing feed gas into said first adsorption zone and cocurrently removing first component-enriched gas therefrom, while partially depressurizing said second adsorption zone by cocurrently transferring gas therefrom and into storage in the gas reservoir or at least one of the gas reservoirs;



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(ii) partially depressurizing said first adsorption zone by cocurrently transferring gas therefrom and into said gas reservoir storage, while countercurrently removing second component-enriched gas from said second adsorption zone;

(iii) purging said second adsorption zone by transferring gas cocurrently out of said first adsorption zone and countercurrently into said second adsorption zone while countercurrently removing second component-enriched gas from said second adsorption zone;

(iv) countercurrently removing second component-enriched gas from said first adsorption zone, while partially pressurizing said second adsorption zone by countercurrently transferring gas thereinto from said gas reservoir storage;

(v) purging said first adsorption zone by countercurrently transferring gas thereinto from said gas reservoir storage and countercurrently removing gas from said first adsorption zone; while further pressurizing said second adsorption zone by cocurrently introducing feed gas thereinto;

(vi) partially pressurizing said first adsorption zone by countercurrently transferring gas thereinto from said gas reservoir storage, while producing first component-enriched product gas by cocurrently introducing feed gas into said second adsorption zone and cocurrently removing first component-enriched gas therefrom; and

(vii) further pressurizing said first adsorption zone by cocurrently introducing feed gas thereinto, while continuing to produce first component-enriched product gas by cocurrently introducing feed gas into said second adsorption zone and cocurrently removing first component-enriched gas therefrom.

**3.** A process according to claim 2, wherein the feed gas is introduced into said second adsorption zone in step (v) and into said first adsorption zone in step (vii) at a first predetermined pressure and the feed gas is introduced into said first adsorption zone in step (i) and into said second adsorption zone in steps (vi) and (vii) at a pressure which increases from said first predetermined pressure to a second predetermined pressure.

**4.** A process according to claim 3, wherein said first predetermined pressure is in the range of 0.6 to 2 bara and said second predetermined pressure is in the range of 1 to 5 bara.

**5.** A process according to claim 4, wherein said first predetermined pressure is in the range of 0.8 to 1.2 bara and said second predetermined pressure is in the range of 1 to 2 bara.

**6.** A process according to claim 4 or claim 5, wherein the pressure in said second adsorption zone during at least part of steps (ii) and (iii) and in said first adsorption zone during at least part of steps (iv) and (v) is in the range of 0.1 to 1 bara.

**7.** A process according to any one of claims 2 to 6, wherein:

steps (i) to (vii) are carried out using a single gas pumping means;

said gas pumping means is used to introduce feed gas into said first adsorption zone during step (i) and into said second adsorption zone during steps (vi) and (vii) and is used to remove second component-enriched gas from said second adsorption zone during steps (ii) and (iii) and from said first adsorption zone during steps (iv) and (v); and

said gas pumping means is used to remove first component-enriched gas from said first adsorption zone during step (i) and from said second adsorption zone during steps (vi) and (vii) and is used to remove second component-enriched gas from said second adsorption zone during steps (ii) and (iii) and from said first adsorption zone during steps (iv) and (v).

**8.** A process according to any one of claims 2 to 7, wherein:

said system has one pair of adsorption zones; and

the gas transferred cocurrently out of said second adsorption zone during step (i) is charged into a first gas res-



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ervoir; the gas transferred cocurrently out of said first adsorption zone during step (ii) is charged into a second gas reservoir; the gas countercurrently transferred to said second adsorption zone during step (iv) is removed from one of said first and second gas reservoirs; and the gas countercurrently transferred to said first adsorption zone during steps (v) and (vi) is removed from the other of said first and second gas reservoirs.

**9.** A process according to any one of claims 2 to 8, further comprising, between steps (i) and (ii) the additional step of producing first component-enriched product gas by cocurrently introducing feed gas into said first adsorption zone and cocurrently removing first component-enriched gas therefrom, while countercurrently removing second component-enriched gas from said second adsorption zones.

**10.** A process according to any one of claims 2 to 9, additionally comprising, between steps (iv) and (v), the additional step of countercurrently removing second component-enriched gas from said first adsorption zone, while partially pressurizing said second adsorption zones by countercurrently transferring first component-enriched product gas thereinto, and between steps (vi) and (vii), the additional step of partially pressurizing said one of said first and second adsorption zones of step (vi) by countercurrently transferring first component-enriched product gas thereinto, while producing first component-enriched product gas by cocurrently introducing feed gas into said second adsorption zone and cocurrently removing first component-enriched gas therefrom.

**11.** A process according to any one of the preceding claims, wherein the feed gas is air, and said second component is nitrogen.

**12.** A system or apparatus for fractionating a feed gas comprising:

(a) a pair of adsorption zones each having an inlet and outlet and containing an adsorbent which selectively adsorbs one component of said feed gas;
(b) a gas storage container;
(c) gas pumping means having an inlet and an outlet;
(d) adsorption zone exhaust gas conduit means connecting the inlet of each adsorption zone to said gas pumping means inlet, said adsorption zone exhaust gas conduit means including valve means to cut off communication between either or both of said adsorption zone inlets and said gas pumping means inlet;
(e) first feed gas conduit means with valve means connecting a feed gas source and said gas pumping means inlet;
(f) second feed gas conduit means with valve means connecting said feed gas source and said adsorption zone inlets;
(g) adsorption zone feed conduit means connecting the outlet of said gas pumping means to the inlet of each adsorption zone, said adsorption zone feed conduit means including valve means to cut off communication between said gas pumping means outlet and either or both of said adsorption zone inlets;
(h) exhaust gas discharge conduit means with valve means connecting said gas pumping means outlet to an exhaust gas discharge point;
(i) product gas conduit means connecting the outlet of each adsorption zone to a product gas discharge point, said product gas conduit means including valve means to cut off communication between either or both of said adsorption zone outlets and said product gas discharge point;
(j) cross-connection conduit means with valve means connecting the outlets of said adsorption zones; and
(k) intermediate storage conduit means connecting the outlets of each adsorption zone to said gas storage container, said intermediate gas storage means conduit means including valve means to cut off communication between either or both of said adsorption zone outlets and said gas storage container.

**13.** A system or apparatus according to claim 12, further comprising:

(a) second product gas conduit means connecting the outlet of each adsorption zone to said gas pumping means inlet, said second product gas conduit means including valve means to cut off communication between either or both of said adsorption zone inlets and said gas pumping means inlet; and

(b) product gas discharge conduit means connecting said gas pumping means outlet to said product gas discharge point, said exhaust gas discharge conduit including valve means;

**14.** A system or apparatus according to claim 12 or claim 13, provided with first and second intermediate gas storage containers, first intermediate storage conduit means connecting the outlet of said first adsorption zone to said first



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intermediate gas storage container, said first intermediate gas storage means conduit means including valve means to cut off communication between either or both of said adsorption zone outlets and said first intermediate gas storage container, and second intermediate storage conduit means connecting the outlet of said second adsorption zone to said second intermediate gas storage container, said second intermediate gas storage means conduit means including valve means to cut off communication between either or both of said adsorption zone outlets and said second intermediate gas storage container.



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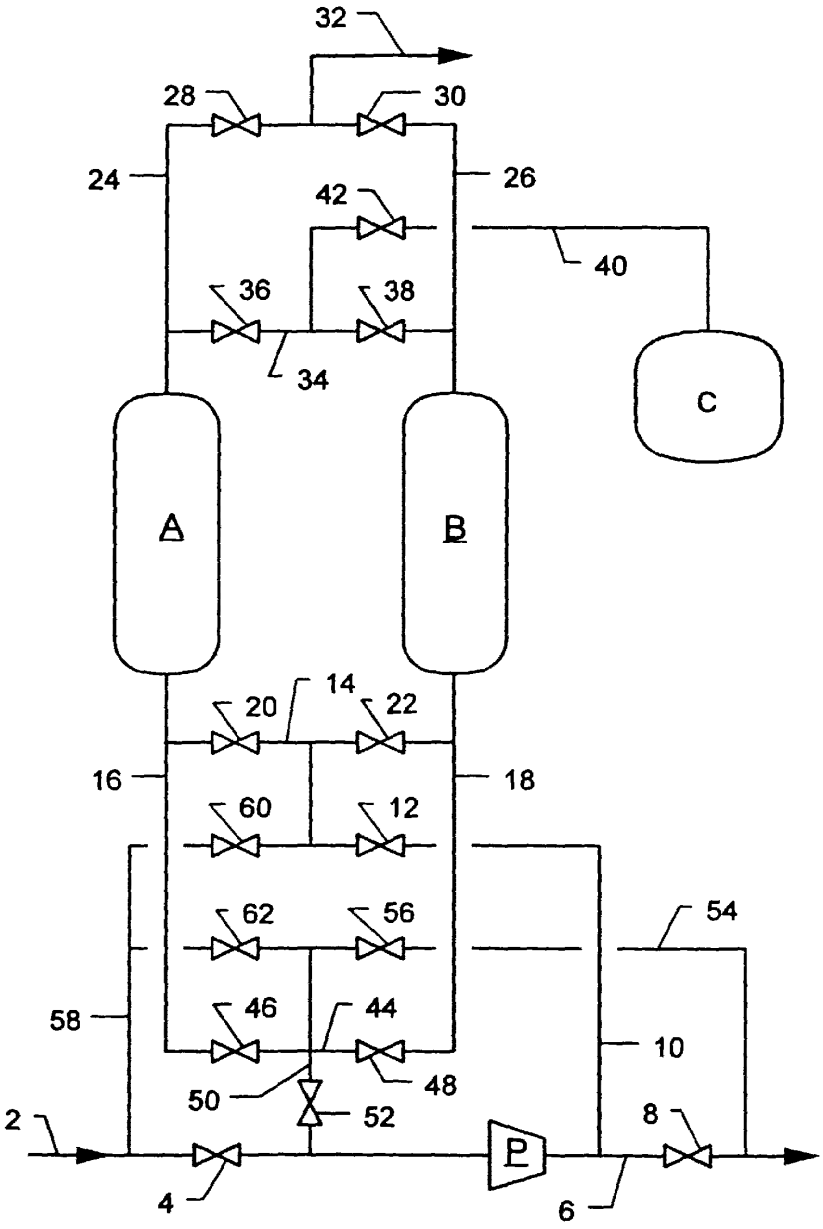



FIG. 1



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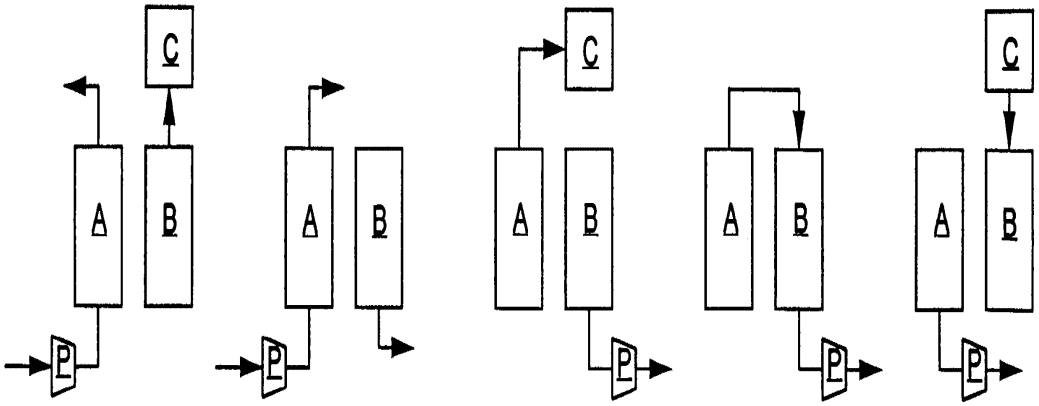



FIG. 2A FIG. 2B FIG. 2C FIG. 2D FIG. 2E

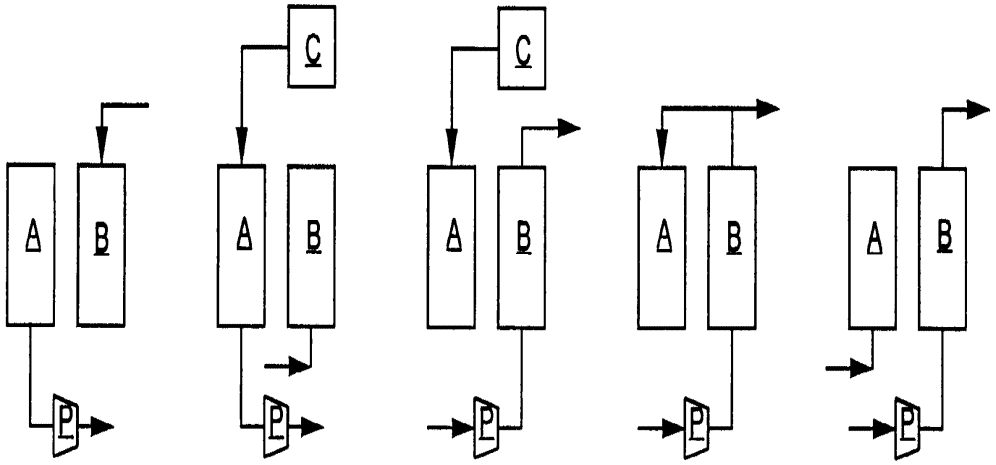



FIG. 2F FIG. 2G FIG. 2H FIG. 2I FIG. 2J



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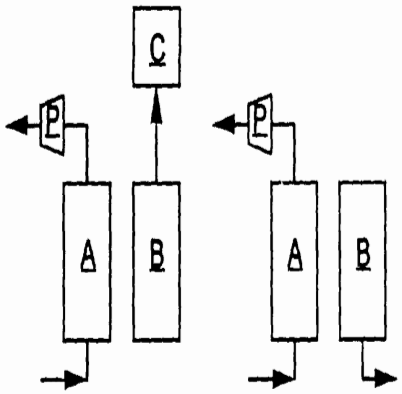



FIG. 3A




FIG. 3B

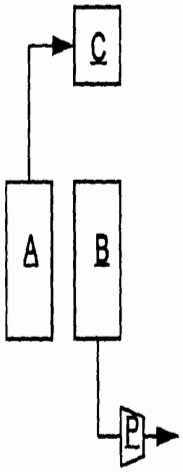



FIG. 3C

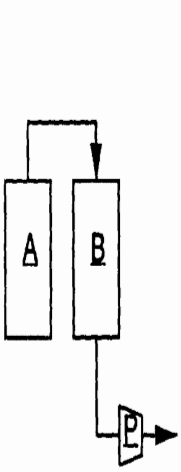



FIG. 3D

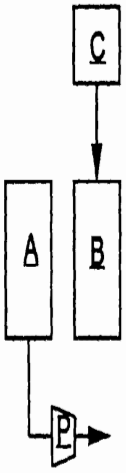



FIG. 3E

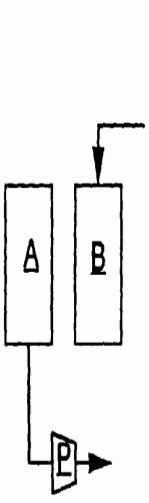



FIG. 3F

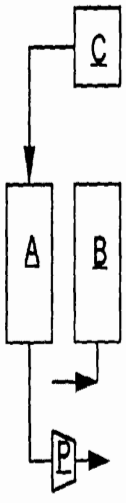



FIG. 3G

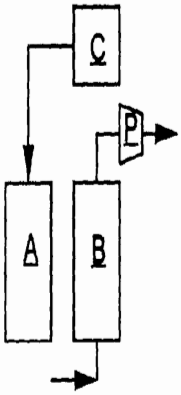



FIG. 3H

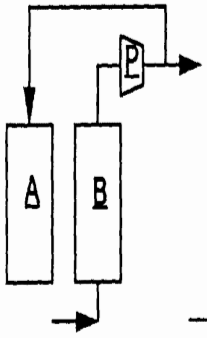



FIG. 3I

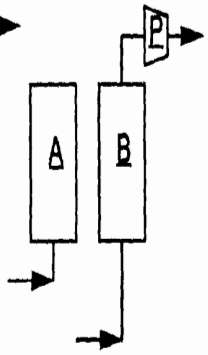



FIG. 3J



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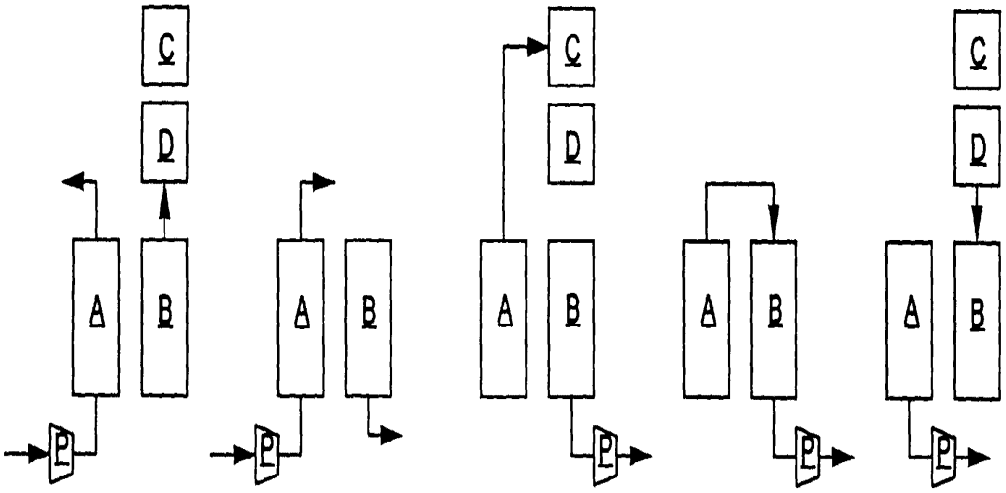



FIG. 4A FIG. 4B FIG. 4C FIG. 4D FIG. 4E

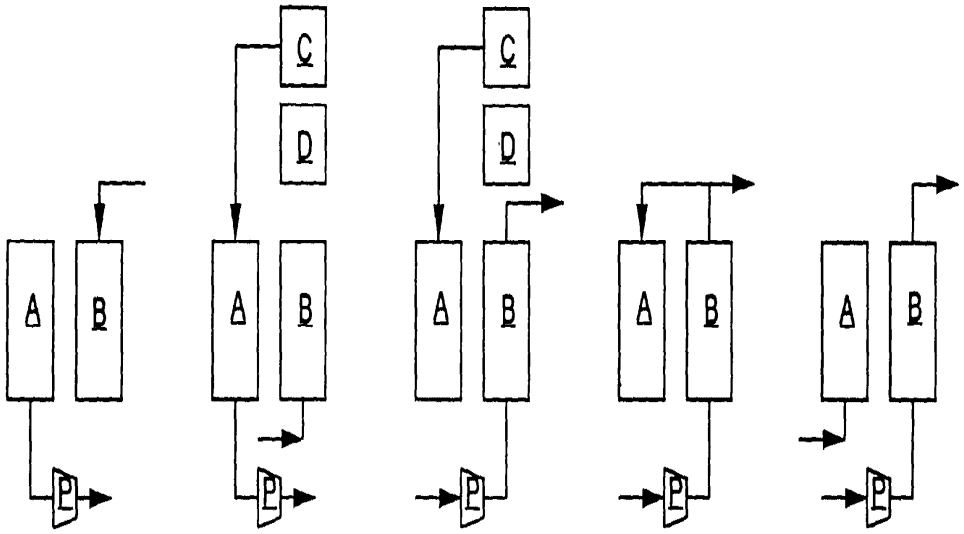



FIG. 4F FIG. 4G FIG. 4H FIG. 4I FIG. 4J



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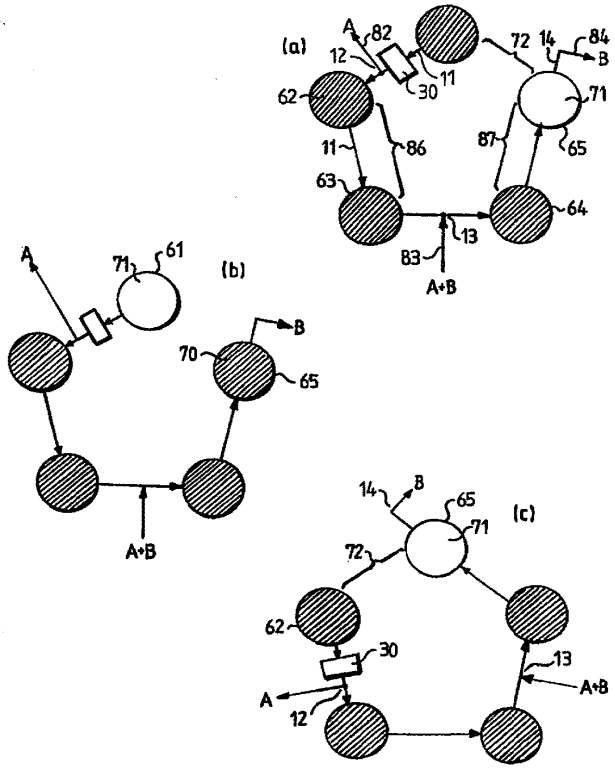
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(57) Abstract

A process for separating a mixture of gases by selective adsorption, in which pressure swing is used to induce both desorption and the flow of gas through at least one simulated moving chromatographic column.



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# GAS SEPARATION BY CONTINUOUS PRESSURE-SWING CHROMATOGRAPHY

## BACKGROUND OF THE INVENTION

This invention relates to separation of mixed gases. More particularly, this invention relates to methods and apparatus to separate gases by adsorption with improved performance and economy.

Cryogenic separation of mixed gases such as air provides a high degree of purity, such as nitrogen with less than 10 ppm oxygen as an impurity, with high efficiency and low operating costs. However the plant and equipment to cryogenically separate air is expensive and practical only on a very large scale

On smaller scale, processes based on adsorption can be attractive because of substantially lower capital investment. However adsorption processes for a separation have been much less efficient and more expensive to operate, especially if high product purity is an objective.

For example in generating high purity nitrogen from air by pressure swing adsorption (or "PSA", including the special case of "vacuum swing" adsorption, "VSA"), yield, i.e. nitrogen recovery from the feedstock air, is a small fraction of feedstock volume, but power consumption and operating cost are determined by the total amount of feedstock air processed.

This has been true even where multiple adsorption beds are used since, even with multiple beds, a substantial portion of the gas component desired as product is exhausted as impure waste when the beds are regenerated. In some cases the multiple beds constitute essentially identical and independent systems in parallel, operated in a phased manner to render the inherently "batch" process somewhat more continuous in product flow. Each bed contributes to capacity but process performance expressed as product purity and yield is not substantially greater with many beds than with few. The whole is not greater than the sum of the parts .

Examples include U.S. Patents No. 5,403,385, No. 5,354,346, No. 5,346,535, No. 5,261,947, No. 5,250,088, No. 5,246,676, No. 5,203,888, No. 5,176,722, No. 5,163,978, No. 5,122,164, No. 5,042,995, No. 5,002,591, No. 4,959,083, No. 4,954,146, No. 4,913,709, No. 4,816,039, No. 4,576,614, No. 4,522,637, No. 4,194,891 and 890, No. 4,168,149, No. 4,144,038 and 037, and No. 4,042,349.

U.S. Patent No. 4,834,780 describes a six-adsorber PSA process in which the six adsorbent beds operate substantially independently but staggered, with only one bed at a time in an adsorption mode.

Simulated moving bed (SMB) processes, in which a solid adsorbent is made to effectively move in the direction countercurrent to internal fluid flow, have also been used for separations in a form of continuous chromatography.

Examples are described in U.S. Patents No. 4,478,721, No. 4,498,991 and No. 5,387,347.

These examples are typical of many in that a circulating desorbent is required, to act as a carrier fluid for the components being separated and to regenerate the adsorbent. In principle the SMB processes can be applied to gases, and have been, but application has been limited.

The article entitled Counter-Current and Simulated Counter-Current Adsorption Separation Processes, by Ruthven and Ching, in Chemical Engineering Science (Vol. 44, No. 5, pp. 1011-1038, 1989) indicates that the desorption or regeneration step "...may involve either near-isothermal displacement or thermal swing operation in which the desorption section runs at elevated temperature." Examples are provided of SMB operation with gases such as pure nitrogen or isopropyl benzene vapor used, as a desorbent and carrier fluid, in the separation of gaseous components.

In the textbook entitled Gas Separation by Adsorption Processes, published by Butterworths, author Ralph T. Yang states, "The simulated moving-bed processes, under the general name of Sorbex, are restricted to liquids and apparently are not competitive with the pressure-swing adsorption process for gas-phase applications..."

Exceptions are cited, but the utility of SMB chromatography for separating gases has been limited by the need for a carrier and desorbent gas.

## SUMMARY OF THE INVENTION

An array of beds connected in series, packed with a pressure-sensitive selective adsorbent material, is operated in the manner of continuous simulated moving bed chromatography, with adsorbent effectively moving counter-current and upstream with respect to internal gas flow. However no desorbent is used. Instead, internal gas flow is manipulated such that the function of a desorbent and carrier fluid is simulated by an effective co-current flow of vacuum as a pseudo-fluid, so that gas is at least partially displaced by vacuum at the upstream end of the array of beds, and displaces vacuum at the downstream end of the array.

The array is organized to include at least one simulated moving chromatographic column, in the product enrichment zone between the feedstock inlet port and the primary product outlet port. Preferably the array includes two

simulated moving columns, one upstream and one downstream with respect to the feedstock inlet port.

Thus a method is provided, combining simulated moving bed chromatography -with pressure swing adsorption and referred to as pressure swing chromatography, for separating gases by selective adsorption, in which pressure swing is used to induce both desorption and the flow of gas through at least one simulated moving chromatographic column. The method provides high product purity combined with high efficiency, with product yield approaching 100%.

## BRIEF DESCRIPTION OF IHE DRAWINGS

FIG. 1, a, b and c, is a schematic diagram of a five-bed system with two simulated moving columns, illustrating gas and effective adsorbent flows.

FIG. 2 is a diagram showing typical axial raffinate concentration profiles within the extract enrichment column, at the beginning and end of a switching cycle, in a simulation of the preferred embodiment.

FIG. 3 is a Table showing a comparison of simulation results for topologically similar methods, with and without a moving extract enrichment column, and with varying size columns and process conditions.

## DETAILED DESCRIPTION AND PREFERRED EMBODIMENTS

The present invention is a method and associated apparatus for using a pressure-dependent selective gas adsorbing medium, such as those used in PSA or VSA gas separation systems, to make an efficient chromatographic separation of a mixture of gases. A mixed feed containing two components, denoted A and B, may be separated into two products; an extract product containing mainly A (the more strongly adsorbed species, or group of species with similarly strong adsorption properties), and a raffinate containing mainly B (the less strongly adsorbed species or group of species).

The method is inherently continuous in nature, though internally cyclical. Feedstock may be added and product withdrawn continuously except for the substantially instantaneous interval while switching valves are being opened and shut.

An internal gas flow is pumped through a plurality of beds in fluid communication in a series, each bed containing a pressure dependent adsorbent packing. At the beginning of a cycle all the beds, except the one in the most downstream position with respect to the internal gas flow, are at operating pressure at which the packing is an effective and selective adsorbent for the

mixture. The most downstream bed is at a lower pressure at which relatively little gas is adsorbed.

During the cycle:

- Gas is pumped from the most upstream bed in the series to generate the internal gas flow and displace gas through the series in the downstream direction.
- An extract product A is withdrawn as a first outlet flow of gas from a first outlet port in fluid communication with the most upstream bed in the series.
- A feedstock flow of mixture A and B is added to the series of beds through an inlet port located downstream from the first outlet port.
- A raffinate product B is withdrawn as a second outlet flow, from a second outlet port in fluid communication with the most downstream bed in the series.

By the end of a cycle, gas pressure in the most downstream bed is increased to operating pressure, while pressure in the most upstream bed is reduced to the lower pressure. At the end of a cycle, switching valves are used to switch the positions of ports and other bed connections, so that each bed effectively advances upstream with respect to the internal gas flow by one position, except for the most upstream bed, which is effectively moved to the most downstream position.

Both the most upstream and the most downstream beds are subject to pressure swings during the operating cycle; from operating to lower pressure and from lower to operating pressure, respectively. Other beds, between the most upstream and most downstream, are at operating pressure throughout the cycle.

The series includes at least one simulated moving chromatographic column, preferably located in a product enrichment zone, which is the region between the inlet port and primary product outlet port. The inlet port and the other outlet port are also separated, by at least one bed. Preferably the series includes two simulated moving columns; one upstream and one downstream of the inlet port so that the inlet port constitutes the boundary between the two simulated moving columns.

A simulated moving column must contain two or more beds. Periodically the most upstream bed of the column, which has been fully enriched or saturated, is removed from the column. It is replaced in its position by the other beds in the column advancing upstream by an increment of one bed, and the most downstream bed of the column is replaced in its position by a fresh bed.

This has the advantage that product can be drawn from only an optimal end portion of the column, for high purity, without losing the product in the remainder of the column. Effectively the product outlet port follows (if extract) or leads (if raffinate) the axial concentration wavefront in the column.

In contrast, if only one bed is present between the inlet and outlet port, it does not undergo simulated motion but is simply replaced when the beds are switched. If product is drawn only from the far end of the moving concentration wavefront, e.g. if beds are switched and the enrichment bed moved away when only a small portion of that wavefront is within it, to obtain high purity, then product yield will be extremely low and much product will be lost.

The significance of concentration wavefront position when the beds are switched is illustrated in FIG. 2, which is discussed in greater detail in connection with the preferred embodiment. The size of the upstream portion of the column which is removed when beds are switched, and of the increment of column motion, is the fraction 1/N of the column, where N is the number of beds in the column. The maximum increment of column motion that can be provided, that permits exploiting the effect of wavefront position, is 1/2 column. Simulations described below indicate that much more extreme product purity, with the same high yield, is obtainable if the increment of column motion is 1/3 column or less.

In accordance with the present invention, each additional bed necessarily becomes part of a simulated moving chromatographic column, increasing the number N of beds in that column and further reducing the fraction 1/N of the column that is removed when beds are switched. Therefore each additional bed increases product purity and yield as well as production capacity and, in contrast with prior art multiple bed systems that are essentially parallel in organization, the whole is greater than the sum of the parts.

If the configuration is to be optimized for a single product, it may sometimes be economical to locate all but one bed on one side, either upstream or downstream depending on which outlet flow is product, of the inlet port. Preferably, the more highly adsorbed species will be the desired product, which will then be drawn as extract.

For example in producing nitrogen from air, using a zeolite adsorbent which preferentially adsorbs nitrogen, it may be economical to position the inlet port with most beds upstream and only one downstream. This will lead to lower yield and higher operating costs than if a downstream moving column were provided, but with lower equipment cost due to one less bed required.

It is not necessary to completely evacuate the most upstream bed. However any residual gas left in that bed at the end of a cycle may reduce purity of the raffinate. Also, the residual gas will reduce the production rate of pure ex-

tract per unit volume of adsorbent. The level of the lower pressure is therefore a compromise, between the cost of drawing high vacuum and the cost incurred as a result of residual gas in the bed when switched.

The first outlet port may be in fluid communication with, and extract A drawn from, either end of the most upstream bed (or even, conceivably, a location within the bed). Similarly, raffinate B may be drawn from either end of the most downstream bed, though the downstream end is preferred.

Also, it should be understood that ports in communication with the series of beds may be in such communication through valves, controls, pumps or compressors and the like, as appropriate, to obtain the required process flows.

Operation of the process will be further understood by reference to FIG. 1 which shows a configuration with five beds, 61, 62, 63, 64 and 65, with three beds upstream and two beds downstream of inlet port 13. Beds 62 and 63 in FIG. la comprise a first simulated moving chromatographic column 86, and beds 64 and 65 comprise a second simulated moving chromatographic column 87.

FIG. la shows conditions at the beginning of a cycle. Beds 61 through 64 contain gas at relatively elevated operating pressure 70, indicated by cross-hatching. Bed 65 contains gas at relatively lower pressure 71. Bed 61 is in fluid communication with bed 62 through pump means 30 and conduit means 11. Bed 62 is in fluid communication with bed 63, 63 with 64, and 64 with 65. However bed 65 is not in fluid communication with bed 61, causing effective dead end 72 between those two beds.

During the cycle gas is displaced through the bed series by pump means 30, from bed 61 and toward bed 65. Meanwhile a flow 82 of extract product A is drawn from first outlet port 12, a flow 84 of raffinate product B is drawn from second outlet port 14, and a flow 83 of feedstock is added through inlet port 13.

Referring to FIG. 1 b., at the end of a cycle but before bed switching, bed 61 contains gas at relatively lower pressure 71 while bed 65 contains gas at operating pressure 70.

Well known switching valve means are then used to advance pump means 30, ports 12, 13 and 14, and dead end 72, by one bed following the direction of internal gas flow so that dead end 72, for example, is now between beds 61 and 62, illustrated in FIG. I.c. In this way, each bed has effectively advanced in the direction counter to internal gas flow. Bed 62, for example, which had been adjacent the most upstream bed, is now the most upstream bed. The bed containing relatively lower pressure 71 is again the most downstream bed, and initial cycle conditions are restored.

FIG. 2 shows the simulated axial oxygen concentration in the two beds that constitute the moving chromatographic column between ports 12 and 13, in the system illustrated schematically in FIG. 1 as applied to the production of nitrogen from air. Simulated process conditions are as indicated in the Table of FIG. 3.

The simulated bed switching interval is 24 seconds, which corresponds to a switching valve and overall system cycle time of two minutes. Power consumption is approximately 60 kW, which corresponds to unit power consumption of approximately 0.5 kw-hr/CCF product, with product at 1 bar. The adsorbent is a commercially available zeolite material with bulk density 37 lb/ft3 that typically adsorbs 0.5% to 1.5«o its weight in gas at atmospheric pressure; for the purpose of simulation, adsorbed gas is 0.5% of the zeolite weight.

Product nitrogen purity resulting from simulation of the preferred embodiment in these conditions is 99.997°/Ot which corresponds to 24 ppm of impurities. Simulated process conditions and results are summarized in the Table of FIG. 3, which compares the preferred embodiment to two alternatives; one without a moving chromatographic column, with only one bed in the product enrichment zone, and one with a larger column than the preferred embodiment.

Simulation results clearly illustrate that process performance, in this case product purity with yield invariant, correlates strongly with the presence and size of the moving column. These three simulations, all with 90% yield as a condition, are denoted Series A in the Table of FIG. 3.

The advantage of the simulated moving column is further clarified by simulating the system's response to a reduction of nitrogen yield to 86%, with all other process conditions held constant. These simulations are denoted Series B in the Table of FIG. 3.

Product purity in the preferred embodiment is increased to 99.9998 (2 ppm impurities). In both simulations which include a moving column, the slight yield reduction leads to improved product purity by an order of magnitude or more. However product purity from the system without a moving column increases only modestly. Much greater benefit accrues, when the concentration wavefront is allowed to move slightly further downstream before bed switching, if the product enrichment zone is a moving column.

Moreover the simulated moving column permits more complete purging. The initial condition of a bed moving into position between the product and inlet ports, from a former position downstream of the inlet port, will be about 95% nitrogen. With one bed between those two ports, it is purged once and then, at the end of a switching cycle, moved upstream to be stripped.

With a column of N beds, each of them is purged N times, each time with product gas of higher purity. However there is no increase in the amount of nitrogen used or needed for purging. Rather, the same purge gas is used N times, being each successive time less pure but substantially more relatively pure than the bed it is purging. Therefore, as the simulation illustrates in the relative performance of different configurations, the moving column yields a very substantial improvement in product purity, with a modest increase in equipment complexity and essentially no increase in operating cost.

The two-bed moving column has been described as preferred because it will serve in appropriate applications at very low cost and is the simplest embodiment for the purpose of explanation. The three-bed moving column is further preferred. It will efficiently yield nitrogen comparable to the highest purity cryogenically produced liquid, at a cost only marginally higher than the two-bed column and still very low compared with that of the cryogenic product.

The locations of compressors, vacuum pumps and the like may be varied as practical as long as the desired flows are obtained. Moreover, the drawing of outlet flows and addition of feedstock may be assisted by separate pump means and include accumulators as required, depending on the details of configuration.

An expander may be included to recover energy from gas that is, for example, being transferred from higher to lower pressure, e.g. into the most downstream bed. Also, provision may be made to heat or cool flowing gas as needed, to maintain a stable temperature in the beds.

Those beds which are at operating pressure need not all be at the same pressure, though having them all at substantially the same pressure leads to the simplest process and operation. Alternatively, higher purity may be obtained by maintaining gas velocity, instead of pressure, uniform over those beds that are at operating pressure, i.e. a high enough pressure for the adsorbent to be effective.

The amounts of extract A and raffinate B drawn from first and second outlet ports will be substantially similar to the amounts of A and B present in the feedstock. However these outlet stream amounts may be modified slightly to enhance performance. For example, making product streams A and B slightly smaller and larger, respectively, leads to higher purity of extract product stream A. This is the way the position of the concentration wavefront, at the time of bed switching, is controlled.

Extract and raffinate streams A and B may be drawn over the entire cycle or during only a portion of the cycle, such as at the beginning or end, for example to reduce power consumption or improve product purity. For example, refer-

ring again to FIG. 3, raffinate stream &4 may be drawn toward the end of the cycle, when the pressure in the most downstream bed 64 has been increased to operating pressure. However flow in the moving column should preferably be maintained continuous. The method is inherently continuous, though intermittent flows may be imposed by controls where advantageous.

The process as described is primarily and best suited for binary separations as has been illustrated. However in a manner analogous to practice in liquid separations, additional ports and beds may be provided where separation of more than two components is desired, e.g. as in the production of separate streams of sucrose, invert, and non-sugars, from cane molasses.

Performance of the invention has been described in connection with the separation of air to produce pure nitrogen. However it should be understood that the invention can be applied to separation of any gas mixture for which an appropriate selective adsorbent exists, such as, for example, oxygen and argon. In connection with separation of other gases; adsorbent selectivity differences may not be as great as between nitrogen and oxygen. Moving chromatographic columns of three or more beds may be necessary rather than preferred.

Air is a unique feedstock, too, in that it is free except for the cost of processing it and its components are environmentally acceptable. These characteristics of air provide design freedom that will not necessarily exist when separating other gases.

While the invention has been described in connection with the preferred embodiments, it is not intended to limit the invention to the particular forms set forth but, on the contrary, it is intended to cover such alternatives, modifications and equivalents as may be included within the spirit and scope of the invention as defined by the appended claims.

CLAIMS

1. A process for separating a mixture of gaseous components in which an internal gas flow is pumped through a plurality of beds in fluid communication in a series, each bed containing a pressure-dependent adsorbent packing, said mixture including a most highly adsorbed component and a less highly adsorbed component, said process being cyclical in nature such that:

(a) at the beginning of a cycle all of said beds except the one in a most downstream position with respect to said internal gas flow are at operating pressure at which said packing is an effective and selective adsorbent for said mixture, and said most downstream Position bed is at a lower pressure at which relatively little gas is adsorbed and,

(b) during said cycle, pumping gas from the most upstream of said beds to generate said internal gas flow and displace gas through said series in the downstream direction, withdrawing a first outlet flow of gas from a first outlet port in fluid communication with said most upstream bed, said first outlet flow consisting primarily of said most highly adsorbed component, and adding a feedstock flow of said mixture to said series of beds through an inlet port downstream from said first outlet port and separated from it by at least one bed, and withdrawing a second outlet flow from a second outlet port, said second outlet port being in fluid communication with the most downstream Positioned bed in said series and separated from said inlet port by at least one bed, said second outlet flow consisting primarily of said less highly adsorbed component, such that,

(c) by the end of said cycle the pressure in said most downstream Position bed is increased to said operating pressure while the pressure in said most upstream bed is reduced to said lower pressure and,

(d) at the end of said cycle, switching the positions of said ports among said plurality of beds such that each bed effectively advances upstream with respect to said internal gas flow by one position, except for said most upstream bed which is effectively moved to said most downstream position,

said series including at least one simulated moving chromatographic column, located between said inlet port and said first outlet port.

2. The process of claim 1 wherein said series includes two simulated moving chromatographic columns, one being upstream and the other downstream of said inlet port.

3. The process of claim 1 wherein said simulated moving chromatographic column moves in increments that do not exceed 1/3 of said column.

4. The process according to claim 1 wherein said highly adsorbed component is the primary product to be purified, said first outlet flow consists primarily of said primary product, and said simulated moving chromatographic column is upstream of said inlet port.

5. The process according to claim 1 wherein said mixture is air and said highly adsorbed component is nitrogen.

6. The process of claim 5 wherein said simulated moving chromatographic column moves in increments that do not exceed 1/3 of said column.

7. The process according to claim 1 wherein said mixture to be separated is a mixture primarily of oxygen and argon.

8. A process for separating a mixture of gases, including the steps of:
(a) inducing, by pressure swing, both desorption and the flow of gas through at least one simulated moving chromatographic column packed with an effective and selective adsorbent for said mixture,
(b) withdrawing a first outlet flow of gas from a first outlet port located upstream, with respect to said flow of gas, of an inlet port through which said mixture is admitted to said process, and
(c) withdrawing a second outlet flow of gas from a second outlet port located downstream of said inlet port,
wherein the most highly adsorbed component of said mixture is the primary product to be purified, said simulated moving chromatographic column is upstream of said inlet port, and said primary product is drawn from said process as said first outlet flow from a location upstream of said simulated moving chromatographic column.

9. The process according to claim 8 wherein said process is substantially continuous.

10. The process according to claim 8 wherein said simulated moving column moves in increments that do not exceed 1/3 of said column.

11. The process according to claim 8 wherein said flow of gas is induced through two simulated moving chromatographic columns in series.

12. The process according to claim 9 wherein said mixture is air.

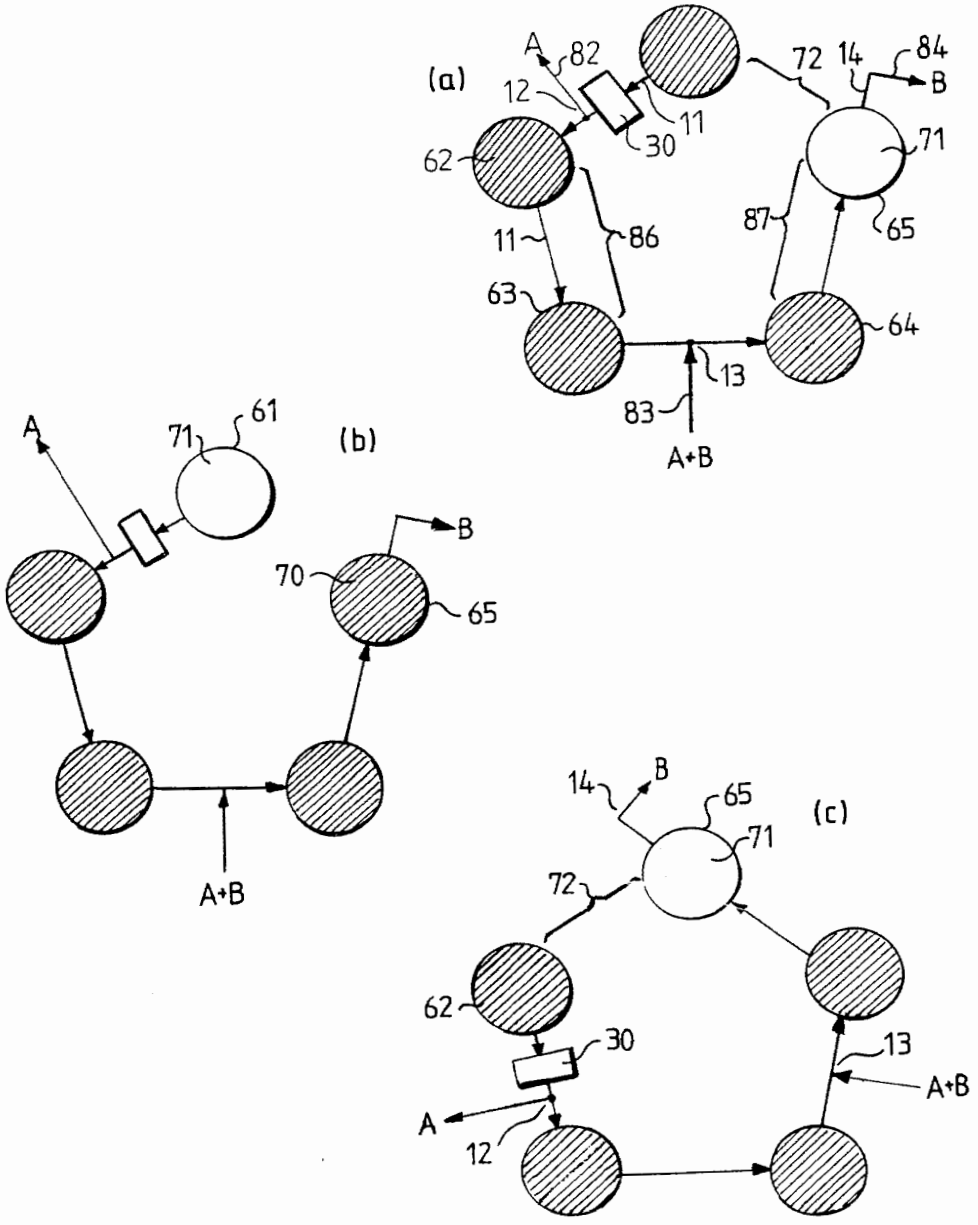
13. The process according to claim 10 wherein said mixture is air.

14. The process according to claim 11 wherein said mixture is air.

15. The process according to claim 11 wherein said mixture is primarily of oxygen and argon.

16. A process for separating a mixture of gases by selective adsorption, in which pressure swing is used to induce both desorption and the flow of gas through at least one simulated moving chromatographic column.

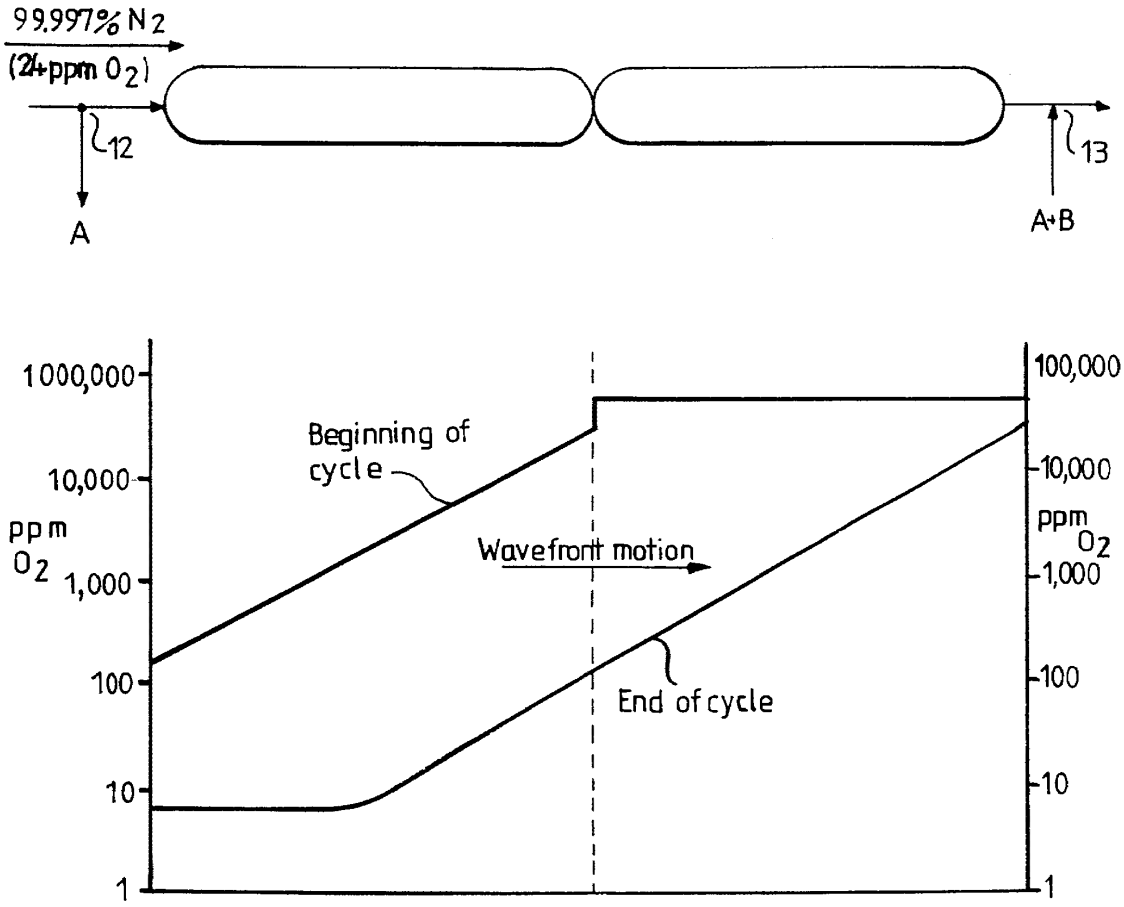
Fig.1






GCE - Exhibit 1071, Page 231
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

Fig. 2






GCE - Exhibit 1071, Page 232
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## Simulation Summary

### Simulated Conditions

| | | | | |
|---|---|---|---|---|
| Product | | 1 bed long, | 2 beds long, | 3 beds long, |
| Enrichment Zone | | bed replaced | moving colum | moving column |
| Total Beds in System | | 4 | 5 | 6 |
| Total Adsorbent Inventory | | 200 $ft^3$ | same | same |
| Feedstock Air Rate | | | 16,800 SCFH | same |
| same | | | | |
| Operating Pressure (Adsorption) | | 1.2 bar | same | same |
| Lower Pressure (Desorption) | | 0.065 bar | same | same |
| Bed Switching Interval | | 30 seconds | 24 seconds | 20 seconds |
| Overall System Cycle Time | | 2 minutes | same | same |
| Product Pressure | | 1 bar | same | same |
| Nitrogen | Series A | 12,000 SCFH | same | same |
| Production Rate | Series B | 11,500 SCFH | same | same |
| Nitrogen Yield | Series A | 90% | same | same |
| | Series B | 86% | same | same |

### Results

| | | | | |
|---|---|---|---|---|
| Product Purity | Series A | 99.7% | 99.997% | 99.9999% |
| and (ppm $O_2$) | | (3,000 ppm) | (24 ppm) | (0.15 ppm) |
| | Series B | 99.8% | 99.9998% | 99.9999998% |
| | | (1,700 ppm) | (2 ppm) | (2 ppb) |

Figure 3

# INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 97/01099

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: B01D 53/047, C01B 21/04
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)

IPC6: B01D, C01B

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

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

WPI,EPODOC

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| E,X | US 5672197 A (RONALD D. ROTCHILD), 30 Sept 1997 (30.09.97), column 2, line 63 - column 7, line 33, figures 1-3, claims 1-12 | 1-16 |
| A | EP 0681860 A2 (THE BOC GROUP, INC.), 15 November 1995 (15.11.95) | 1-16 |
| A | EP 0766989 A1 (AIR PRODUCTS AND CHEMICALS, INC.), 9 April 1997 (09.04.97) | 1-16 |
| A | US 5112367 A (CHARLES C. HILL), 12 May 1992 (12.05.92) | 1-16 |

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

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## INTERNATIONAL SEARCH REPORT

Information on patent family members

02/12/97

International application No.
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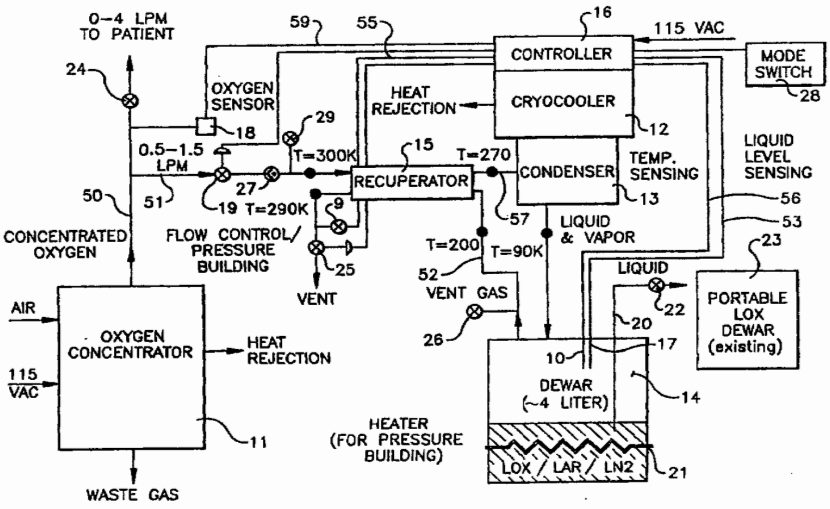
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**Published**
*With international search report.*

**(54) Title:** METHODS AND APPARATUS TO GENERATE LIQUID AMBULATORY OXYGEN FROM AN OXYGEN CONCENTRATOR




**(57) Abstract**

In one preferred embodiment, the present invention splits off some of the excess capacity gas flow from a gas concentrator (11) which is then stored (14) via liquefaction. The stored gas can then be used as a portable supply. A portion of the oxygen gas flow generated by the oxygen concentrator is channeled to a condenser (13) which receives and liquefies the oxygen gas using cryocooler (12). A storage dewar (14) is used for storing the oxygen liquefied by the condenser. Liquid is then selectively transferred to a smaller portable dewar (23). A controller can be used for monitoring the parameters of liquefaction, including oxygen concentration, the amount of liquid oxygen in the dewar, and for controlling the parameters of liquid oxygen generation and transfer.

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DESCRIPTION

# METHODS AND APPARATUS TO GENERATE LIQUID AMBULATORY OXYGEN FROM AN OXYGEN CONCENTRATOR

## Background of the Invention

The field of this invention relates to using an oxygen concentrator to create a portable supply of supplementary oxygen for ambulatory respiratory patients so that they can lead normal and productive lives -- as the typical primary oxygen sources are too bulky to carry or require excessive power to operate.

There is a burgeoning need for home and ambulatory oxygen. Supplemental oxygen is necessary for patients suffering from lung disorders; for example, pulmonary fibrosis, sarcoidosis, or occupational lung disease. For such patients, oxygen therapy is an increasingly beneficial, life-giving development. While not a cure for lung disease, supplemental oxygen increases blood oxygenation, which reverses hypoxemia. This therapy prevents long-term effects of oxygen deficiency on organ systems -- in particular, the heart, brain and kidneys. Oxygen treatment is also prescribed for Chronic Obstructive Pulmonary Disease (COPD), which afflicts about 25 million people in the U.S., and for other ailments that weaken the respiratory system, such as heart disease and AIDS. Supplemental oxygen therapy is also prescribed for asthma and emphysema.

The normal prescription for COPD patients requires supplemental oxygen flow via nasal cannula or mask twenty four hours per day. The average patient prescription is

two liters per minute of high concentration oxygen to increase the oxygen level of the total air inspired by the patient from the normal 21% to about 40%. While the average oxygen flow requirement is two liters per minute, the average oxygen concentrator has a capacity of four to six liters of oxygen per minute. This extra capacity is occasionally necessary for certain patients who have developed more severe problems but they are not generally able to leave the home (as ambulatory patients) and do not require a portable oxygen supply.

There are currently three modalities for supplemental medical oxygen: high pressure gas cylinders, cryogenic liquid in vacuum insulated containers or thermos bottles commonly called "dewars," and oxygen concentrators. Some patients require in-home oxygen only while others require in-home as well as ambulatory oxygen depending on their prescription. All three modalities are used for in-home use, although oxygen concentrators are preferred because they do not require dewar refilling or exchange of empty cylinders with full ones.

Only small high pressure gas bottles and small liquid dewars are portable enough to be used for ambulatory needs (outside the home). Either modality may be used for both in-home and ambulatory use or may be combined with an oxygen concentrator which would provide in-home use.

As we describe below, the above-described current methods and apparatus have proven cumbersome and unwieldy and there has been a long-felt need for improved means to supply the demand for portable/ambulatory oxygen.

For people who need to have oxygen but who need to operate away from an oxygen-generating or oxygen-storage source such as a stationary oxygen system (or even a portable system which cannot be easily carried), the two most prescribed options generally available to patients are: (a) to carry with them small cylinders typically in a wheeled stroller; and (b) to carry portable containers typically on a shoulder sling. Both these gaseous oxygen and liquid oxygen options have substantial drawbacks. But from a medical view, both have the ability to increase the productive life of a patient.

The major drawback of the gaseous oxygen option is that the small cylinders of gaseous oxygen can only provide gas for a short duration. Oxygen conserving devices that limit the flow of oxygen to the time of inhalation may be used. However, the conserving devices add to the cost of the service and providers have been reluctant to add it because there often is no health insurance reimbursement. Indeed, the insurance reimbursement for medical oxygen treatment appears to be shrinking.

Another drawback of the gaseous oxygen option is the source of or refill requirement for oxygen once the oxygen has been depleted from the cylinder. These small gas cylinders must be picked up and refilled by the home care provider at a specialized facility. This requires regular visits to a patient□s home by a provider and a substantial investment in small cylinders for the provider because so many are left at the patient□s home and refilling facility. Although it is technically possible to refill these cylinders in the patient□s home using a commercial

oxygen concentrator that extracts oxygen from the air, this task would typically require an on-site oxygen compressor to boost the output pressure of the concentrator to a high level in order to fill the cylinders. Additionally, attempting to compress the oxygen in pressurized canisters in the home is dangerous, especially for untrained people. This approach of course presents several safety concerns for in-home use. For example, in order to put enough of this gas in a portable container, it must typically be compressed to high pressure (~2000 psi). Compressing oxygen from 5 psi (the typical output of an oxygen concentrator) to 2000 psi will produce a large amount of heat. (Enough to raise the temperature 165□ C per stage based on three adiabatic compression stages with intercooling.) This heat, combined with the oxygen which becomes more reactive at higher pressures, sets up a potential combustion hazard in the compressor in the patient's home. Thus, utilizing and storing a high pressure gas system in the patient's home is dangerous and not a practical solution.

The convenience and safety issues are not the only drawbacks of this compressed oxygen approach. Another drawback is that the compressors or pressure boosters needed are costly because they require special care and materials needed for high pressure oxygen compatibility. For example, a Rix Industries, Benicia, CA, 1/3 hp unit costs about $10,000 while a Haskel International, Burbank, CA, air-powered booster costs about $2200 in addition to requiring a compressed air supply to drive it. Litton Industries and others also make oxygen pressure boosters.

Turning now to the liquid oxygen storage option, its main drawback is that it requires a base reservoir -- a stationary reservoir base unit about the size of a standard beer keg -- which has to be refilled about once a week. The liquid oxygen can then be obtained from a base unit and transferred to portable dewars which can be used by ambulatory patients. Also, with the liquid oxygen option, there is substantial waste, as a certain amount of oxygen is lost during the transfer to the portable containers and from evaporation. It is estimated that 20% of the entire contents of the base cylinder will be lost in the course of two weeks because of losses in transfer and normal evaporation. These units will typically boil dry over a period of 30 to 60 days even if no oxygen is withdrawn.

There are other complications. Typically, supplemental oxygen is supplied to the patient by a home care provider, in exchange for which it receives a fixed monetary payment from insurance companies or Medicare regardless of the modality. Oxygen concentrators for use in the home are preferred and are the least expensive option for the home care provider. For outside the home use however, only small high pressure gas bottles and small liquid dewars are portable enough to be used for ambulatory needs. One of these two modalities may be used for both in-home and ambulatory use or may be combined with an oxygen concentrator which would provide in-home use. In either case, the home care provider must make costly weekly or biweekly trips to the patient's home to replenish the oxygen. One of the objects of this invention is to eliminate these costly "milk runs."

Portable oxygen concentrators are commercially available for providing patients with gaseous oxygen. These devices are "portable" solely in the sense that they can be carried to another point of use such as in an automobile or in an airplane. At present, there are no home oxygen concentrators commercially available that can provide liquid oxygen. One type of medical oxygen concentrator takes in air and passes it through a molecular sieve bed, operating on a pressure swing adsorption cycle, which strips most of the nitrogen out, producing a stream of ~90% oxygen, for example, as shown in U.S. Patent Nos. 4,826,510 and 4,971,609 (which are incorporated herein by reference). While, as set out in the Information Disclosure Statement, complex oxygen liquefaction systems have been disclosed for use by the military in jet aircraft, and while large-scale commercial plants have been disclosed, this technology has not yet found its way into the home to help individual patients and to benefit the general public. A truly portable oxygen concentrator has not yet been perfected and this event is unlikely, at least in the near future, because the power requirements are too large to be provided by a lightweight battery pack.

Since liquid oxygen requires periodic delivery and home oxygen concentrators are not commercially available that would create liquid oxygen, there has existed a long-felt need for a device or method having the capability to concentrate oxygen from the air, liquefy it, and transfer it into portable dewars in a home environment, and for a home oxygen concentrator unit which allows excess flow capacity from the concentrator to be stored by either compression or liquefaction for later use.

Summary of Invention

The present invention presents a much safer and less expensive way of providing portable oxygen for patients who do not want to be tied to a stationary machine or restricted by present oxygen technology. In one preferred embodiment, the present invention splits off some of the excess capacity gas flow from a PSA (pressure swing adsorption) or membrane gas concentrator which has a relatively stable base load. This small portion of the excess flow capacity, about one liter per minute (□1LPM) is stored via liquefaction. The stored gas can then be used as a portable supply away from the location of the gas concentrator. The daily six hour range capacity for a two liter per minute patient can be accumulated by liquefying a one liter per minute gas flow for less than 24 hours. Therefore, the entire daily requirement for mobility can be produced every day if needed.

A summary of one of the many representative embodiments of the present invention is disclosed including a home liquid oxygen ambulatory system for supplying a portable supply of oxygen, where a portion of the gaseous oxygen output obtained from an oxygen concentrator is condensed into liquid oxygen, comprising: (a) an oxygen concentrator which separates oxygen gas from the ambient air; (b) an outlet flow line to transfer flow of oxygen gas from said oxygen concentrator for patient use; (c) a valve placed in the outlet flow line for splitting off a portion of the oxygen gas flow generated by the oxygen generator; (d) a generally vertically oriented, gravity assisted, condenser for receiving and liquefying the split off portion of the

oxygen gas flow; (e) a cryocooler associated with said condenser; (f) a first storage dewar in fluid communication with said condenser for storing the oxygen liquefied by the condenser, the first storage dewar having an outlet selectively engageable to and in fluid communication with at least one second smaller dewar and a fluid path for supplying liquid oxygen from the first dewar to the second dewar; (g) a heater for heating said first storage dewar; (h) a controller for monitoring (i) oxygen concentration of the oxygen gas flowing from said concentrator, and (j) the amount of liquid oxygen in said first dewar, and for controlling the parameters of liquid oxygen generation and transfer from said first storage dewar.

Another representative embodiment is the feature where the flow rate into the condenser is chosen to exceed the capacity of the condenser. In particular, only 20 to 90% of the incoming flow into the condenser is condensed to minimize the liquefaction of argon, nitrogen and trace gases, and to purge the system.

Additionally, the controller may control condenser parameters so that the condenser temperature varies in the range from approximately 69.2 to 109.7K, the condenser pressure varies from approximately 5 to 65 psia, and the concentrations of gas into the condenser varies with the oxygen range being 80 to 100%, the nitrogen range being 0 to 20%, and the argon range being 0 to 7%.

A unique condenser design is also disclosed where the condenser is in thermal contact with a cryocooler for use in liquefying oxygen and comprises: (a) an inlet conduit

for receiving oxygen; (b) an outer member; (c) an inner member; (d) a passage defined by said outer and inner members; (e) said inner member having radial slots to passages; (f) means for circulating said oxygen in said condenser.

Also disclosed is a representative method for controlling a home ambulatory liquid oxygen system comprised of an oxygen concentrator, a controller having a microprocessor, a condenser, a cryocooler and a storage dewar, where all or only a portion of the oxygen flow is utilized for liquefaction, comprising: (a) providing the microprocessor with a database and control functions; (b) sensing the parameters relating to the concentration and supply of gaseous oxygen, the level of liquid oxygen in the dewar, and the pressure of the condenser; (c) providing the microprocessor with these sensed parameters and having the microprocessor calculate optimal conditions; (d) controlling servomechanisms to regulate the system so that optimal conditions are realized as a function of said calculations.

The feature is also described wherein the liquid dewar will be periodically boiled dry to eliminate any small amounts of water and hydrocarbons that may pass through the gas concentrator.

The above-summarized apparatus and methods, more specifically set out in the claims, fill long-felt needs without posing any new safety issues to the patient in that, for example, there are no potentially dangerous canisters of high pressure compressed oxygen. The end result is that patients can ultimately use equipment with

which they are familiar. For example, patients on liquid oxygen currently perform liquid transfers from large (30-50 liquid liter) dewars to small (0.5-1.2 liquid liter -- corresponding to six hours of support) portable dewars. The present invention will provide a means of supplying ambulatory oxygen for a lower life cycle cost than the conventional method. Unlike industrial or military use liquefiers, which can take up whole rooms, the claimed oxygen liquefier (not including the oxygen concentrator) weighs less than 60 pounds and takes up less than six cubic feet of volume. There are currently about 700,000 patients in the United States using ambulatory oxygen with an average yearly cost of about $1,960 per patient. The estimated annual cost of oxygen per patient with the present invention is about $540.

Brief Description of the Drawings

Figure 1 is a block diagram of the invention in the medical oxygen preferred embodiment where gaseous oxygen is split off and liquefied for storage in a stationary dewar or container.

Figure 2 shows another preferred embodiment of the invention for ambulatory supplemental oxygen using a portable LOX dewar.

Figure 3 shows a typical temperature-composition diagram for oxygen-argon mixtures at typical dewar pressures.

Figure 4 shows a typical temperature-composition diagram for nitrogen-oxygen mixtures at typical dewar pressures.

Figure 5 shows typical oxygen concentration test data for gas streams into the condenser and out of the dewar during liquefaction, and out of the dewar as the

liquid is re-vaporized. (Oxygen source is a PSA oxygen concentrator.)

Figure 6 shows the controller block diagram for operation of the system.

Figures 7A through 7D are flow charts showing the controller logic.

Figure 8 shows the controller input levels and output states for the start-up mode of the preferred embodiment.

Figure 9 shows the controller input levels and output states for the condense mode of the preferred embodiment.

Figure 10 shows the controller input levels and output states for the transfer mode of the preferred embodiment.

Figure 11 shows the controller input levels and output state for the boil dry mode of the preferred embodiment.

Figure 12 illustrates basic components of a pulse tube refrigerator.

Figure 13 illustrates another embodiment of a cryocooler which may be used in the subject invention.

Figure 14 is a side cross-sectional view of the preferred embodiment of the condenser.

Figure 15 is an end cross-sectional view of the preferred embodiment of the condenser corresponding to Figure 14.

Figure 16 shows a side cross-sectional view of another preferred embodiment of the condenser.

Figure 17 shows an end cross-sectional view of another preferred embodiment of the condenser corresponding to Figure 16.

Figure 18 is an isometric view of the embodiment of the condenser shown in Figures 16 and 17.

Detailed Description of the Preferred Embodiment

A flow chart of the preferred embodiment of the invention is set out in Figure 1. Its main components include an oxygen concentrator 11, a cryocooler 12, a condenser 13, and a storage/collection dewar or vacuum insulated container 14. In the preferred embodiment, the oxygen concentrator 11 operates on a pressure swing adsorption cycle and essentially strips all or most of the nitrogen from air along with other minor components such as $H_2O$, $CO_2$, CO, $NO_x$, etc. The result is a stream of dry gas with high oxygen concentration (~92%) flowing in fluid outlet 50. A portion of the gas from this output in fluid outlet 50 is routed to a condenser 13 in association with a cryocooler (or cryogenic refrigerator) 12 through flow lines 51 and 57. The cryocooler provides cooling of the condenser heat exchanger 13 to liquefaction temperatures, causing the oxygen in contact therewith to go from the gaseous to the liquid phase. The condenser 13 typically must be insulated from ambient heating and may in practice even be located inside the dewar 14. In order to lessen the load on the cryocooler 12, a recuperator 15 may be used to pre-cool the incoming stream utilizing the vent flow through line 52 out of the dewar as a cooling medium. In practice, this recuperator 15 may also be located within the dewar 14 to reduce ambient heating.

Controller 16 may be equipped with a microprocessor, adequate memory, software and ancillary equipment comprising a computer which can be used to monitor and control the operation of the system. The controller 16 may be provided with signals from liquid level sensor 17, oxygen sensor 18, pressure transducer 9, and temperature sensor 10 via lines 53, 59, 55 and 56, respectively.

These signals are sensed and processed by the computer, with the controller operating valve 19, valve 25, heater 21, and cryocooler 12, in accordance with predetermined programs.

The controller also provides output indicators for the patient. The liquid level in the dewar is continuously displayed and the patient is alerted when the oxygen concentration is low and when the system is ready for them to transfer liquid to a portable dewar. A modem or wireless link may be included to enable remote monitoring of the key parameters of the system by the home care provider as well as information which is useful for repair, maintenance, billing, and statistical studies of patients for the medical oxygenation market. Key system parameters of interest include the number of liquid transfers performed, the oxygen concentration history, number of run hours on the cryocooler, and time of the last boil-dry as well as number of boil dries performed. The controller may include a computer and/or a microprocessor located either integrally with the liquefaction system claimed herein or remotely therefrom but in communication therewith using either a modem and telephone lines or with a wireless interface. The computer and/or microprocessor may include memory having a database, or may be remotely connected to a memory or database using a network. An Optimal Liquefaction Schedule for optimal operation of the liquefaction system is set out in Figures 7-10 and may be stored in said controller using the memory and database. The controller can sense optimum parameters of the system and optimally control, including by activating servomechanisms, liquefaction and transfer of liquid oxygen.

Dewar 14 is equipped with a dip tube 20 and heater 21. Heater 21 is used to build pressure in the dewar in order to expel liquid out the dip tube 20 when so desired. A quick disconnect valve 22 or other flow control means is located on the end of the dip tube. This allows connection of a portable LOX dewar 23, which can then be carried by the patient requiring a mobile/ambulatory supply of oxygen.

In another embodiment of this system shown in Figure 2, the dewar 14 could be eliminated and replaced with a portable dewar 23 which is modified slightly from those existing today. The new portable dewar would interface with the condenser 13, recuperator 15, and controller 16. This embodiment requires a slightly different control scheme from that given for the preferred embodiment as the transfer and boil-dry modes are eliminated. Any small amount of accumulated water and hydrocarbons are eliminated from the portable dewar 23 after each use by allowing it to warm to room temperature before reuse.

In operation, in the preferred embodiment of Figure 1, where a pressure swing adsorption (□PSA□) system is used, air is drawn into the oxygen concentrator 11, where it is compressed by an oilless compressor, cooled, and passed through molecular sieve beds operating on the pressure swing adsorption cycle, as shown in U.S. Patents 5,366,541; 5,112,367; 5,268,021 and Re. 35,009, which are incorporated herein by reference. This PSA system produces a 5-6 liters per minute (LPM) gas stream with high oxygen concentration at 3-7 pounds per square inch gauge (psig). The composition of this gas stream varies

but is typically 90-95% oxygen, 5-6% argon, 0-4% nitrogen, <15 parts per million (ppm) water vapor, and <1 ppm hydrocarbons. Exhaust from the PSA cycle (80-84% nitrogen, 15-19% oxygen, 0.6-0.8% argon, and trace amounts of water vapor, carbon dioxide and hydrocarbons) is vented into the atmosphere as waste gas. In the preferred embodiment, the high concentration oxygen stream in fluid outlet 50 is split with 0-4 lpm going through control valve 24, for patient consumption, and 0.5-1.5 lpm through line 51 and control valve 19 for liquefaction. Oxygen sensor 18, monitors the oxygen concentration produced by oxygen concentrator 11. If the oxygen concentration falls below 88%, controller 16 will close valve 19 and turn off the cryocooler 12.

Even though 88% oxygen is adequate as supplemental oxygen therapy, if this was liquefied, as will be described below, the initial revaporized stream may have a reduced oxygen content because of the close boiling points of the components of the mixture. The temperature of the split gas stream entering the recuperator 15 is about room temperature. It is cooled to about 270K (or colder) by the vent gas from the dewar flowing through the other side of the recuperator via line 52. The recuperator 15 reduces the load on the cryocooler by using the cold vent gas to pre-cool the oxygen-rich gas stream flowing into the condenser 13. From the recuperator 15 the high oxygen concentration stream flows through a line 57 to the condenser 13, which is cooled to ~90K by the cryocooler 12.

The condenser 13 provides cold surfaces to further cool and condense the flow. It is important to note that

the gas passing through the condenser 13 is a mixture of oxygen, argon, and nitrogen. The normal boiling points of these components are: 90.18K, 87.28K, and 77.36K respectively. Because of the close boiling points of the components of this mixture, there was initial skepticism because of the concern that all the nitrogen and argon would condense along with the oxygen. If this concern was realized, when this liquid mixture was revaporized, the lower boiling point components; i.e., nitrogen and argon, would boil off first, resulting in flow with high concentrations of nitrogen, argon and a much lower oxygen concentration than that which was supplied to the condenser -- which would make the process of oxygen treatment ineffective or a failure.

This concern is explained in Figures 3 and 4 which are temperature composition diagrams for binary mixtures of oxygen-argon and oxygen-nitrogen. In these diagrams taken from K.D. Timmerhaus and T.M. Flynn, *Cryogenic Process Engineering,* Plenum Press, 1989, pp. 296-297, the upper curve at a given pressure defines the dew point and the lower curve defines the bubble point. Looking at Figure 4 for a pressure of 0. 10 1 MPa, if there is a gas mixture with 10 mole percent nitrogen (point 1), condensation will start when the gas has cooled to the dew point curve (point 2g) which is at a temperature of about 89.5K in this case. Because oxygen has a higher boiling point than nitrogen, the initial liquid formed (point 2f) will have only 7.4 mole percent nitrogen. If the temperature is lowered to point 3, the liquid will have the composition of point 3f while the remaining vapor will

have the composition of point 3g. As the temperature is lowered further to point 4f or below, all of the mixture liquefies and the composition is 10 mole percent nitrogen, the same as at point 1. If this liquid is heated, the nitrogen which has a lower boiling point will vaporize first. Thus, the composition of the first vapor formed will be that of point 4g or about 30 mole percent. As the remaining liquid boils, the mole percent of nitrogen in the vapor drops back to 10 mole percent when point 2g is reached. It is believed that the composition swings with a ternary mixture of oxygen, argon and nitrogen will be even more pronounced than those shown in Figures 3 and 4 for binary mixtures. Fortunately, this concern was avoided when the system was set so that only 20 to 90% of the incoming flow to the condenser was actually condensed and when the condenser was controlled in accordance with the parameters as explained herein. This is believed to work because the excess flow operates to purge the vapor with higher impurity concentration from the system and avoid the aforementioned problem. Instead, the results realized were that a high concentration stream of oxygen could be liquefied and stored as set out in the portable ambulatory device claimed herein.

Figure 5 shows typical oxygen concentration test data for condensing and re-vaporizing part of the product outlet stream from the oxygen concentrator 11 in the preferred embodiment. For the first 120 minutes after the system was turned on, the system was cooling down without any net liquid accumulation in the dewar 14. From this point up to about 500 minutes, condensation continued with liquid accumulation. During this time phase, the inlet stream to the condenser 13 had an oxygen concentration of

95%, while the vent flow through line 52 had an oxygen concentration of only 92-93%. After 500 minutes the inlet stream and condenser cooling were stopped. The oxygen concentration of the re-vaporized liquid increased as the liquid boiled off due to the lower boiling point components (argon and nitrogen) boiling off first. This change in oxygen concentration presents no problem for medical ambulatory use because the oxygen concentration remains above 85%.

Because of the aforementioned mixture problem, it is important and even critical not to let the amount of argon and nitrogen in the liquid become too high or when it is revaporized, the oxygen concentration will initially be much lower than that conventionally used in supplemental oxygen therapy (>85%). This can be accomplished by selecting the proper condenser temperature, which is a function of pressure, and by not condensing all of the incoming flow. If only part of the incoming flow (20-90%) is liquefied, the remainder of the flow will purge the vapor with higher impurity concentration from the system. A condenser temperature of about 90K (for □17 psia) minimizes the amount of argon and nitrogen liquefied without overly diminishing the yield of oxygen. Hence there will be both liquid and vapor leaving the condenser. The liquid will fall into the dewar 14 and collect. The vapor which has not condensed is vented to the atmosphere through line 52 and the recuperator 15.

The amount of incoming flow liquefied is controlled by setting the mass flow rate relative to the cooling capacity of the cryocooler. The parameters of the condenser and/or cryocooler can be stored in the memory of

the controller and/or computer and the controller regulating the incoming flow depending on the parameters stored and/or sensed. Having a mass flow rate which exceeds the cooling capacity of the cryocooler/condenser combination, prevents the incoming flow from being completely liquefied. The mass flow rate is controlled by the amount of flow restriction between inlet valve 19 and flow control valve 25. This includes the flow losses of the valves themselves as well as those in the recuperator, condenser, and all of the interconnecting plumbing.

The pressure in the dewar 14 is maintained slightly above ambient pressure while the cryocooler is operating by valve 25. It is desirable to keep the pressure in the condenser as high as possible because this increases the condensation temperature (as shown in figures 3 and 4) which eases the requirements on the cryocooler. Once again this can be controlled by the controller and/or the computer, microprocessor and memory system.

This pressure regulating function of the solenoid on-off valve 25 is accomplished by the pressure transducer 9 and controller 16. Alternately, a back pressure regulating valve (such as a Tescom BB-3 series) or a suitable servomechanism may be used in lieu of the actively controlled solenoid. Liquid keeps accumulating in the dewar 14 until the liquid level sensor 17 signals the controller that the dewar is full or until the oxygen sensor 18 signals that the oxygen concentration of fluid exiting the oxygen concentrator 11 is too low.

In the best mode, operating parameters for optimal operation of the system for the condenser should be that

the condenser surface temperature should be in the range from 69.2-109.7K and pressure should be in the range from 5-65 psia. The gas concentrations into the condenser for medical use should have oxygen in the range of 80-100%, nitrogen from 0-20%, and argon from 0-7%.

In order to transfer liquid from the dewar 14; e.g. to fill a portable LOX dewar 23, the pressure in the dewar 14 must be increased so that liquid can be forced up the dip tube 20. As shown in Figure 1, heater 21 is used for this purpose. Heater 21 may be immersed in the liquid oxygen or attached to the outer surface of the inner vessel. The controller 16 ensures that the cryocooler 12 is turned off and valve 25 is closed before the heater 21 is energized. The heater 21 remains turned on until the pressure, measured by pressure transducer 9, reaches about 22 psig.

In order to eliminate accumulation of solid water and hydrocarbons which may be supplied in trace amounts from the oxygen concentrator, the dewar 14 will be warmed to room temperature periodically (preferably after about 30 fillings of a portable dewar, or every two months). This procedure is accomplished most economically when the inventory of liquid in the storage dewar is low; e.g. shortly after liquid transfer and a portable dewar has been filled. In this □boil-dry□ mode, valve 19 will be closed, the cryocooler 12 is turned-off, valve 25 is open, and heater 21 is energized. The heater will boil-off the remaining liquid in the dewar and with it any trace amounts of water and hydrocarbons which are condensed and solidified in the liquid oxygen or on the cold surfaces. The heater 21 will remain turned on until the dewar

temperature, measured by temperature sensor 10, has warmed to about 300K. Any remaining water vapor will be flushed out by gaseous oxygen during the subsequent cool-down.

At initial start-up or after a periodic boil-dry phase, the dewar, condenser, recuperator, and all associated hardware are at room temperature and must be cooled down. This is accomplished in the □start-up□ mode, where valve 19 (see Figure 1) is open, the heater is off, the cryocooler is on, and valve 25 is modulated to control the pressure/flow rate. It is desired to keep the pressure and hence the density of the gas as high as possible while maintaining the flow rate.

The higher density gas will have better heat transfer with the dewar walls and associated hardware. It is noted that higher flow rates will enhance the convection heat transfer but may exceed the cooling capacity. Based on the cooling characteristics of the cryocooler between room temperature and 90K, the flow rate can be changed to minimize the cool-down time.

The dewar 14 is equipped with at least one relief valve 26 as a safety feature. Another relief valve 29 is provided and in communication with the inlet gas stream 51, before flowing into the recuperator 15. This serves as a back-up for relief valve 26 as well as providing a means to eliminate accumulated water from the recuperator 15 during periods when the cryocooler 12 is off, if valve 25 is closed. A check valve 27 is also provided to prevent backflow into the oxygen concentrator in the event of a malfunction.

Figure 6 provides a block diagram of the controller 16 control system with sensor input value ranges and output states. It also shows interfaces to an indicator and a modem or wireless interface. The mode switch 28 may be used by the patient to request the system to prepare for a liquid transfer to a portable dewar. The indicator then provides a visual signal that the system is building pressure in the dewar. Once the pressure has reached the desired value, a visual and/or audio signal is given to alert the patient that the system is ready to transfer liquid. The controller may also be programmed to perform an unattended liquid transfer. The modem, telephone line or wireless interface connections are optional hardware that may be added to the controller to enable remote monitoring of the system by the home care provider (e.g., to assist with maintenance and repair) or insurance companies or health providers/administrators (e.g., to assess if patients are using enough ambulatory oxygen to justify payments, etc.).

Figures 7A-D show a logic flow chart for the controller for the normal operation modes. This can also be referred to as the □input/output control schedule.□ The mode switch 28 can also be used by a repair or factory technician to put the controller in a calibration mode which serves as a method to check and reset the program. As shown in Figure 6, the indicator provides liquid level readout, transfer request status, and low oxygen concentration information to the patient. All of the sensors are continuously scanned to provide the controller with the latest information. Figures 8 through 11 provide detailed output states as a function

of input levels for the normal operating modes (start-up, condense, transfer, and boil-dry), which can be referred to as the □Optimal Liquefaction Operational Schedule.□

For example, Figure 8 relates to the start-up mode; i.e., when the system is first turned on or after the boil-dry cycle. As shown in Figures 6 through 8 and as depicted in Figure 1, at start-up mode, the liquid sensor 17 shows zero liquid volume in the dewar and, when the oxygen sensor 18 shows an oxygen concentration greater than 88%, valve 19 is open, heater 21 is off, cryocooler 12 is on, and the indicator or the controller indicates a cool-down state. Valve 25 is modulated to control pressure.

Once the system attains a cool enough temperature, steady state or normal operational condense mode is used. As shown in Figure 9, the input to the controller 16 is such that when the oxygen sensor indicates oxygen concentration being greater than 88% and when the other criteria in the left-hand column of Figure 9 are achieved, the output states set out in the right portion of the chart are attained. For example, when the level of the dewar is sensed as being full, the liquid level sensor indicates a level of approximately 100%, causing closure of valves 19 and 25, keeping the heater off, turning the cryocooler off, and having the indicator signal that the dewar is full.

The transfer mode in Figure 10 is the stage where one can fill the portable thermos bottles or dewars 23 from the main storage dewar 14. The top portion of

Figure 10, for example, shows controller readouts where, if the liquid sensor indicates a liquid level of less than 20%, then the conclusion is computed that there is not enough liquid to transfer into the portable dewar from the main dewar as shown. When the operator wants to increase the pressure in the storage dewar to force the liquid oxygen into the portable dewar, the heater is activated, and when the pressure sensor indicates that the pressure exceeds 22 psig, as shown on the last line in the left-hand column of Figure 10, the heater 21 is then turned off and the controller readout or indicator shows that the transfer of liquid oxygen can be made to the portable dewar. Finally, Figure 11 indicates the boil-dry mode, with valve 25 open to allow the vapor to escape, and the various parameters relating thereto.

Figure 12 shows a schematic of a pulse tube refrigerator, the preferred embodiment of the cryocooler 12 in Figure 1. Because the cooling load on the condenser is small (7-15W), the pulse tube refrigerator is preferred for use in the subject ambulatory oxygen system because of its good efficiency with only one moving part, the pressure oscillator. Pulse tube refrigerators are shown in US Patent Nos. 5,488,830; 5,412,952 and 5,295,355 the disclosure of which are hereby incorporated by reference. Figure 11 depicts a pulse tube refrigerator of the double inlet type. Other types of pulse tube refrigerators (PTR) could also be used such as the basic PTR or the inertance tube PTR (Zhu et al., 9$^{th}$ International Cryocooler Conference, NH, June 1996).

The double inlet pulse tube refrigerator as shown in Figure 12 is comprised of a pressure oscillator 30, primary heat rejector 31, regenerator 32, heat acceptor 33, pulse tube 34, orifice rejector 35, bypass orifice 36, primary orifice 37, and reservoir volume 38. The preferred refrigerant gas in the PTR closed and pressurized circuit is helium but various other gases such as neon or hydrogen could also be used. In operation, the PTR essentially pumps heat accepted at low temperature in the heat acceptor 33 to the orifice heat rejector 35 where it is rejected at near ambient temperature. Although Figure 12 depicts a □U-tube□ configuration of the PTR, in-line and coaxial configurations are other possible options. Depicted therein is a piston type pressure oscillator, but other types are possible such as those utilizing diaphragms or bellows.

Figure 13 shows a schematic of another embodiment of the cryocooler. This is a vapor compression cycle cryocooler using a mixed gas refrigerant such as shown in US Patent No. 5,579,654; a 1969 German Patent by Fuderer & Andrija; British Patent No. 1,336,892. Other types of cryocoolers will work as long as they meet the important criteria of small size, convenience and low cost. In Figure 13, the refrigerant is compressed by the compressor to high pressure. Then it is cooled by the aftercooler with heat Qh being rejected to the environment. Oil is separated in the oil separator. Oil flows back to the compressor inlet through a flow restriction. The refrigerant gas flows to a heat exchanger where it is cooled by the returning cold stream. Some components of the mixture may condense in

this process. The liquid/gas refrigerant mixture flows through a throttle valve where its pressure is reduced and its temperature drops. This cold refrigerant enters the evaporator where the heat load Qc is absorbed and some liquid is boiled into vapor. This vapor flows up the cold side of the heat exchanger absorbing heat from the incoming stream. Then it flows back to the compressor. Heat Qc is accepted at cold temperature Tc. This is where the condenser would interface with the cryocooler.

It is noted that with this type of cryocooler, it may be possible to remove some of the heat from the oxygen stream at a temperature warmer than Tc.

One possible geometry of the generally vertically oriented, gravity assisted condenser 13 in Figure 1 is shown in Figures 14 and 15. The incoming gas from the oxygen concentrator flows from conduit 57 to chamber 58 and then is distributed through an annular passage 59 between the outer tube 41 and inner rod 42. The inner rod 42 is made of a high thermal conductivity material such as OFHC (Oxygen Free High Conductivity) copper, to minimize the temperature gradient between the surface on which the oxygen condenses (13) and the cryocooler 12. The cold end of the cryocooler is shown by cross-hatched member 61. Due to surface tension, the axial slots or grooves 43 will draw in liquid as it condenses. This will enhance heat transfer from the incoming gas by preventing a liquid film from forming over the entire condenser surface. Condensed liquid will drip off the bottom of the rod 42 while non-condensed gases flow out the end of the annulus 60. It is possible to liquefy

all of the incoming flow to the condenser provided the cryocooler has sufficient cooling capacity and temperature capability. However, in order to minimize the amount of nitrogen and argon condensed, the preferred embodiment only condenses between 20-90% of the incoming flow. The incoming flow rate can be determined by the appropriate sizing of flow restrictions downstream of and by controlling valve 19. As mentioned previously, the mass flow rate is chosen to exceed the cooling capacity of the condenser/cryocooler so that only part of the incoming flow is liquefied. Also, the pressure in the condenser is maintained as high as possible while maintaining the desired flow rate. The higher pressure increases the condensation temperature which in turn reduces the requirements on the cryocooler.

Figures 16 and 17 show another embodiment of the condenser that allows easier integration with the mixed gas refrigerant cryocooler. This configuration also allows access to the liquid in the dewar through the center of the condenser. The cold end of the cryocooler 45 is in thermal contact with an outer tube 46 and an inner tube 47, both of which are made of a high thermal conductivity material such as OFHC copper and which utilize flanges 62 and 64 to interface with the cryocooler. The inner tube has axial slots or grooves 48 cut into its outer surface (see, Figure 17) to increase the surface area and to wick condensed liquid, preventing a liquid film from forming over its entire surface. Gas enters the condenser through port 63. The liquid and vapor flow down through an annular passage

49. An isometric view of this embodiment is shown in Figure 18.

Thus, an improved home/ambulatory liquid oxygen system is disclosed. While the embodiments and applications of this invention have been shown and described, and while the best mode contemplated at the present time by the inventors has been described, it should be apparent to those skilled in the art that many more modifications are possible, including with regard to scaled-up industrial applications, without departing from the inventive concepts therein. Both product and process claims have been included and in the process claims it is understood that the sequence of some of the claims can vary and still be within the scope of this invention. The invention therefore can be expanded, and is not to be restricted except as defined in the appended claims and reasonable equivalence departing therefrom.

CLAIMS

1. A home liquid oxygen ambulatory system for supplying a portable supply of oxygen, where a portion of the gaseous oxygen output obtained from an oxygen concentrator is condensed into liquid oxygen, comprising:

(a) an oxygen concentrator which separates oxygen gas from the ambient air;
(b) an outlet flow line to transfer flow of oxygen gas from said oxygen concentrator for patient use;
(c) a valve placed in the outlet flow line for splitting off a portion of the oxygen gas flow generated by the oxygen generator;
(d) a condenser for receiving and liquefying the split off portion of the oxygen gas flow;
(e) a cryocooler associated with said condenser;
(f) a first storage dewar in fluid communication with said condenser for storing the oxygen liquefied by the condenser, the first storage dewar having an outlet selectively engageable to and in fluid communication with at least one second smaller dewar and a fluid path for supplying liquid oxygen from the first dewar to the second dewar;
(g) a heater for heating said first storage dewar;

(h) a controller for monitoring (i) oxygen concentration of the oxygen gas flowing from said concentrator, and (ii) the amount of liquid oxygen in said first dewar, and for controlling the parameters of liquid oxygen generation and transfer from said first storage dewar.

2. The liquid oxygen system of claim 1 wherein the oxygen concentrator is a pressure swing adsorption (□PSA□) type oxygen concentrator.

3. The liquid oxygen system of claim 1 wherein the flow rate into the condenser is chosen to exceed the capacity of the condenser.

4. The liquid oxygen system of claim 1 wherein only 20 to 90% of the split off portion flowing into the condenser is condensed to minimize the liquefaction of argon, nitrogen and trace gases.

5. The liquid oxygen system of claim 1 wherein the controller controls condenser parameters so that the condenser temperature varies in the range from approximately 69.2 to 109.7K, the condenser pressure varies from approximately 5 to 65 psia, and the concentrations of gas into the condenser varies approximately as follows:

oxygen: 80 to 100%
nitrogen: 0 to 20%
argon: 0 to 7%

6. The liquid oxygen system of claim 1 wherein vent gas from the first storage dewar is recovered and used to pre-cool the gas into the condenser.

7. The liquid oxygen system of claim 1 wherein the condenser is in thermal contact with the cryocooler and comprises:

(a) an inlet conduit for receiving the split off portion of oxygen;
(b) an outer member;
(c) an inner member having a chamber in communication with said inlet conduit;
(d) a passage defined by said outer and inner members;
(e) said inner member having axial slots; and
(f) means for circulating said oxygen in said passage defined by said inner and outer members;

8. The liquid oxygen system of claim 1 wherein a generally vertically oriented, gravity assisted condenser for use in liquefaction of oxygen from its gaseous to its liquid phase comprises:

(a) an inlet for receiving a stream of gaseous oxygen;
(b) an outer member;
(c) an inner member;
(d) a passage defined by said inner and outer members, the inlet being in communication with said passage;

(e) the inner member having axial slots to enhance heat transfer and to avoid liquid film build-up; and

(f) at least one of said members being in thermal contact with said cryocooler.

9. The liquid oxygen system of claim 1, further comprising a recuperator interfaced between the oxygen concentrator and the condenser.

10. The liquid oxygen system of claim 1, wherein the controller senses pressure in said first dewar and controls said heater in response thereto.

11. The liquid oxygen system of claim 1 wherein the first storage dewar will be periodically boiled dry to eliminate any trace gases that may pass through the gas concentrator.

12. The liquid oxygen system of claim 1, wherein the condenser comprises a generally vertically oriented, gravity assisted, circular housing with an inner center grooved core defining an annulus and with axial slits to wick away the condensed liquid.

13. The liquid oxygen system of claim 1 wherein the controller is located remotely from the oxygen source.

14. The liquid oxygen system of claim 1 wherein the controller monitors the oxygen concentration and amount of liquid oxygen and controls the parameters of

liquid oxygen generation and transfer remotely using a modem.

15. The liquid oxygen system of claim 1 wherein the controller monitors the oxygen concentration and amount of liquid oxygen and controls the parameters of liquid oxygen generation and transfer remotely using a wireless interface.

16. A control system for a home ambulatory liquid oxygen system having an oxygen concentrator, condenser, cryocooler, a heater and a storage dewar, the control system comprising:

(a) an oxygen concentration sensor which senses the concentration of oxygen gas generated by the oxygen concentrator and generates a first signal in response thereto;
(b) a liquid level sensor which senses the liquid level in the dewar and generates a second signal in response thereto;
(c) a temperature sensor which senses the temperature in the dewar and generates a third signal in response thereto;
(d) a microprocessor for receiving the first, second and third signals for computing the flow concentration of gaseous oxygen out of the concentrator and into the condenser, the level of liquid oxygen in the dewar, the temperature in the dewar and for controlling the transfer of liquid oxygen from said storage dewar.

17. The control system in claim 16 wherein the microprocessor is located remotely from the home ambulatory system and connected to the sensors and controllers using a modem.

18. The control system in claim 16 wherein the microprocessor is located remotely from the home ambulatory system and connected to the sensors and controllers using a wireless interface.

19. The control system in claim 16 wherein the actual flow rate into the condenser is chosen to exceed the designed flow capacity of the condenser allowing the excess flow to purge the system.

20. The liquid oxygen system of claim 16 wherein only 20 to 90% of the incoming flow into the condenser is condensed to minimize the liquefaction of argon, nitrogen and trace gases.

21. The apparatus of claim 16 wherein the controller controls condenser parameters so that the condenser temperature varies in the range from approximately 69.2 to 109.7K, the condenser pressure varies from approximately 5 to 65 psia, and the concentrations of gas into the condenser varies approximately as follows:

oxygen: 80 to 100%
nitrogen: 0 to 20%
argon: 0 to 7%

22. An ambulatory liquid oxygen system where liquid oxygen is formed from gaseous oxygen obtained from an oxygen concentrator comprising:

(a) an oxygen concentrator which separates oxygen gas from the air line;
(b) a first output line from said oxygen concentrator for delivering oxygen gas from said concentrator;
(c) a first valve placed in said first output line to control the output flow of oxygen;
(d) a condenser in cooperation with a cryocooler for providing cooling for said condenser;
(e) a second output line in communication between the first valve and the condenser for delivering the oxygen gas from the concentrator output to the condenser for cooling to form liquid oxygen;
(f) a storage dewar for storing liquid oxygen liquefied by the condenser;
(g) a third output line in communication between said condenser and storage dewar for delivering liquid oxygen from the condenser to the storage dewar;
(h) a heater for heating liquid oxygen in said dewar;
(i) an oxygen sensor to measure the oxygen concentration of flow from said concentrator;
(j) a liquid level sensor in said dewar;
(k) a temperature sensor in said dewar;

(l) a pressure sensor in communication with said dewar; and

(m) a controller for receiving output from the oxygen sensor, liquid level sensor, temperature sensor and pressure sensor, and for controlling the flow of input gaseous oxygen to the condenser and for controlling liquefaction and transfer from the storage dewar in accordance with the sensed conditions.

23. The system of claim 22 wherein the oxygen concentrator is of the Pressure Swing Adsorption (□PSA□) type.

24. The system of claim 22 wherein the condenser parameters are maintained as follows:

temperature from approximately 69.2 to 109.7K;
pressure from approximately 5 to 65 psia;
gas concentrations into condenser:
oxygen from approximately 80 to 100%,
nitrogen from approximately 0 to 20%, and
argon from approximately 0 to 7%

25. The system of claim 22 further comprising a recuperator between the oxygen concentrator and condenser for pre-cooling the flow of gas into the condenser.

26. The liquid oxygen system of claim 22 wherein the flow rate into the condenser is chosen to exceed the designated capacity of the condenser.

27. The liquid oxygen system of claim 22 wherein only 20 to 90% of the incoming flow to the condenser is condensed to minimize the liquefaction argon, nitrogen and trace gases.

28. The liquid oxygen system of claim 22 wherein the generally vertically oriented, gravity assisted condenser is in contact with the cryocooler and comprises:

(a) an inlet for receiving gaseous oxygen;
(b) an outer tubular member;
(c) an inner member;
(d) a passage defined by said outer and inner members;
(e) said inner member having axial slots cut into its outer surface;
(f) means for circulating said oxygen in said passage; and
(g) an outlet for releasing liquid oxygen,

wherein when said oxygen gas enters said condenser at said inlet and passes through said passage, condensation takes place causing a transition from gas to liquid phase.

29. The liquid oxygen system of claim 22 where the generally vertically oriented, gravity assisted, condenser comprises:

(a) an inlet for receiving a stream of oxygen;
(b) an outer member;

(c) an inner member;
(d) passage defined by said inner and outer members;
(e) the inner member having axial slots to enhance heat transfer and to avoid liquid film build-up; and
(f) an outlet for the oxygen which is liquefied.

30. The liquid oxygen system in claim 22 wherein the controller is located remotely from the oxygen generator.

31. The liquid oxygen system in claim 22 wherein the controller monitors the oxygen concentration and amount of liquid oxygen and controls the parameters of liquid oxygen generation and transfer remotely using a modem.

32. The liquid oxygen system in claim 22 wherein the controller monitors the oxygen concentration and amount of liquid oxygen and controls the parameters of liquid oxygen generation and transfer remotely using a wireless interface.

33. A method for controlling an ambulatory oxygen generator and oxygen liquefier having an oxygen concentrator, a condenser, a storage dewar and a controller comprising the following steps:

(a) generating a gaseous supply of oxygen using the oxygen concentrator for supplying oxygen to a patient;
(b) splitting off a portion of the gaseous supply to be liquefied;
(c) cooling said split-off supply of oxygen using the condenser to transform the oxygen from gaseous phase to liquid phase;
(d) storing the liquid oxygen in the storage dewar;
(e) sensing a concentration of oxygen being supplied by the oxygen concentrator, the level of liquid oxygen in said dewar and the temperature and pressure in said dewar; and
(f) the controller calculating conditions and controlling the operation of the above system to allow for liquefaction and operative transfer of liquid oxygen to a portable dewar.

34. A method for controlling a home ambulatory liquid oxygen system comprised of an oxygen generator, a controller having a microprocessor, a condenser, a cryocooler and a storage dewar, where all or only a portion of the oxygen flow is utilized for liquefaction, comprising:

(a) providing the microprocessor with a database and control functions;
(b) sensing the parameters relating to the concentration and supply of gaseous

oxygen, the level of liquid oxygen in the dewar, and the pressure of the condenser;

(c) providing the microprocessor with these sensed parameters and having the microprocessor calculate optimal conditions;

(d) controlling servomechanisms to regulate the system so that optimal conditions are realized as a function of said calculations.

35. An ambulatory home oxygen concentrator liquefaction system comprising:

(a) an oxygen concentrator which can isolate oxygen gas from the air;

(b) a first output means from the concentrator to channel the oxygen which is isolated;

(c) a second means to split off all or a portion of the gaseous oxygen from the output means of said concentrator for liquefaction;

(d) a condenser which can cause gaseous oxygen to change phase into liquid oxygen;

(e) means to channel the oxygen stream from said output means to a condenser;

(f) means to liquefy said oxygen stream in said condenser using a cryocooler; and

(g) means to collect the condensed liquid oxygen using a first dewar, wherein said first dewar includes a heater which can

be used to effectuate the transfer of liquid oxygen from the first dewar to a second dewar for storing a quantity of liquid oxygen from which smaller quantities can be transferred for moveable oxygen treatment.

36. The apparatus of claim 35 wherein the oxygen concentrator is a pressure swing adsorption (□PSA□) type oxygen concentrator.

37. The liquid oxygen system of claim 35 wherein the flow rate into the condenser is chosen to exceed the capacity of the condenser.

38. The liquid oxygen system of claim 35 wherein only 20 to 90% of the incoming flow to the condenser is condensed to minimize the liquefaction argon, nitrogen and trace gases.

39. The apparatus of claim 35 wherein the controller controls condenser parameters or ranges so that the condenser temperature varies in the range from approximately 69.2 to 109.7K and the condenser pressure varies from approximately 5 to 65 psia, and the concentrations of gas into the condenser varies approximately as follows:

Oxygen: 80 to 100%
Nitrogen: 0 to 20%
Argon: 0 to 7%

40. The apparatus of claim 35 wherein vent gas from the storage dewar is recovered and used to pre-cool the gas into the condenser.

41. The apparatus of claim 35, further comprising a recuperator interfaced between the oxygen concentrator and the condenser.

42. The apparatus of claim 35, further comprising a controller which senses pressure in the first dewar and controls said heater in response thereto.

43. The system set forth in claim 35 wherein the liquid dewar will be periodically boiled dry to eliminate any trace gases that may pass through the gas concentrator.

44. The apparatus of claim 35, wherein the generally vertically oriented, gravity assisted condenser comprises a circular housing with an inner center grooved core defining an annulus and with axial slits to wick away the condensed liquid.

45. The liquid oxygen system in claim 42 wherein the controller is located remotely from the oxygen generator.

46. The liquid oxygen system in claim 42 wherein the controller monitors the oxygen concentration and amount of liquid oxygen and controls the parameters of liquid oxygen generation and transfer remotely using a modem.

47. The liquid oxygen system in claim 42 wherein the controller monitors the oxygen concentration and amount of liquid oxygen and controls the parameters of liquid oxygen generation and transfer remotely using a wireless interface.

48. A controller for a home ambulatory liquid oxygen system having a computer, an oxygen concentrator, condenser, and storage dewar comprising:

(a) means for sensing the oxygen pressure of flow out of the concentrator;
(b) means for sensing the oxygen concentration of flow out of the concentrator;
(c) means for sensing the liquid level in the dewar;
(d) means for sensing flow parameters at the condenser;
(e) means for sensing the temperature at the dewar;
(f) means for controlling input of gaseous oxygen to the condenser;
(g) means for initiating and controlling the output of liquid oxygen from the storage dewar;
(h) a programmable control means coupled to each means for sensing, said programmable control means to be used for storing at least one required input/output control schedule, for processing the inputted signals and for actuating one or more control means.

49. A system for liquefying oxygen utilizing an oxygen concentrator, condenser, cryocooler and storage dewar comprising:

(a) a first flow control means for channeling off gaseous fluid flow from an oxygen concentrator;

(b) a second flow control means for advancing said gaseous fluid to a condenser;

(c) means for cooling said condenser to change the phase of said oxygen flow from gas to liquid;

(d) a conduit for transferring the liquid oxygen from the condenser to a storage dewar;

(e) a third flow control means for advancing the flow of liquid oxygen out of the storage dewar;

(f) a controller for sensing the oxygen concentration of flow into the condenser, for sensing the liquid level in the storage dewar, and for controlling said first, second, and third flow control means;

(g) programmable control means in said controller coupled to each of said flow control means, for storing flow delivery and liquefaction parameters for use by the controller in optimizing liquefaction of oxygen.

50. The liquid oxygen system of claim 49 wherein the flow rate into the condenser is controlled to exceed the capacity of the condenser.

51. The liquid oxygen system of claim 49 wherein only 20 to 90% of the incoming flow to the condenser is condensed to minimize the liquefaction argon, nitrogen and trace gases.

52. The liquid oxygen system of claim 49 wherein contact with a cryocooler for use in liquefying oxygen comprises:

(a) an inlet for receiving gaseous oxygen;
(b) an outer tubular member;
(c) a generally vertically oriented inner member;
(d) a passage defined by said outer and inner members;
(e) said generally vertically oriented inner member having axial slots cut into its outer surface;
(f) means for circulating said oxygen in said passage; and
(g) an outlet for releasing liquid oxygen;

wherein when said oxygen gas enters said condenser at said inlet and passes through said passage, condensation takes place causing a transition from gas to liquid phase.

53. The liquid oxygen system of claim 49 where the generally vertically oriented, gravity assisted condenser for use in liquefaction of oxygen from its

gaseous to its liquid phase and for use with a cryocooler comprises:

(a) an inlet for receiving a stream of oxygen;
(b) an outer member;
(c) an inner member;
(d) passage defined by said inner and outer members;
(e) the inner member having axial slots to enhance heat transfer and to avoid liquid film build-up
(f) an outlet for the oxygen which is liquefied.

54. A generally vertically oriented, gravity assisted condenser in thermal contact with a cryocooler for use in liquefying oxygen comprising:

(a) an inlet for receiving gaseous oxygen;
(b) an outer tubular member;
(c) an inner member;
(d) a passage defined by said outer and inner members;
(e) said inner member having axial slots cut into its outer surface;
(f) means for circulating said oxygen in said passage; and
(g) an outlet for said liquid oxygen;

wherein when said oxygen gas enters said condenser at said inlet and passes through said passage, condensation takes place causing a transition from gas to liquid phase.

55. A generally vertically oriented, gravity assisted condenser for use in liquefaction of oxygen from its gaseous to its liquid phase and for use with a cryocooler comprising:

(a) an inlet for receiving a stream of oxygen;
(b) an outer member;
(c) an inner member;
(d) passage defined by said inner and outer members;
(e) the inner member having axial slots to enhance heat transfer and to avoid liquid film build-up; and
(f) an outlet for the liquid oxygen to exit the condenser.

56. A home liquefaction system including an oxygen concentrator, controller with a programmable control means, condenser, cryocooler and storage dewar comprising:

(a) an oxygen concentrator which separates oxygen gas from the air;
(b) a first flow line from said oxygen concentrator for delivering oxygen gas from said concentrator;
(c) a first valve placed in said first flow line to control the output flow of oxygen;

(d) a condenser in cooperation with a cryocooler for providing cooling for said condenser;

(e) a second flow line in communication between the first valve and the condenser for delivering the oxygen gas from the concentrator output to the condenser for cooling to form liquid oxygen;

(f) a storage dewar for storing liquid oxygen liquefied by the condenser;

(g) a third flow line in communication between said condenser and storage dewar for delivering liquid oxygen from the condenser to the storage dewar;

(h) a heater for heating liquid oxygen in said dewar;

(i) an oxygen sensor to measure the oxygen concentration of flow from said concentrator;

(j) a liquid level sensor in said dewar;

(k) a temperature sensor in said dewar;

(l) a pressure sensor for sensing pressure in said dewar;

(m) a second valve for regulating flow of liquid oxygen from said dewar;

(n) a controller for receiving output from the oxygen sensor, liquid level sensor, temperature sensor and pressure sensor, and having means for storing at least one parameter from the Optimum Liquefaction Schedule, for controlling the flow of input gaseous oxygen to the condenser, and for optimizing liquefaction and

transfer from the storage dewar in accordance with the sensed conditions.

57. The apparatus of claim 56 wherein said programmable control means including means for storing information relating to optimum condenser parameters and for displaying an indicium of the modes of operation prior to activating any of said flow control means.

58. A system as in claim 56 wherein said programmable control means includes means for scheduling intermittent actuation of one or more of said valves so as to permit the system to be operated in accordance with the Optimum Liquefaction Schedule;

59. A system as in claim 56 including means for adjusting actuation of selected flows and conditions so as to optimize condenser parameters in response to said programmable control means and Optimum Liquefaction Schedule.

60. A system as in claim 56 including means for visually displaying controller parameters.

61. A system in claim 56 including means for transferring said liquid oxygen from said storage dewar to the delivery port at a controllable rate and including means to actuate said means in accordance with a predetermined Optimum Liquefaction Schedule;

62. An oxygen liquefaction system utilizing a concentrator to increase the oxygen concentration of an oxygen enriched product gas comprising:

(a) a first output line for transporting said product gas from said concentrator;

(b) a first valve for selectively withdrawing all or a portion of said product gas from said conduit to be liquefied;

(c) flow rate sensor independent of said first valve for sensing the rate of withdrawal of the product gas from said conduit to be liquefied and for communicating the sensed rate of withdrawal of said product gas to a modem;

(d) an oxygen sensor for sensing the oxygen concentration in said oxygen enriched product gas selectively withdrawn for liquefaction and for communicating the sensed oxygen concentration in said product gas to a first modem;

(e) a condenser for receiving the product gas withdrawn from said first flow control means;

(f) a second output line from said first flow control means oxygen concentrator for delivering oxygen gas from said concentrator for a first use;

(g) a second flow rate sensor in said second output line;

(h) a second oxygen sensor in said second output line;

(i) a cryocooler for providing cooling for said condenser;

(j) a storage dewar for storing liquid oxygen liquefied by the condenser;

(k) a third output line in communication between said condenser and storage dewar for delivering liquid oxygen from the condenser to the storage dewar;

(l) a heater for heating liquid oxygen in said dewar;

(m) a liquid level sensor for sensing the level in said dewar;

(n) a temperature sensor for sensing the temperature in said dewar;

(o) a pressure sensor for sensing the pressure in said dewar;

(p) a second modem for receiving quantitative signals communicated from said flow rate sensing means and said oxygen sensing means and for transmitting said signals to a remote controller; and

(q) a remote controller in telephonic communication with said first and second modems for receiving transmitted signals from said modems and having means for displaying the liquefaction parameters in the liquefaction process.

63. The oxygen liquefaction system of claim 62 wherein the concentrator increases the oxygen concentration of an oxygen enriched product gas recovered therefrom using a plurality of molecular sieve beds for receiving atmospheric air for selectively adsorbing nitrogen therefrom, an electric motor driven compressor including means for providing line voltage

for operating said compressor, and a valve cooperating with said compressor for alternately charging said sieve beds with atmospheric air and timing means for switching said valve to alternately charge said sieve beds according to a timing cycle.

64. The oxygen liquefaction system of claim 62 wherein said flow rate sensors comprise a pressure transducer for measuring product gas pressure therein.

65. The system of claim 64 including a microprocessor cooperating with said pressure transducers and having means for determining the rate of flow of product gas into the condenser in response to pressure sensings therein.

66. The system of claim 65 wherein said test apparatus includes visual display means for displaying the product gas flow rate and the oxygen concentration into the condenser and the pressure and temperature in the storage dewar.

67. The system of claim 62 wherein said remote control apparatus includes means for selectively signaling said modem to transmit said signals from said liquefaction apparatus.

68. The system of claim 62 including a microprocessor having means for receiving the sensed rate of product gas withdrawal and oxygen concentration in said product gas from said oxygen sensor, the pressure, temperature, and liquid level in the storage dewar, and having memory means provided with minimum

selected parameters at different conditions, means for comparing the sensed parameters with the minimum selected parameters, and means for signaling said modem to transmit said signals to said controller when said sensed parameters fall below said minimum selected parameters.

69. The oxygen concentrator of claim 68 wherein said first modem is connected to said microprocessor and said second remote modem is connected to said controller at a remote location, and wherein said modems are in telephonic communication.

70. The concentrator of claim 69 wherein said microprocessor includes means for signaling said first modem to transmit said signals to said controller when said sensed oxygen concentration or liquefaction falls below said minimum selected parameters.

71. The apparatus of claim 70 wherein said microprocessor cooperates with said first means and said second means and wherein said controller includes selective digital display means for displaying liquefaction parameters.

72. The system in claim 70 wherein the entry system not including the oxygen concentrator weighs no more than about 60 pounds and takes up less than about six cubic feet of volume.

73. An ambulatory liquid oxygen system where liquid oxygen is formed from gaseous oxygen obtained from an oxygen concentrator comprising:

(a) an oxygen concentrator which separates oxygen gas from the air;

(b) a first output means from said oxygen concentrator for delivering oxygen gas from said concentrator;

(c) a first valve placed in said first output means to control the output flow of oxygen;

(d) a condenser in cooperation with a cryocooler for providing cooling for said condenser;

(e) a second output line in communication between the first valve and the condenser for delivering the oxygen gas from the concentrator output to the condenser for cooling to form liquid oxygen;

(f) a storage dewar for storing liquid oxygen liquefied by the condenser;

(g) a third output line in communication between said condenser and storage dewar for delivering liquid oxygen from the condenser to the storage dewar;

(h) a heater for heating liquid oxygen in said dewar;

(i) an oxygen sensor to measure the oxygen concentration of flow from said concentrator;

(j) a liquid level sensor in said dewar;

(k) a temperature sensor in said dewar;

(l) a pressure sensor in communication with said dewar;

(m) a controller for receiving output from the oxygen sensor, liquid level sensor, temperature sensor and pressure sensor, and for controlling the flow of input gaseous oxygen to the condenser and for optimizing liquefaction and transfer from the storage dewar in accordance with the sensed conditions.

74. An ambulatory home oxygen concentrator liquefaction system comprising:

(a) an oxygen concentrator which can isolate oxygen gas from the air;
(b) a first output means from the concentrator to channel the oxygen which is isolated;
(c) a second means to split off all or a portion of the gaseous oxygen from the output means of said concentrator for liquefaction;
(d) a condenser which can cause gaseous oxygen to change phase into liquid oxygen;
(e) means to channel the oxygen stream from said output means to a condenser;
(f) means to liquefy said oxygen stream in said condenser using a cryocooler;
(g) means to collect the condensed liquid oxygen using a dewar;
(h) wherein said dewar includes a heater which can be used to effectuate the transfer of liquid oxygen from the dewar

for storing a quantity of liquid oxygen from which smaller quantities can be transferred for moveable oxygen treatment.

75. A system for liquefying oxygen utilizing an oxygen concentrator, condenser, cryocooler and storage dewar comprising:

(a) a first flow control means for channeling off gaseous fluid flow from an oxygen concentrator;
(b) a second flow control means for advancing said gaseous fluid to a condenser;
(c) means for cooling said condenser to change the phase of said oxygen flow from gas to liquid;
(d) a conduit for transferring the liquid oxygen from the condenser to a storage dewar;
(e) a third flow control means for advancing the flow of liquid oxygen out of the storage dewar;
(f) a controller for sensing the oxygen concentration of flow into the condenser, for sensing the liquid level in the storage dewar, and for controlling said first, second, and third flow control means;
(g) programmable control means in said controller coupled to each of said flow control means, for storing flow delivery and liquefaction parameters for use by

the controller in optimizing liquefaction of oxygen.

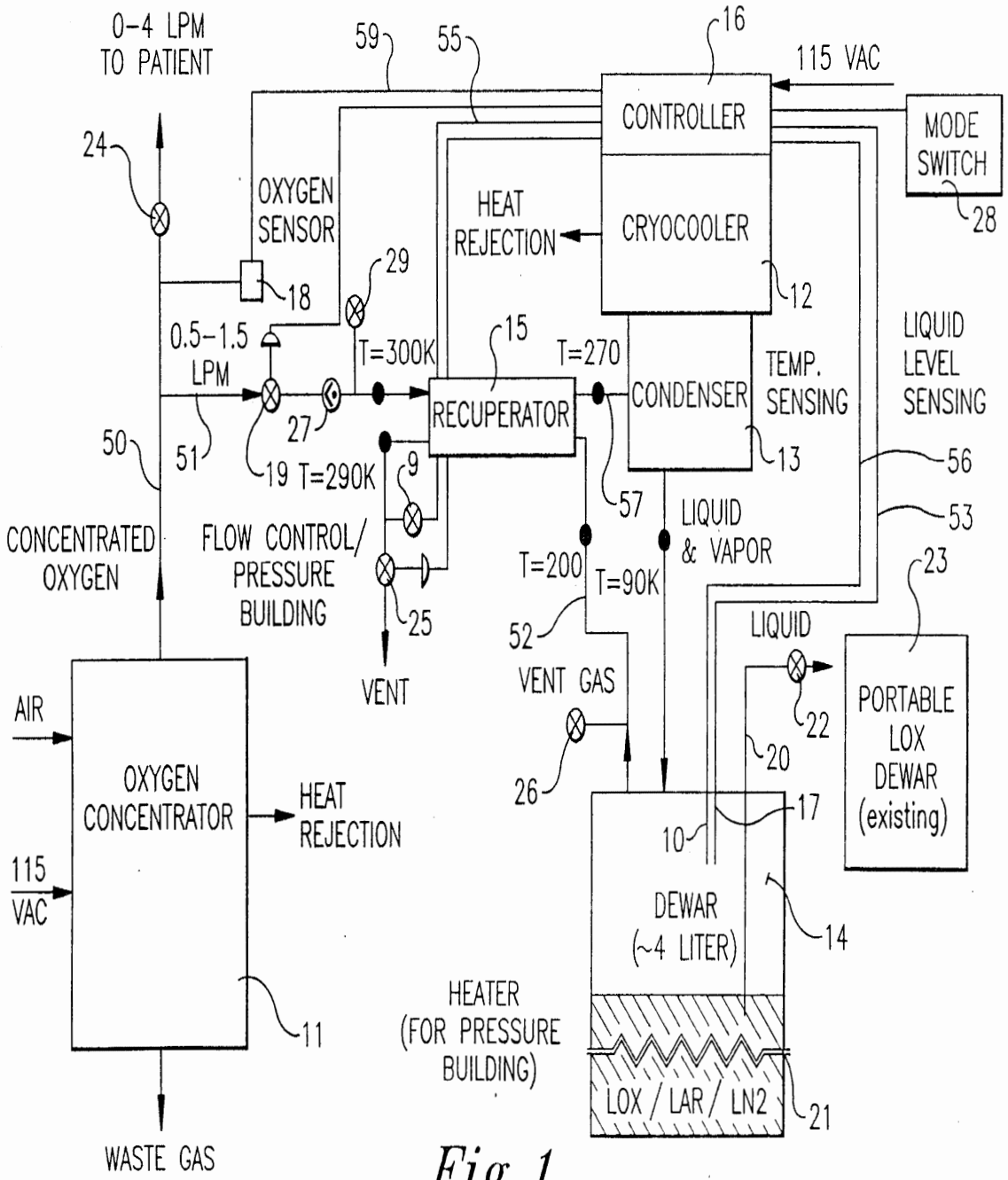

0-4 LPM
TO PATIENT
24
59
55
16
115 VAC
CONTROLLER
MODE
SWITCH
28
OXYGEN
SENSOR
18
HEAT
REJECTION
CRYOCOOLER
12
29
0.5-1.5
LPM
T=300K
15
T=270
LIQUID
LEVEL
SENSING
RECUPERATOR
CONDENSER
TEMP.
SENSING
27
50
51
19 T=290K
9
13
56
57
53
CONCENTRATED
OXYGEN
FLOW CONTROL/
PRESSURE
BUILDING
LIQUID
& VAPOR
T=200
T=90K
23
25
52
LIQUID
VENT
VENT GAS
22
PORTABLE
LOX
DEWAR
(existing)
AIR
OXYGEN
CONCENTRATOR
HEAT
REJECTION
26
20
17
10
115
VAC
DEWAR
(~4 LITER)
14
HEATER
(FOR PRESSURE
BUILDING)
LOX / LAR / LN2
21
11
WASTE GAS

*Fig. 1*

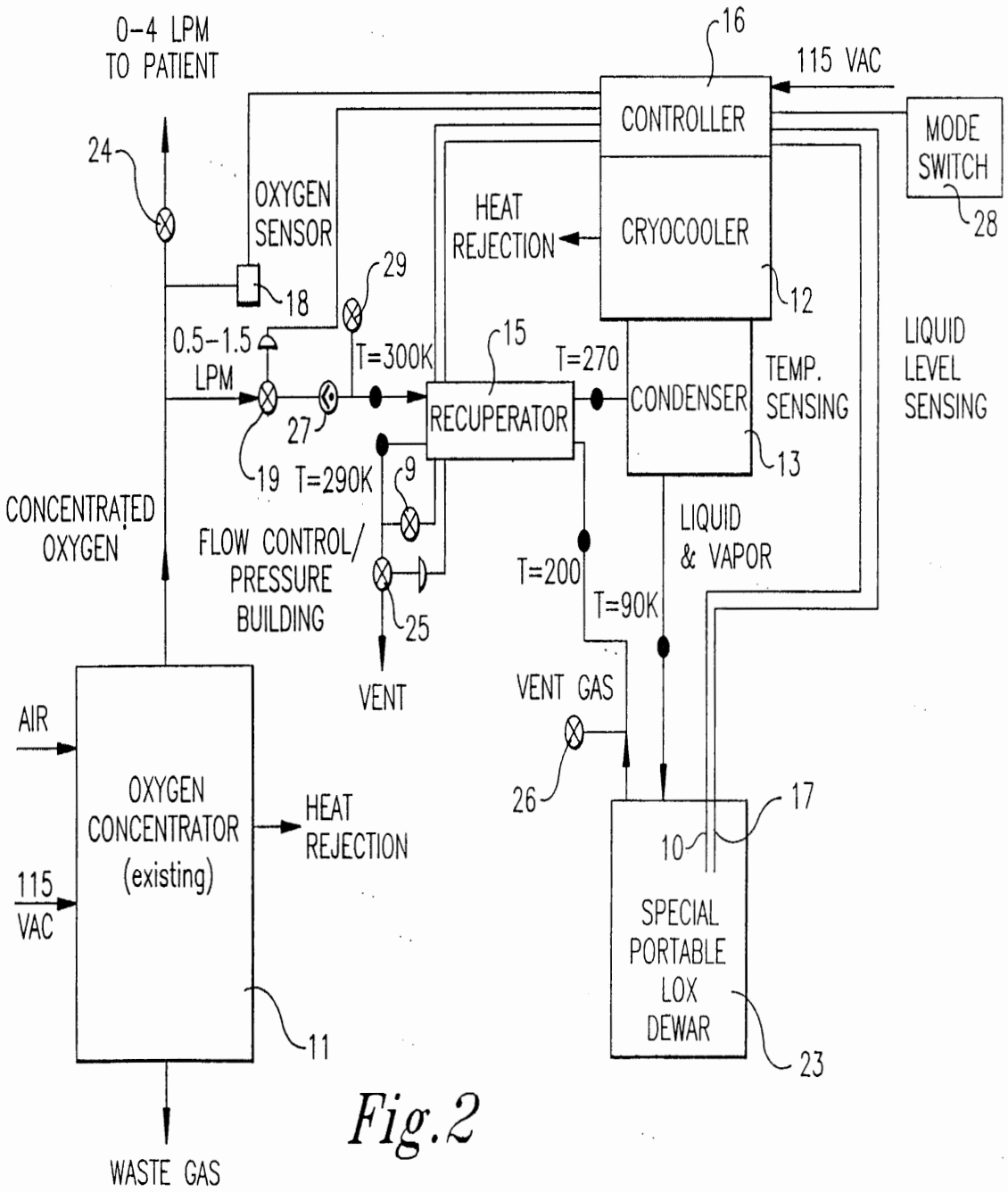
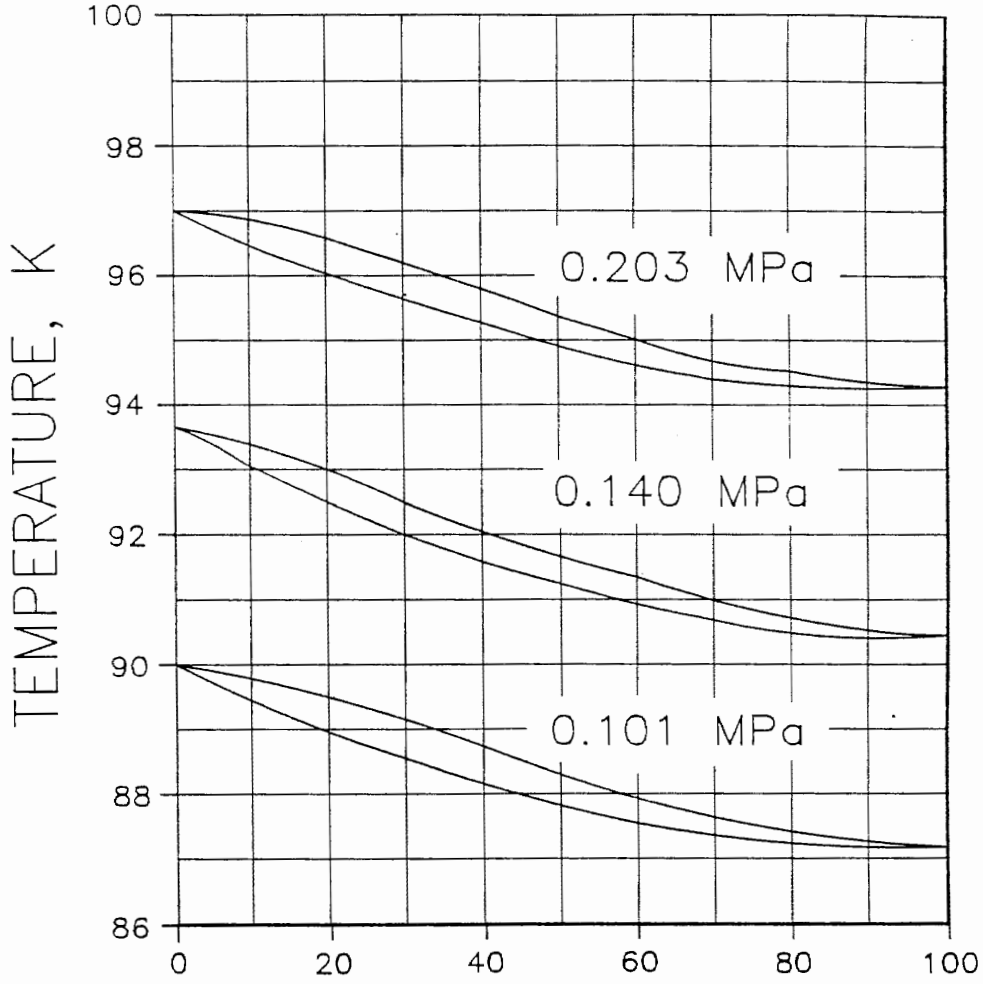
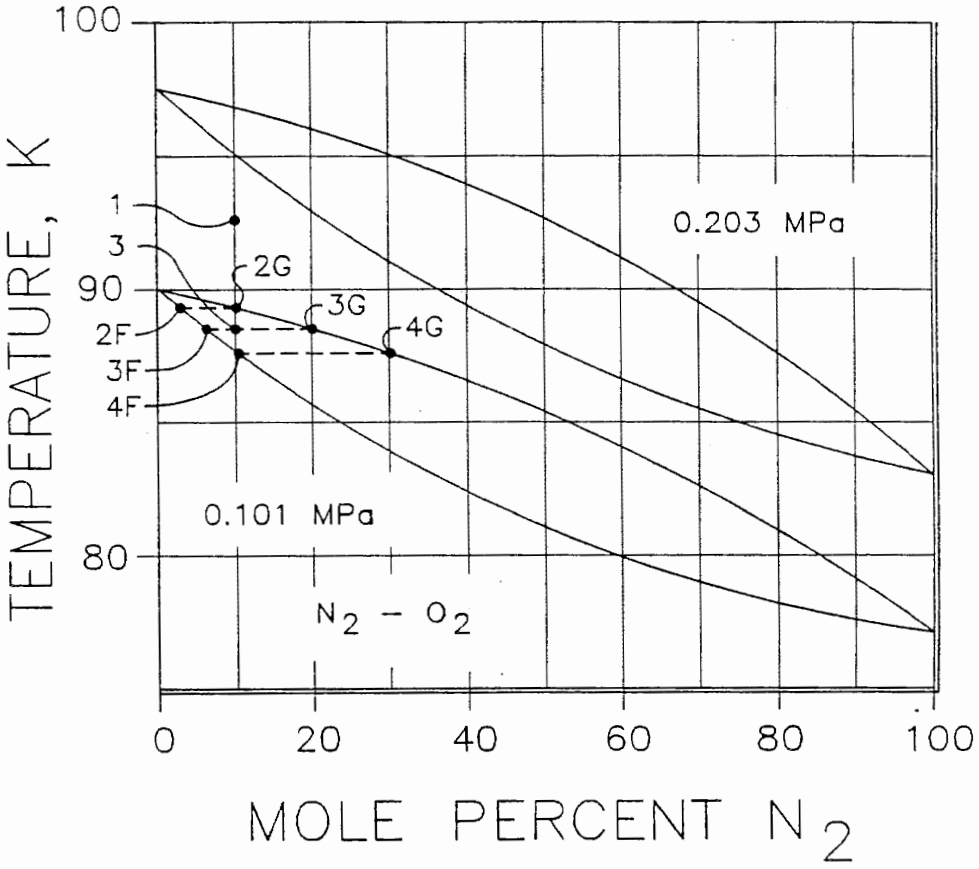

0-4 LPM
TO PATIENT
16
115 VAC
CONTROLLER
MODE
SWITCH
28
24
OXYGEN
SENSOR
HEAT
REJECTION
CRYOCOOLER
12
29
18
0.5-1.5
LPM
T=300K
15
T=270
LIQUID
LEVEL
SENSING
RECUPERATOR
CONDENSER
TEMP.
SENSING
27
19 T=290K
9
13
CONCENTRATED
OXYGEN
FLOW CONTROL/
PRESSURE
BUILDING
LIQUID
& VAPOR
T=200
T=90K
25
VENT
VENT GAS
AIR
OXYGEN
CONCENTRATOR
(existing)
HEAT
REJECTION
26
17
10
115
VAC
SPECIAL
PORTABLE
LOX
DEWAR
11
23
WASTE GAS

*Fig.2*



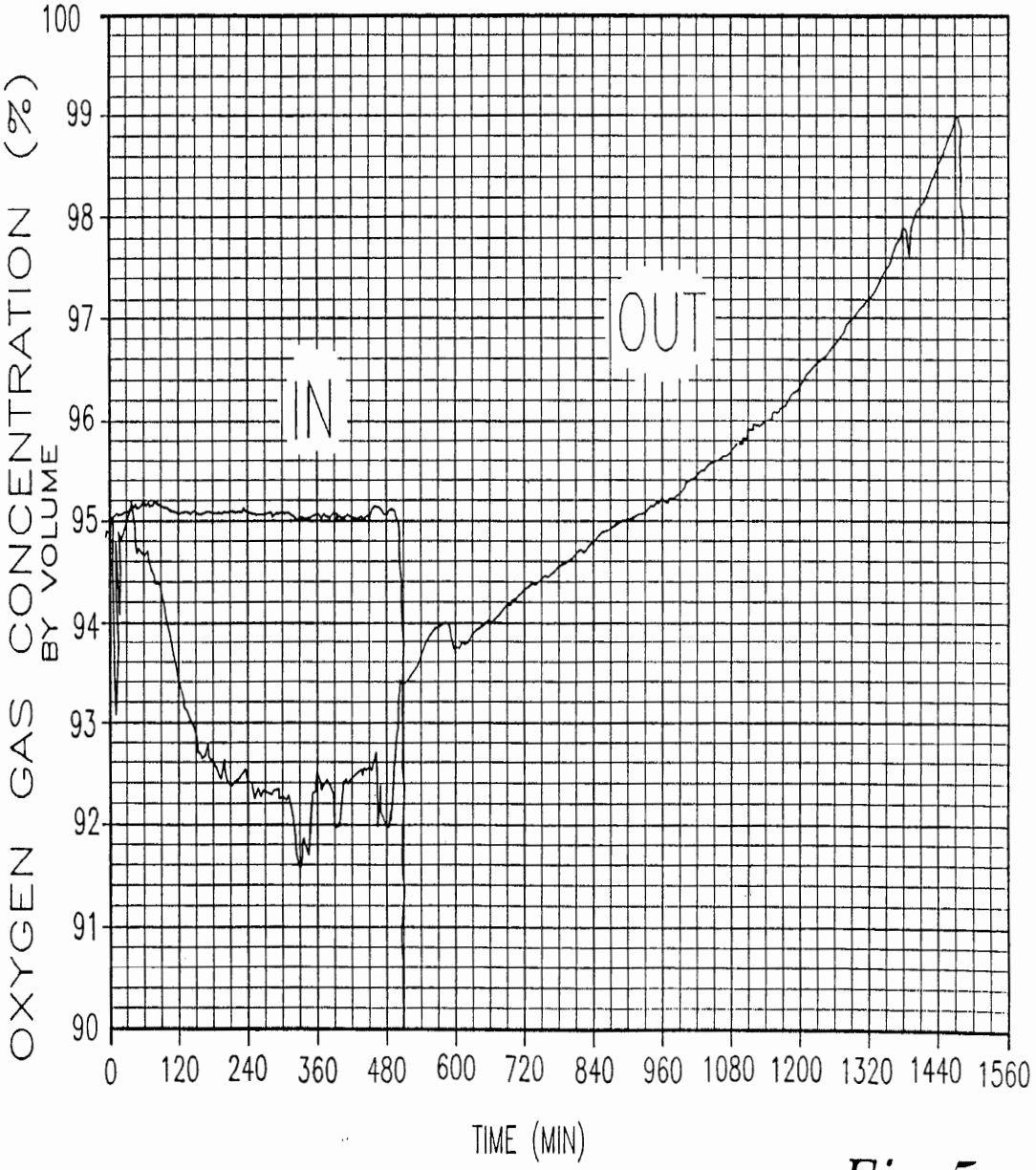

TEMPERATURE – COMPOSITION DIAGRAM
FOR OXYGEN – ARGON MIXTURES

*Fig. 3*



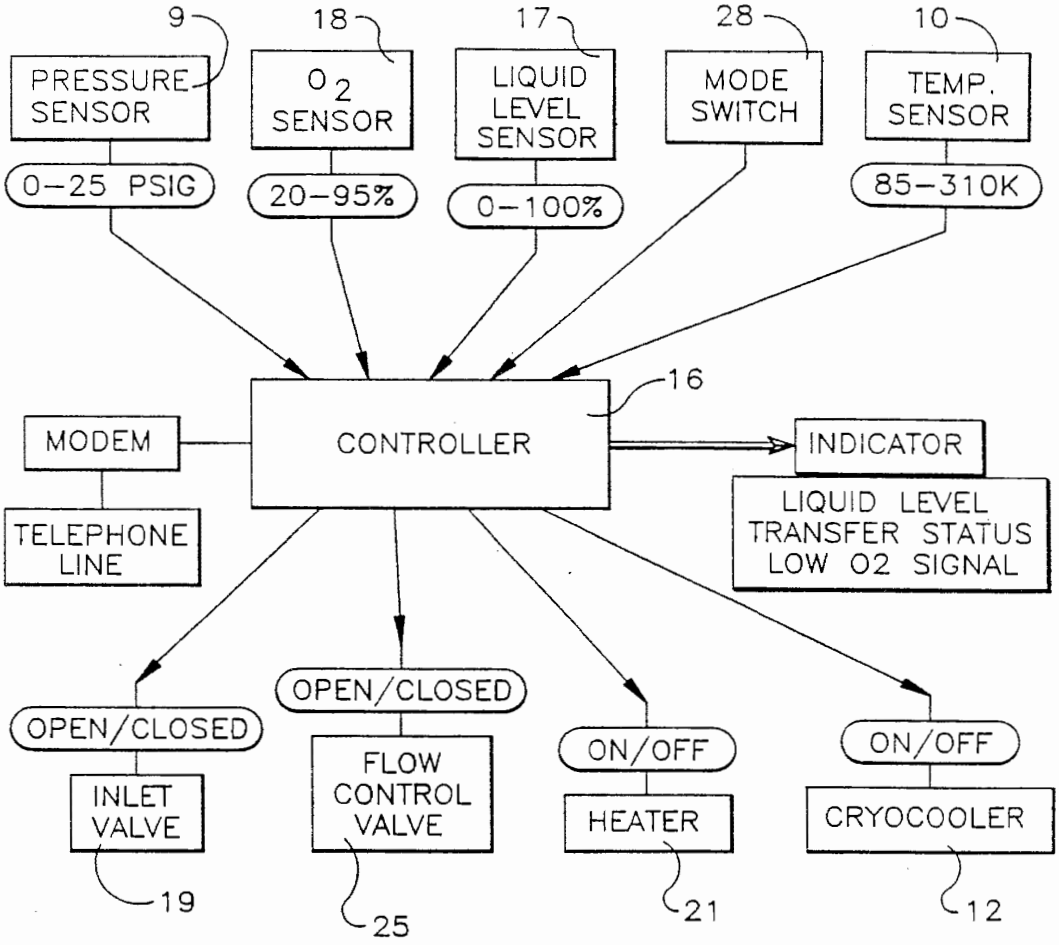

TEMPERATURE COMPOSITION DIAGRAM
FOR NITROGEN OXYGEN MIXTURES

*Fig. 4*




OXYGEN CONCENTRATION TEST DATA

*Fig.5*




CONTROL BLOCK DIAGRAM

*Fig. 6*

*Fig. 7A*

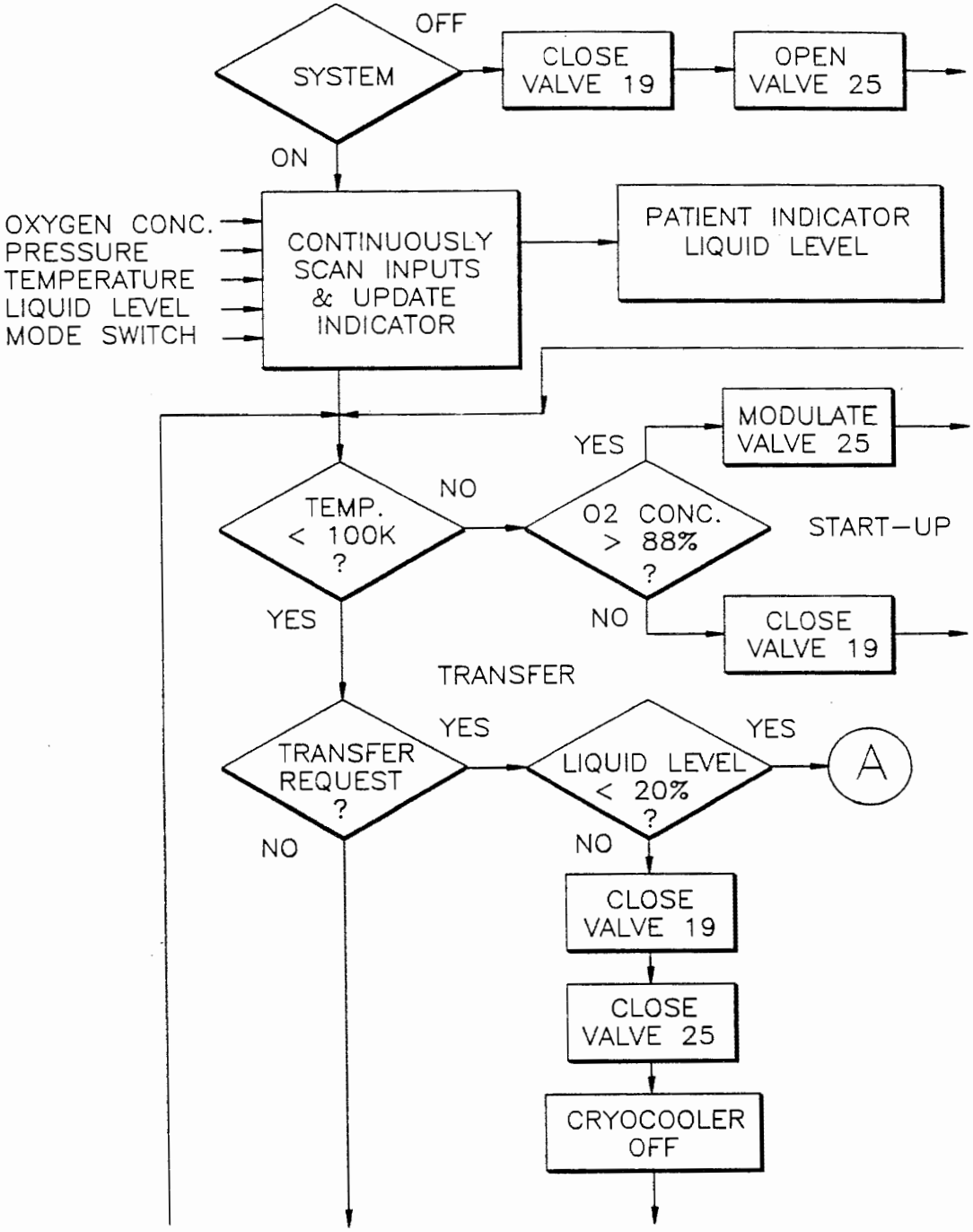
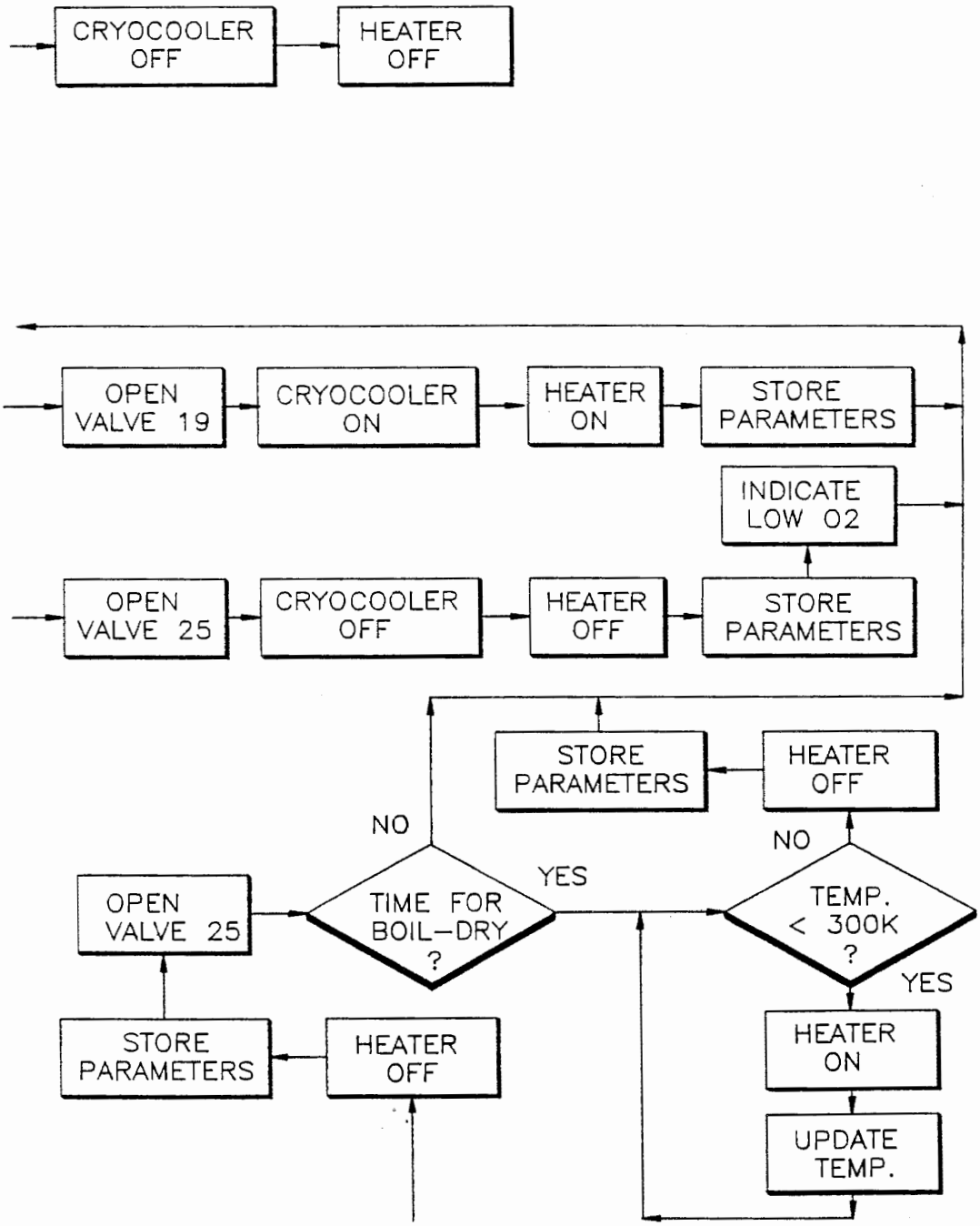

SYSTEM
OFF
CLOSE VALVE 19
OPEN VALVE 25
ON
OXYGEN CONC.
PRESSURE
TEMPERATURE
LIQUID LEVEL
MODE SWITCH
CONTINUOUSLY SCAN INPUTS & UPDATE INDICATOR
PATIENT INDICATOR LIQUID LEVEL
MODULATE VALVE 25
YES
TEMP. < 100K ?
NO
O2 CONC. > 88% ?
START-UP
YES
NO
CLOSE VALVE 19
TRANSFER
TRANSFER REQUEST ?
YES
LIQUID LEVEL < 20% ?
YES
A
NO
NO
CLOSE VALVE 19
CLOSE VALVE 25
CRYOCOOLER OFF

*Fig. 7 B*


CRYOCOOLER OFF
HEATER OFF
OPEN VALVE 19
CRYOCOOLER ON
HEATER ON
STORE PARAMETERS
INDICATE LOW 02
OPEN VALVE 25
CRYOCOOLER OFF
HEATER OFF
STORE PARAMETERS
STORE PARAMETERS
HEATER OFF
NO
YES
NO
OPEN VALVE 25
TIME FOR BOIL-DRY ?
TEMP. < 300K ?
YES
STORE PARAMETERS
HEATER OFF
HEATER ON
UPDATE TEMP.

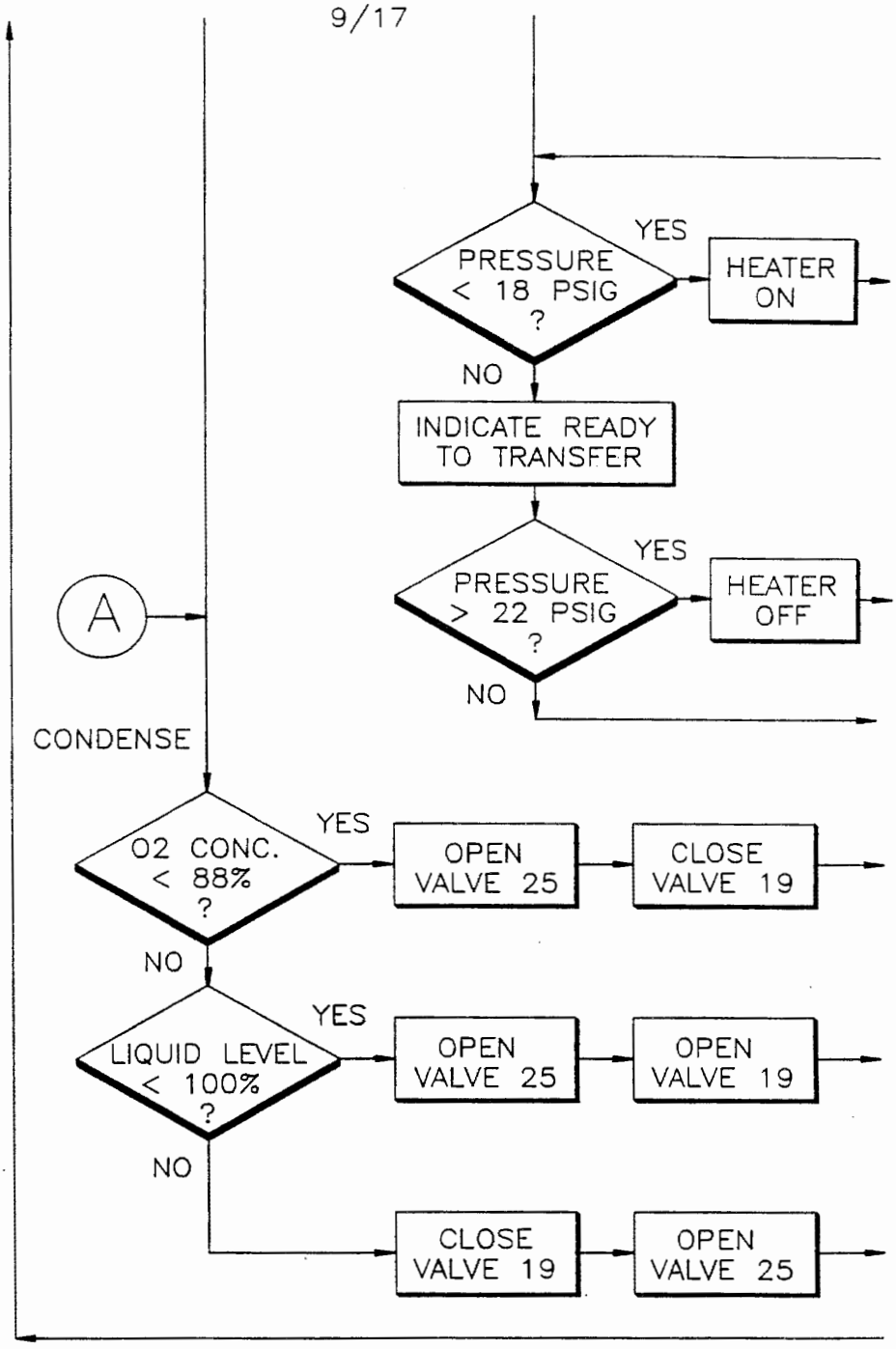

PRESSURE < 18 PSIG ?
YES
HEATER ON
NO
INDICATE READY TO TRANSFER
PRESSURE > 22 PSIG ?
YES
HEATER OFF
NO
A
CONDENSE
O2 CONC. < 88% ?
YES
OPEN VALVE 25
CLOSE VALVE 19
NO
LIQUID LEVEL < 100% ?
YES
OPEN VALVE 25
OPEN VALVE 19
NO
CLOSE VALVE 19
OPEN VALVE 25

*Fig. 7C*

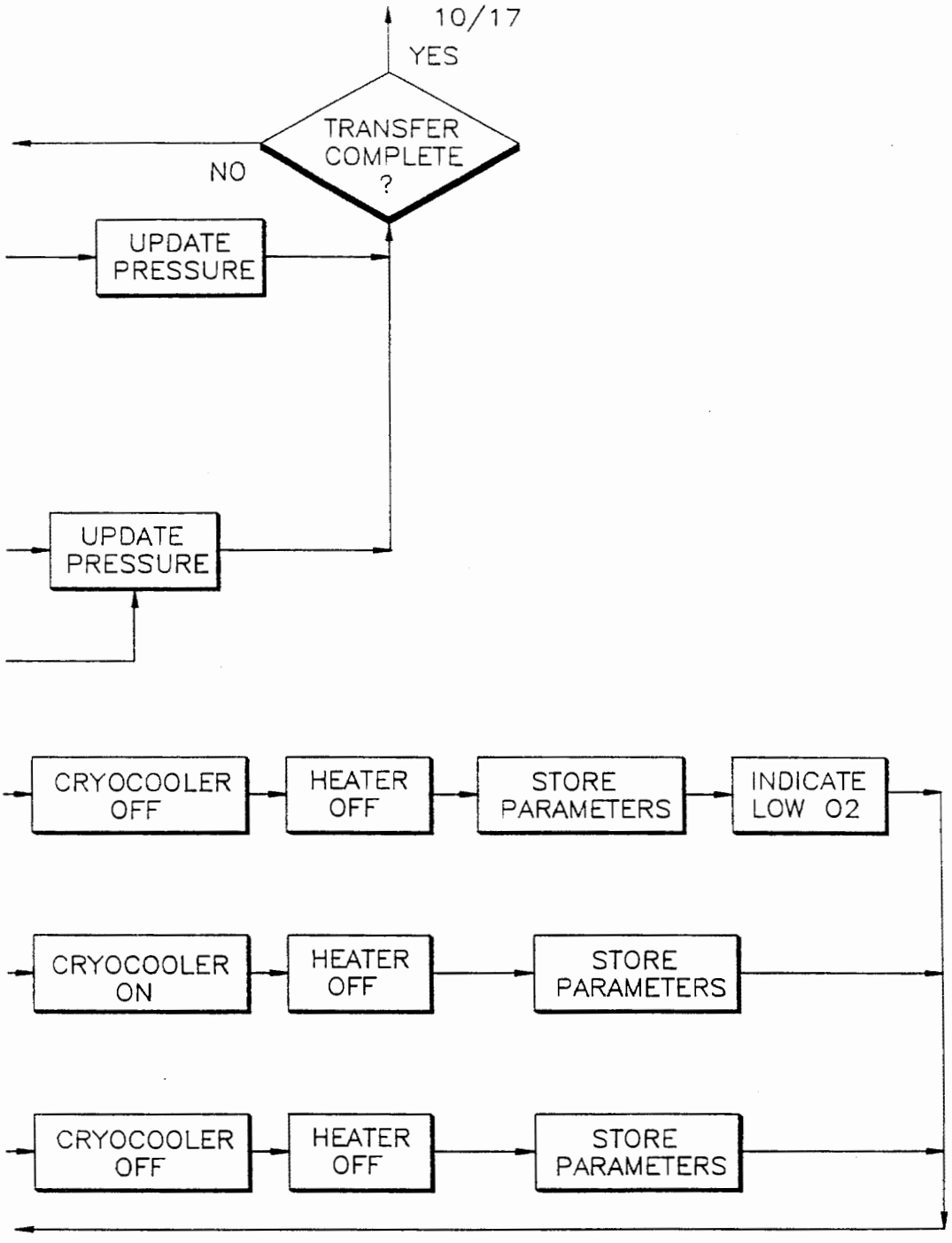

10/17
YES
TRANSFER COMPLETE ?
NO
UPDATE PRESSURE
UPDATE PRESSURE
CRYOCOOLER OFF
HEATER OFF
STORE PARAMETERS
INDICATE LOW 02
CRYOCOOLER ON
HEATER OFF
STORE PARAMETERS
CRYOCOOLER OFF
HEATER OFF
STORE PARAMETERS

*Fig. 7D*

*Fig. 8*

START–UP MODE

INPUT LEVELS & OUTPUT STATES

| INPUT | LEVEL | OUTPUT | STATE |
|---|---|---|---|
| OXYGEN SENSOR | >88% | VALVE 19 | OPEN |
| LIQUID LEVEL SENSOR | 0% | VALVE 25 | OPEN/CLOSED * |
| | | HEATER | OFF |
| | | CRYOCOOLER | ON |
| | | INDICATOR | COOLDOWN |

* MODULATED TO CONTROL PRESSURE

*Fig. 9*

CONDENSE MODE

INPUT LEVELS & OUTPUT STATES

| INPUT | LEVEL | OUTPUT | STATE |
|---|---|---|---|
| OXYGEN SENSOR | >88% | VALVE 19 | OPEN |
| LIQUID LEVEL SENSOR | <100% | VALVE 25 | OPEN |
| | | HEATER | OFF |
| | | CRYOCOOLER | ON |
| | | INDICATOR | CONDENSE |
| OXYGEN SENSOR | ≤88% | VALVE 19 | CLOSED |
| LIQUID LEVEL SENSOR | <100% | VALVE 25 | OPEN |
| | | HEATER | OFF |
| | | CRYOCOOLER | OFF |
| | | INDICATOR | $O_2$ LOW |
| OXYGEN SENSOR | >88% | VALVE 19 | CLOSED |
| LIQUID LEVEL SENSOR | ≥100% | VALVE 25 | OPEN |
| | | HEATER | OFF |
| | | CRYOCOOLER | OFF |
| | | INDICATOR | FULL |

*Fig. 10*

TRANSFER MODE

INPUT LEVELS & OUTPUT STATES

| INPUT | LEVEL | OUTPUT | STATE |
|---|---|---|---|
| OXYGEN SENSOR<br>LIQUID LEVEL SENSOR | 20–95%<br><20% | VALVE 19<br>VALVE 25<br>HEATER<br>CRYOCOOLER<br>INDICATOR | CLOSED<br>OPEN<br>OFF<br>OFF<br>LIQ. LOW |
| OXYGEN SENSOR<br>LIQUID LEVEL SENSOR<br>PRESSURE SENSOR | 20–95%<br>≥20%<br><18 PSIG | VALVE 19<br>VALVE 25<br>HEATER<br>CRYOCOOLER<br>INDICATOR | CLOSED<br>CLOSED<br>ON<br>OFF<br>WAIT |
| OXYGEN SENSOR<br>LIQUID LEVEL SENSOR<br>PRESSURE SENSOR | 20–95%<br>≥20%<br>>18 PSIG | VALVE 19<br>VALVE 25<br>HEATER<br>CRYOCOOLER<br>INDICATOR | CLOSED<br>CLOSED<br>ON<br>OFF<br>TRANSFER |
| OXYGEN SENSOR<br>LIQUID LEVEL SENSOR<br>PRESSURE SENSOR | 20–95%<br>≥20%<br>>22 PSIG | VALVE 19<br>VALVE 25<br>HEATER<br>CRYOCOOLER<br>INDICATOR | CLOSED<br>CLOSED<br>OFF<br>OFF<br>TRANSFER |

*Fig. 11*

BOIL–DRY MODE

INPUT LEVELS & OUTPUT STATES

| INPUT | LEVEL | OUTPUT | STATE |
|---|---|---|---|
| OXYGEN SENSOR<br>LIQUID LEVEL SENSOR<br>TEMPERATURE SENSOR | 20–95%<br><100%<br><300K | VALVE 19<br>VALVE 25<br>HEATER<br>CRYOCOOLER<br>INDICATOR | CLOSED<br>OPEN<br>ON<br>OFF<br>BOIL–DRY |
| OXYGEN SENSOR<br>LIQUID LEVEL SENSOR<br>TEMPERATURE SENSOR | 20–95%<br>0–100%<br>≥300K | VALVE 19<br>VALVE 25<br>HEATER<br>CRYOCOOLER<br>INDICATOR | CLOSED<br>OPEN<br>OFF<br>OFF<br>WARM |

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DOUBLE INLET PULSE TUBE REFRIGERATOR

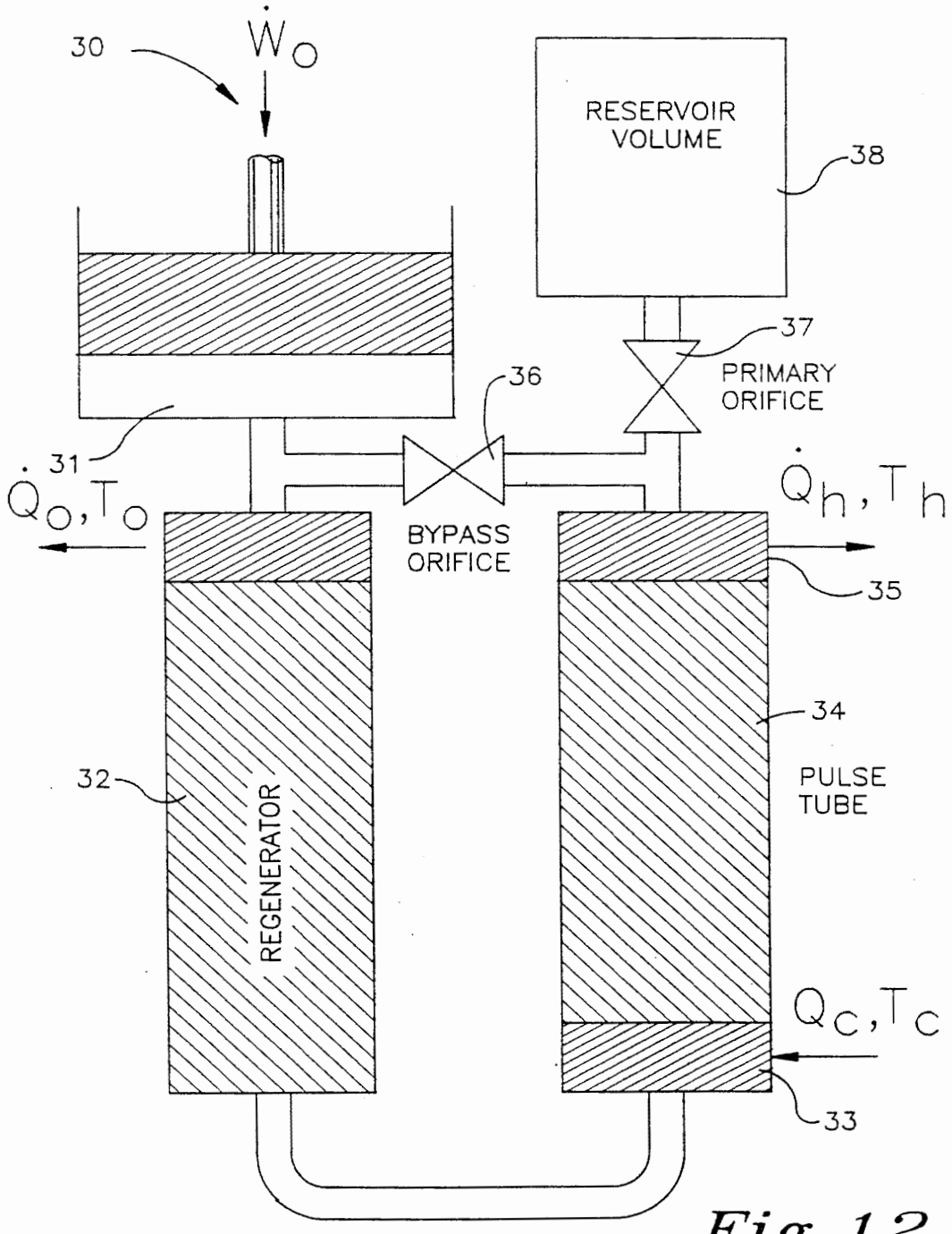



*Fig. 12*

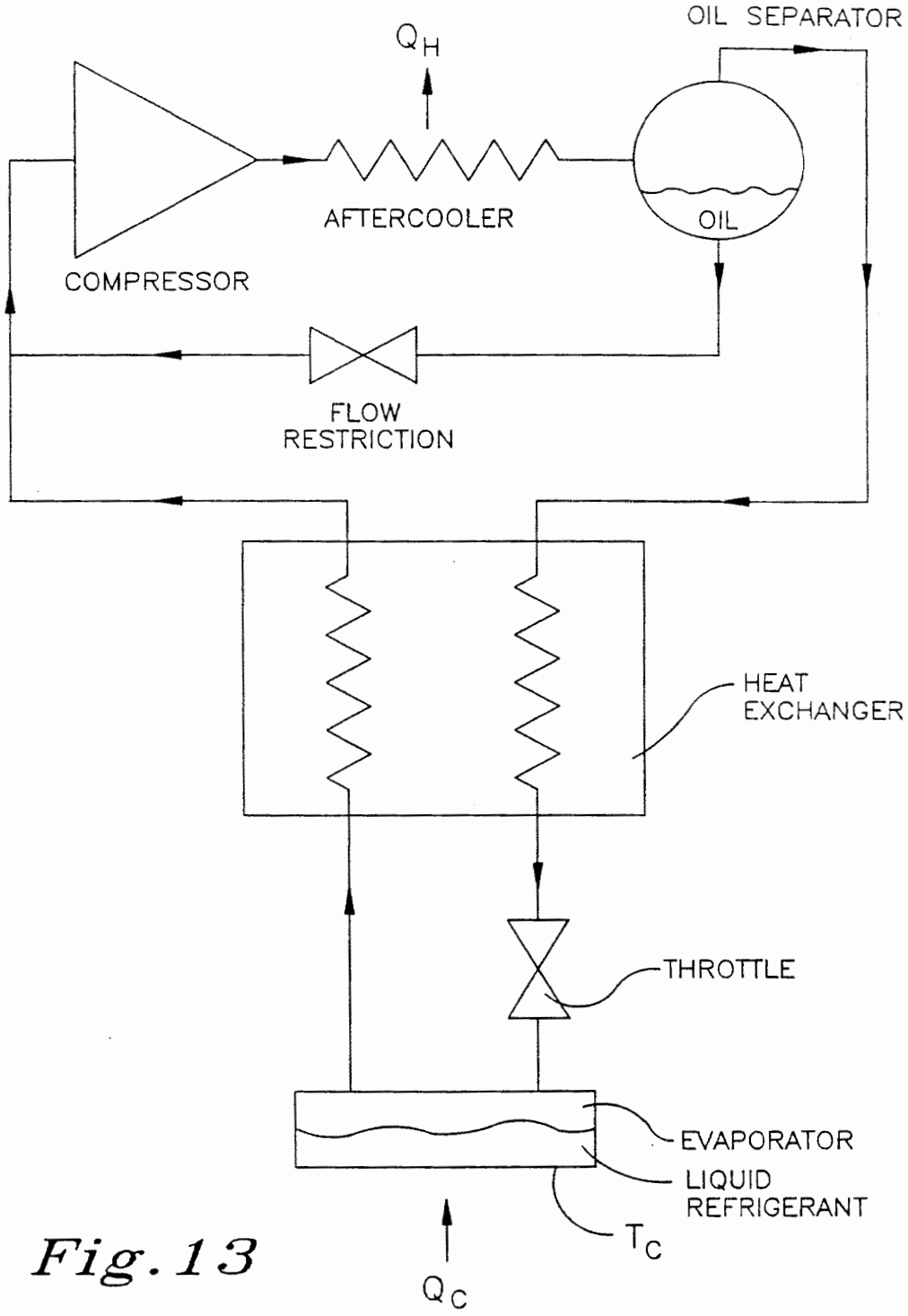
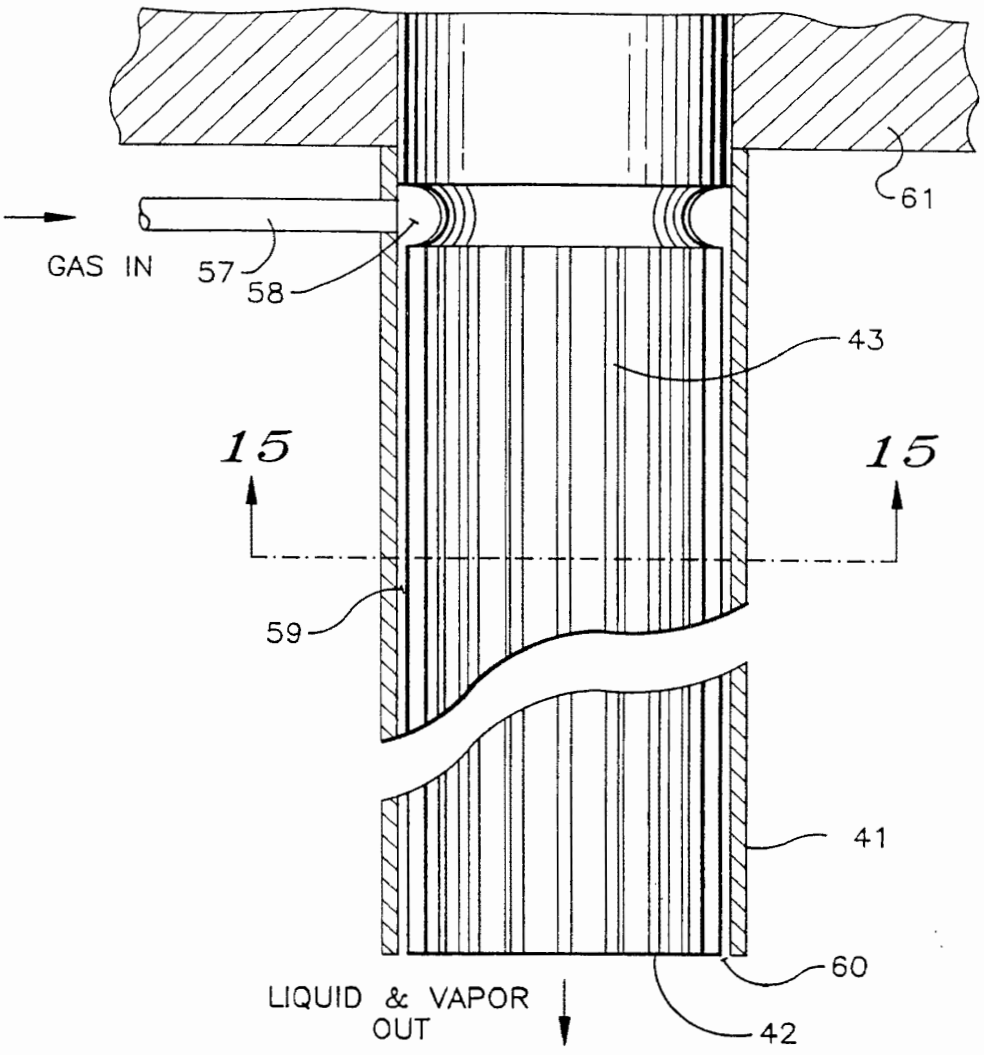

OIL SEPARATOR
$Q_H$
AFTERCOOLER
OIL
COMPRESSOR
FLOW
RESTRICTION
HEAT
EXCHANGER
THROTTLE
EVAPORATOR
LIQUID
REFRIGERANT
$T_C$
$Q_C$

*Fig. 13*

Fig. 14



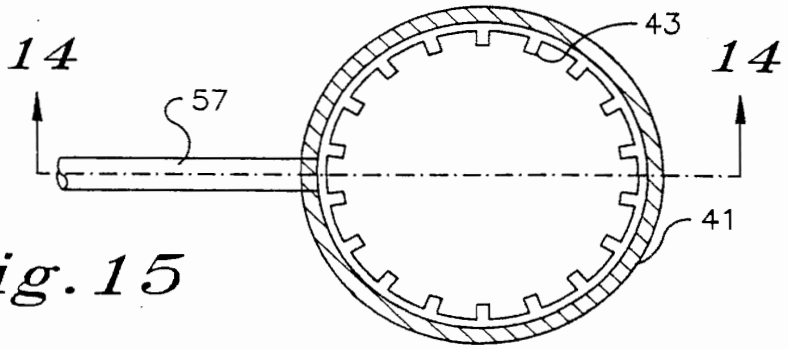




Fig. 15

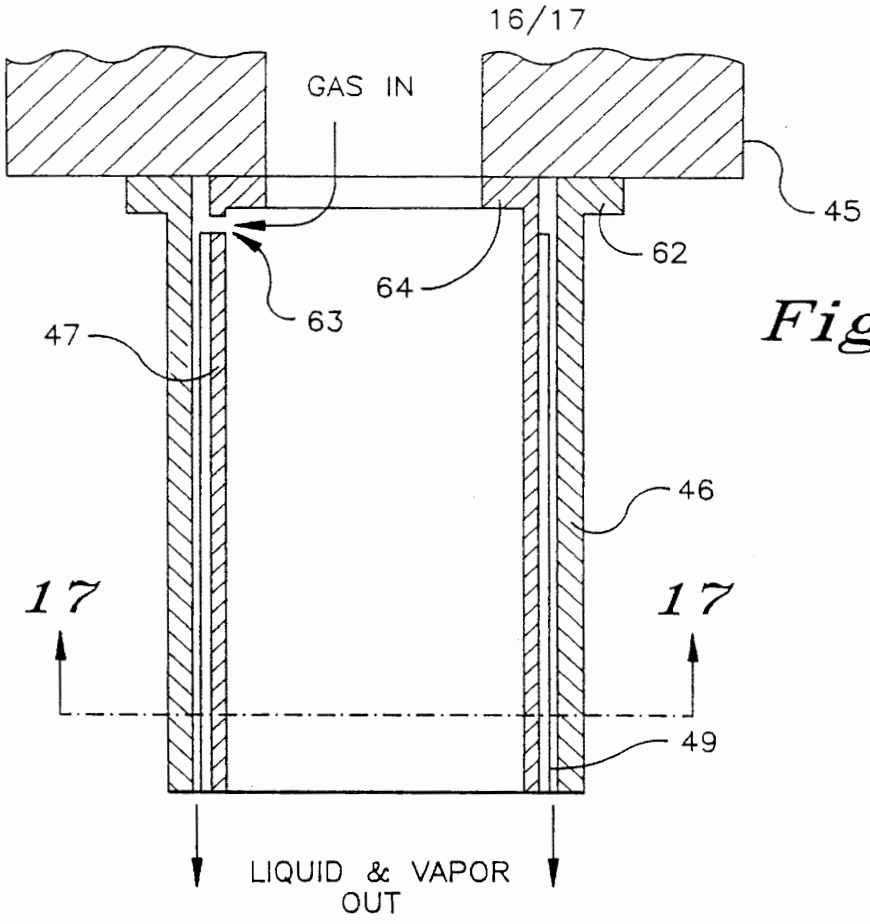

16/17
GAS IN
45
62
64
63
47
46
17
17
49
LIQUID & VAPOR
OUT

Fig. 16

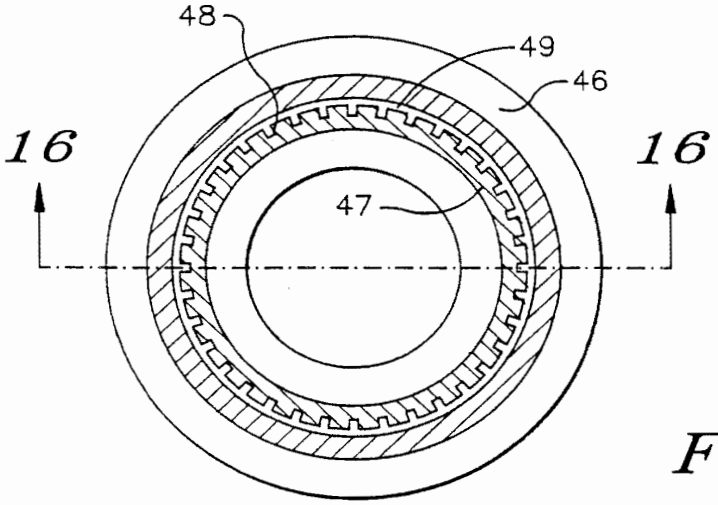

48
49
46
16
16
47

Fig. 17

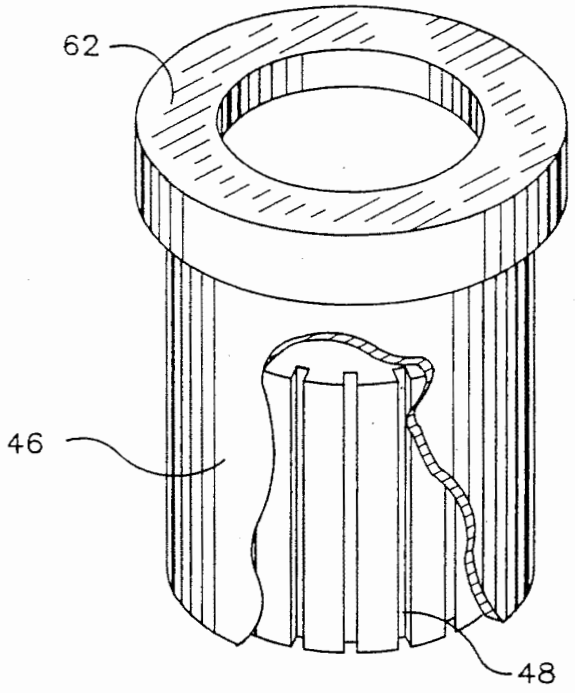



*Fig. 18*

# INTERNATIONAL SEARCH REPORT

Interi nal Application No
PCT/US 98/11154

**A. CLASSIFICATION OF SUBJECT MATTER**
IPC 6 F25J3/06 B01D8/00 B01D5/00 F28D7/10 F25J3/00

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

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Minimum documentation searched (classification system followed by classification symbols)
IPC 6 F25J B01D F28D F28B F28F

Documentation searched other than minimumdocumentation 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**

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|---|---|---|
| A | US 4 870 960 A (HRADEK RICHARD W)<br>3 October 1989<br>--- | 1-48,<br>56-61,<br>73,74 |
| A | US 4 627 860 A (ROWLAND ROBERT O)<br>9 December 1986<br>--- | 1-48,<br>56-61,<br>73,74 |
| A | GB 1 416 163 A (BOC INTERNATIONAL LTD)<br>3 December 1975<br>--- | 49-53,<br>62-72,75 |
| A | US 5 584 194 A (GARDNER THOMAS W)<br>17 December 1996<br>--- | 49-53,<br>62-72,75 |
| A | US 5 163 297 A (YANI MASAYOSHI ET AL)<br>17 November 1992<br>---<br>-/-- | 54,55 |

[X] Further documents are listed in the continuation of box C.

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GCE - Exhibit 1071, Page 313
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

# INTERNATIONAL SEARCH REPORT

Intern al Application No
PCT/US 98/11154

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

| Category ° | Citation of document, with indication,where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| A | US 4 211 086 A (HULSTYN JAN ET AL)<br>8 July 1980<br>--- | |
| A | US 4 971 609 A (PAWLOS ROBERT A)<br>20 November 1990<br>cited in the application<br>--- | |
| A | US 4 826 510 A (MCCOMBS NORMAN R)<br>2 May 1989<br>cited in the application<br>----- | |

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GCE - Exhibit 1071, Page 314
GCE Gas Control Equipment Inc. v. VBOX, Inc.
IPR2023-00527

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Information on patent family members

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PCT/US 98/11154

| Patent document cited in search report | Publication date | Patent family member(s) | Publication date |
|---|---|---|---|
| US 4870960 A | 03-10-1989 | NONE | |
| US 4627860 A | 09-12-1986 | US 4516424 A | 14-05-1985 |
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| | | JP 1183402 A | 21-07-1989 |

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(30) Priority Data:
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(63) Related by Continuation (CON) or Continuation-in-Part (CIP) to Earlier Application
US 60/076,263 (CIP)
Filed on 27 February 1998 (27.02.98)

(71) Applicant *(for all designated States except US)*: PRAXAIR TECHNOLOGY, INC. [US/US]; 39 Old Ridgebury Road M1–557, Danbury, CT 06810–5113 (US).

(72) Inventors; and
(75) Inventors/Applicants *(for US only)*: ACKLEY, Mark, William [US/US]; 96 Church Street, East Aurora, NY 14052 (US). LEAVITT, Frederick, Wells [US/US]; 114 Sundridge Drive #4, Amherst, NY 14228 (US).

(74) Agents: BIEDERMAN, Blake, T. et al.; Praxair, Inc., 39 Old Ridgebury Road M1–557, Danbury, CT 06810–5113 (US).

(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

**Published**
*With international search report.*

(54) Title: RATE–ENHANCED GAS SEPARATION

(57) Abstract

The invention is based upon the recognition that intrinsic sorption rates of adsorbent materials have a significant impact upon process performance. In a preferred embodiment an adsorption process uses an adsorbent zone comprising an adsorbent selected from the group consisting of: A–zeolite, Y–zeolite, NaX, mixed cation X–zeolite, LiX having a $Sio_2/A1_2O_3$ silica/alumina ratio of less than 3.0, chabazite, mordenite, clinoptilolite, silica–alumina, alumina, silica, titanium silicates and mixtures thereof, wherein said adsorbent has a mass transfer coefficient for nitrogen of $k_{N2} \geq 12\ s^{-1}$ and an intrinsic diffusivity for $N_2$, when measured at 1.5 bar and 300K, of $D_p \geq 3.5 \times 10^{-6}\ m^2/s$. Other embodiments include the development of process parameters around which such materials should be used and methods of making such materials.

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# RATE-ENHANCED GAS SEPARATION

## FIELD OF THE INVENTION

The invention relates to pressure swing adsorption processes and more particularly to PSA processes for the production of high purity oxygen (e.g. oxygen having a purity of 90-95 vol. $\%O_2$). More particularly, the invention is directed towards increasing the adsorbent productivity while reducing the $O_2$ product cost.

## BACKGROUND OF THE INVENTION

There has been significant development of the various PSA, VSA and VPSA methods for air separation over the past thirty years, with major advances occurring during the last decade. Commercialization of these processes and continued extension of the production range can be attributed primarily to improvements in the adsorbents and process cycles, with advances in adsorber design contributing to a lesser degree. Highly exchanged lithium molecular sieve adsorbents, as illustrated by Chao in U.S. Pat. No. 4,859,217, are representative of advanced adsorbents for oxygen production. Advanced adsorbents of the types mentioned above are the result of improvements in equilibrium properties.

Improving process efficiency and reducing the cost of the light component product can be accomplished by decreasing the amount of adsorbent required and by increasing the product recovery. The former is generally expressed in terms of bed size factor (BSF) in lbs adsorbent/TPDO (ton per day of contained $O_2$), while the latter is simply the fraction of light component (i.e. oxygen) in the feed (i.e. air) that is captured as

product. Improvement in adsorbents and reduction in cycle time are two primary methods of reducing BSF.

While shorter cycles lead to shorter beds and higher adsorbent utilization, product recovery generally suffers unless adsorption rate is increased. This phenomena can be ideally characterized in terms of the size of the mass transfer zone (MTZ), i.e. the mass transfer zone becomes an increasing fraction of the adsorbent bed as the bed depth decreases. Since the adsorbent utilization with respect to the heavy component (e.g. nitrogen) is much lower in the MTZ than in the equilibrium zone, working capacity declines as this fraction increases.

The effect of particle size upon the size of the MTZ is conceptually straightforward in a single long adsorption step where a contaminant in relatively low concentration is removed from the feed stream on the basis of its higher equilibrium affinity to the adsorbent. When the adsorbate/adsorbent combination is characterized by a favorable isotherm, a steady state transfer zone is envisioned that moves through the adsorber at a constant speed. Distinct equilibrium and mass transfer zones can be identified in the process. Under such conditions, and when the resistance to mass transfer is dominated by intraparticle pore diffusion, it has long been recognized that reducing the adsorbent particle size results in higher adsorption rates and smaller mass transfer zones. Unfortunately, pressure drop across the adsorbent bed increases with decreasing particle size and leads to difficulty in particle retention in the bed and an increased tendency for fluidization.

This ideal concept becomes blurred when the isotherms are unfavorable and/or the mass transfer zone is continuously developing or spreading throughout the adsorption step. Adding the remaining minimum steps of depressurization, desorption and pressurization to create a complete adsorption process cycle further complicates the behavior and character of the mass transfer zone. Nevertheless, the idealized concept of the MTZ has been applied in the prior art as a basis to affect improvements in process performance.

Jain (U.S. Pat. No. 5,232,474) discloses improving adsorbent utilization by decreasing the adsorbent volume and/or increasing the product purity, wherein the removal of $H_2O$ and $CO_2$ prior to cryogenic air separation is described using a pressure swing adsorption (PSA) process. The adsorber is configured entirely with alumina or with layers of alumina and 13X molecular sieve adsorbents. Smaller particles (0.4mm to 1.8mm) are used to achieve a smaller bed volume.

Umekawa (JP Appl. No. 59004415) shows a lower pressure drop and smaller adsorber for air purification by using a deep layer of large particles (3.2mm) followed by a shallow layer of small particles (1.6mm) of the same adsorbent. The bed size and pressure drop of this layered configuration are lower than for beds constructed either of all 3.2mm or all 1.6mm particles. The 1.6mm particles occupy only a small fraction (low concentration part) of the mass transfer zone in the layered configuration.

Miller (U.S. Pat. No. 4,964,888) has suggested using larger particles (>14 mesh or 1.41mm) in the equilibrium zone and small particles ( <14 mesh) in the mass transfer

zone. This reduces the size of the MTZ while minimizing the excessive pressure drop increase that would occur if only small particles were used in both zones. Cyclic adsorption process times greater than 30s are indicated.

Garrett (UK Pat. Appl. GB 2 300 577) discloses an adsorption apparatus containing particles in the size range between 6 mesh (3.36mm) and 12 mesh (1.68mm) deployed in either discrete layers or as a gradient of sizes with the largest particles located near the feed inlet and the smallest particles located downstream near the outlet of the adsorber in both configurations.

Very small adsorbent particles (0.1mm to 0.8mm) are necessary for the fast cycles and high specific pressure drop that characterize a special class of processes known as rapid pressure swing adsorption (RPSA). Typical RPSA processes have very short feed steps (often less than 1.0s) operating at high feed velocities, include a flow suspension step following the feed step and generally have total cycle times less than 20s (often less than 10s). The behavior of the adsorption step is far removed from the idealized MTZ concept described above. In fact, the working portion of the bed is primarily mass transfer zone with only a relatively small equilibrium zone (in equilibrium with the feed conditions) in RPSA. A major portion of the adsorber is in equilibrium with the product and provides the function of product storage. The high pressure drop (on the order of 12psi/ft)/short cycle combination is necessary to establish an optimum permeability and internal purging of the bed which operates continuously to generate product.

RPSA air separation processes using 5A molecular sieve have been described by Jones et al. (U.S. Pat. No.

4,194,892) for single beds and by Earls et al. (U.S. Pat. No. 4,194,891) for multiple beds. Jones has also suggested RPSA for $C_2H_4/N_2$, $H_2/CH_4$, $H_2/CO$ and $H_2/CO/CO_2/CH_4$ separations using a variety of adsorbents. The RPSA system is generally simpler mechanically than conventional PSA systems, but conventional PSA processes typically have lower power, better bed utilization and higher product recovery.

In somewhat of a departure from the original RPSA processes, Sircar (U.S. Pat. No. 5,071,449) discloses a process associated with a segmented configuration of adsorbent layers contained in a single cylindrical vessel. One or more pairs of adsorbent layers are arranged such that the product ends of each layer in a given pair face each other. The two separate layers of the pair operate out of phase with each other in the cycle. The intent is for a portion of the product from one layer to purge the opposing layer - the purge fraction controlled by either a physical constriction placed between the layers and/or by the total pressure drop across a layer (ranging from 200 psig to 3 psig). Particles in the size range of 0.2mm to 1.0mm, total cycle times of 6s to 60s, adsorbent layer depths of 6 inches to 48 inches and feed flow rates of one to 100 lbmoles/hr/ft$^2$ are broadly specified. An optional bimodal particle size distribution is suggested to reduce interparticle void volume. The process is claimed to be applicable to air separation, drying, and $H_2/CH_4$, $H_2/CO$ and $H_2/CO/CO_2/CH_4$ separations.

Alpay et al. (Chem. Eng. Sci., 1994) studied the effects of feed pressure, cycle time, feed step time/cycle time ratio and product delivery rate in RPSA

air separation for several ranges of particle sizes (0.15mm to 0.71mm) of 5A molecular sieve. His study showed that process performance was limited when adsorbent particles were either too small or too large. This was because ineffective pressure swing, low permeability and high mass transfer resistance (due to axial dispersion) were limiting at the lower end of particle size range, while high mass transfer resistance became limiting due to the size of the particles at the larger end of the particle size spectrum. Alpay found maximum separation effectiveness (maximum $O_2$ purity and adsorbent productivity) for particles in the size range 0.2mm to 0.4mm.

RPSA is clearly a special and distinct class of adsorption processes. The most distinguishing features of RPSA compared to conventional PSA can be described with respect to air separation for $O_2$ production. The pressure drop per unit bed length is an order of magnitude or more larger and the particle diameter of the adsorbent is usually less than 0.5mm in RPSA. Total cycle times are typically shorter and the process steps are different in RPSA. Of these contrasting features, pressure drop and particle size constitute the major differences.

Other patents suggest the use of small particles in conventional PSA processes. Armond et al. (UK Pat. Appl. GB 2 091 121) discloses a superatmospheric PSA process for air separation in which short cycles ($\leq$45s) are combined with small particles (0.4mm to 3.0mm) to reduce the process power and the size of the adsorbent beds. Oxygen of 90% purity is produced under the preferred

cycle times of 15s to 30s and particle sizes of 0.5mm to 1.2mm.

Hirooka et al. (U.S. Pat. No. 5,122,164) describes 6, 8 and 10-step VPSA processes for separating air to produce $O_2$. While the main thrust of this patent is the cycle configuration and detailed operation of the various cycle steps to improve yield and productivity, Hirooka utilizes small particles to achieve faster cycles. A broad particle range is specified (8x35 US mesh or 0.5mm to 2.38mm), but 12x20 US mesh or 0.8mm to 1.7mm is preferred. Half-cycle times of 25s to 30s are indicated (total cycle times of 50s to 60s).

Hay et al. (U.S. Pat. No. 5,176,721) also discloses smaller particles to produce shorter cycles, preferably in air separation. A vertical vessel with horizontal flow across the adsorbent is depicted. Broad range characteristics include particles less than 1.7mm diameter, cycle times between 20s - 60s and pressure drop across the adsorbent less than 200mb (2.85psig).

An alternative configuration includes an upstream portion of the adsorbent bed with particles of size greater than 1.7mm, in which case the particle fraction smaller than 1.7mm comprises 30% to 70% of the total adsorbent mass. The aspect ratio of the bed (largest frontal length to bed depth ratio) is specified to be between 1.5 and 3.0. Small particle fraction alternatives of 0.8mm to 1.5mm and 0.4mm to 1.7mm are also given, as well as adsorbent pressure drop as low as 50mbar (0.7 psig).

Wankat (CRC Press, 1986; Ind. Eng. Chem. Res., 1987) describes a concept that he terms "intensification" whereby decreased particle diameter is employed to

produce shorter columns and faster cycles. By non-dimensionalizing the governing mass balance equations for the adsorption process, a set of scaling rules are suggested which preserve the performance of the process in terms of product recovery, purity and pressure drop while increasing the adsorbent productivity. These theoretical results are based upon the similarity of dynamic adsorption behavior (at the same dimensionless times and column locations). The similarity concept presumes an idealized constant pattern MTZ, with the length of the mass transfer zone ($L_{MTZ}$) directly proportional to the square of the particle diameter when pore diffusion is controlling. Furthermore, decreasing $L_{MTZ}$ increases the fraction of bed utilized. Wankat indicates that increasing ($L/L_{MTZ}$) beyond a value of two to three, where L is the bed depth, results in minimal improvement in the fractional bed utilization. A layer of small-size particles placed on top of a layer of large-size particles is also suggested as a way to sharpen the mass transfer front. Some of the practical limitations to smaller scale and faster operation have been noted and include fluidization, column end effects, wall channeling and particle size distribution. The intensification concept was later extended to include non-isothermal and non-linear equilibrium effects in PSA processes by Rota and Wankat (AIChE J., 1990).

Moreau et al. (U.S. Pat. No. 5,672,195) has suggested higher porosity in zeolites to achieve improved $O_2$ yield and throughput in PSA air separation. A preferred porosity range of 0.38 to 0.60 is claimed in conjunction with a minimum rate coefficient. Moreau states that commercially available zeolites are not

suitable for their invention since porosity is lower than 0.36.

Lu et al.(Sep. Sci. Technol. 27, 1857-1874 (1992); Ind. Eng. Chem. Res. 32: 2740-2751 (1993)) have investigated the effects of intraparticle forced convection upon pressurization and blowdown steps in PSA processes. Intraparticle forced convection augments macropore diffusion in large-pore adsorbents where the local pressure drop across the particle is high and where the pores extend completely through the particle. The higher intraparticle permeability is associated with high particle porosity, e.g. porosities ($\varepsilon_p$)= 0.7 & 0.595.

## OBJECTS OF THE INVENTION

It is therefore an object of the invention to increase efficiency, reduce cost and extend the production range of high performance adsorption processes for the separation of gases.

It is a further object of the invention to increase efficiency, reduce cost and extend the production range of high performance adsorption processes for production of oxygen.

## SUMMARY OF THE INVENTION

The invention is based upon the recognition that intrinsic sorption rates, in particular the effective macropore diffusivity, of adsorbent materials have a significant impact upon process performance. In a preferred embodiment an adsorption process uses an adsorbent zone comprising an adsorbent selected from the group consisting of A-zeolite, Y-zeolite, NaX, mixed cation X-zeolite, LiX having a $SiO_2/Al_2O_3$ ratio of less

than 2.30, chabazite, mordenite, clinoptilolite, silica-alumina, alumina, silica, titanium silicates and mixtures thereof, wherein said adsorbent has a mass transfer coefficient for nitrogen of $k_{N2} \geq 12\ s^{-1}$ and an intrinsic diffusivity for $N_2$, when measured at 1.5 bar and 300K, of $D_p \geq 3.5 \times 10^{-6}\ m^2/s$.

Other preferred embodiments include the development of process parameters around which such materials should be used and preferred methods for increasing effective diffusivity.

## BRIEF DESCRIPTION OF THE DRAWINGS

Other objects, features and advantages will occur to those skilled in the art from the following description of preferred embodiments and the accompanying drawings in which:

Fig. 1 is a schematic diagram showing the apparatus used to measure intrinsic adsorption rate.

Fig. 2 is a graph showing the effect of intrinsic diffusivity and mass transfer rate coefficient upon product recovery and bed size factor for a 60s cycle;

Fig. 3 is a graph showing the variation of mass transfer coefficient with particle size for various levels of intrinsic diffusivity;

Fig. 4 is a schematic showing the eight step VPSA cycle used in the examples of the invention;

Fig. 5 is a graph showing VPSA performance for 15s and 60s cycles as a function of nitrogen mass transfer coefficient using LiX adsorbent;

Fig. 6 is a graph showing the effect of particle size and cycle time upon VPSA performance at a fixed intrinsic diffusivity of $N_2$; and

Fig. 7 is a graph showing preferred cycle time/particle size combinations for various $N_2$ intrinsic diffusivities.

## DETAILED DESCRIPTION OF THE INVENTION

The invention is based upon the recognition that intrinsic sorption rates of adsorbent materials have a significant impact upon process performance, the dominant intrinsic factor being the effective macropore diffusivity.

The objects of the invention are accomplished by implementing higher rates of mass transfer and by combining these with fast cycles and shallow beds. The preferred adsorption rate is established through a combination of the internal physical or intrinsic mass transfer rate properties of the adsorbent particle and the particle size in such a way to achieve significantly improved overall process performance. By the term "sorption rate" we mean the rate at which the adsorbate loading changes in a given time period in an adsorbent particle for a given adsorption separation process. This sorption rate is approximately proportional to the inverse of particle radius squared and is directly proportional to the "effective diffusivity" (also referred to as the "intrinsic diffusivity "). An increase in effective diffusivity results in an increase in adsorption rate. By the term "intrinsic diffusivity" we mean the transport property that is due to the intrinsic characteristics of the adsorbent particle including, but not limited to the structure, size, shape and length, etc. of the macropores. Ideally, a material's intrinsic or effective diffusivity is

independent of particle size. In a preferred embodiment, the adsorption rate increases as the cycle time and bed depth decrease.

In practicing the invention, improved process efficiency is obtained by first affecting the largest sorption rate of the adsorbent that can be practically attained through modification of that material's internal physical properties, followed by an additional increase in adsorption rate through reduction in the adsorbent particle size. The necessary particle size is related to the required overall mass transfer rate coefficients and the cycle time/bed depth that lead to the lowest product cost. This strategy reduces the particle size only as much as is necessary to achieve high performance. This leads to the use of the largest particle size that satisfies the rate criteria, thereby resulting in the smallest bed pressure drop for given intrinsic properties of the adsorbent.

By increasing the mass transfer rate according to the invention, one may reduce the size of the mass transfer zone ($L_{MTZ}$) relative to the bed depth (L), and, consequently, increase the working capacity of the adsorbent bed. In contrast to the invention, the prior art discloses attempts to reduce $L_{MTZ}$ by reducing particle size alone, with little regard to the coupled effects of the mass transfer rate, cycle time, bed depth and pressure drop upon process performance. In this sense, the prior art is ambiguous and merely indicates rather broad ranges of particle sizes that are not associated with specific values of cycle time and other process parameters. Furthermore, the prior art has failed to recognize that one may reduce the size of the mass

transfer zone by manipulating the effective pore diffusivity of the adsorbent particle.

In particular, the prior art simply teaches that smaller particles lead to shorter transfer zones which in turn facilitate shorter beds and faster cycles. However, there are several problems that occur with decreasing particle size. First, pressure drop per unit bed length ($\Delta P/L$) increases, with decreasing particle size. This leads to an increase in overall bed pressure drop $\Delta P$, unless the bed depth (L) is decreased. Further, onset of fluidization occurs at decreasing flow velocities as the particle size decreases. Although velocity can be reduced by increasing frontal bed area to lessen both the increase in pressure drop and the onset of fluidization, there are limitations to such area increases and in all cases a reduction in feed velocity generally results in a decrease in bed utilization. Finally, smaller particles are more difficult to immobilize and retain in the adsorber.

The present invention, on the other hand, recognizes that process performance is linked directly to mass transfer rate. In particular, such performance is the result of the coupled effects of mass transfer rate with process conditions such as cycle time, feed temperature and adsorption/desorption pressures. The invention further recognizes that particle size is only one of several adsorbent parameters effecting mass transfer rate and that the particle size required to achieve a desired rate varies depending upon the intrinsic mass transfer rate properties of the adsorbent particle. Since the particle size alone does not establish the rate characteristic of the adsorbent, specification of this

parameter alone for the equilibrium and mass transfer zones (as is suggested by prior art) does not insure maximum process performance.

The present invention considers the coupling of the effects of mass transfer rates (and the associated particle properties), the cycle time and the bed depth to significantly improve gas separation efficiency, (i.e. improvements represented by increases in adsorbent productivity, decreases in process power requirements and/or increases in product recovery). The methodology is especially applicable to the production of oxygen in PSA processes incorporating $N_2$-selective adsorbents, e.g. type X or type A zeolites or advanced adsorbents such as highly Li-exchanged type X or other monovalent cation-exchanged zeolites. While this invention has been demonstrated for the case of air separation, the general methodology applies equally well to other gas phase separations that: (1) depend upon differences in equilibrium adsorption selectivity; and (2) in which the mass transfer resistance is dominated by diffusion in the macropores of the adsorbent particle, i.e. pores of dimension at least an order of magnitude greater than the diameter of molecules diffusing into or out of the particle. For zeolites, this dimension is the order of 30Å to 40Å. For the purpose of this invention, macropores are defined as those pores in the range of approximately 0.0030μm to 20μm which corresponds also to the range of measurement by the standard mercury porosimetry method. Adsorbents may be deployed by this invention in one or more distinct adsorption zones, e.g. pretreatment and main adsorbent zones. One or more adsorbents may be contained in each zone, and the zones do not have to be

contained in the same adsorbent vessel. The pretreatment zone is located nearest the feed inlet and its purpose is to remove any undesirable contaminants from the feed stream. Typical contaminants in air separation include water and carbon dioxide. Those skilled in the art will appreciate that zeolites, activated alumina, silica gel as well as other appropriate adsorbents may be utilized in the pretreatment zone. The main adsorbent zone is positioned downstream of the pretreatment zone (during the adsorption step) and contains adsorbent(s) selective for the primary heavy component(s) in the feed. The pretreatment zone may be excluded if there are no contaminants in the feed stream.

Processes described herein are those in which the separation of at least two components of a gas phase mixture is affected by differences in the equilibrium adsorption capacities of the components in the main adsorbent, i.e. at least one component in the mixture is more selectively adsorbed at equilibrium in comparison to the adsorption of the other components. The present invention is not concerned with kinetic adsorption processes where the primary separation mechanism results from the differences in the rates of diffusion of the components into the adsorbent.

As indicated above, the prior art projects an idealized concept of the mass transfer zone in which $L_{MTZ}$ is independent of the cycle time and bed depth. Due to the presence of gradients in temperature, pressure and adsorbate loading in the adsorbent bed throughout all steps of an actual bulk separation process however, the motion of the adsorption/desorption mass transfer fronts is not ideal.

Since process performance declines as the ratio of $L/L_{MTZ}$ decreases, the goal of performance improvement must be to maintain or increase this ratio. For air separation with highly-exchanged LiX zeolites, ratios of approximately 4.0 are desirable. Wankat (1987), however, teaches that increasing the ratio $L/L_{MTZ}$ beyond a value of 2.0 to 3.0 results in minimal improvement in process performance. For the purpose of the discussions below, the $L_{MTZ}$ is evaluated at the end of the adsorption step.

$L_{MTZ}$ is a consequence of the resistance to mass transfer which determines the adsorption rate. A linear driving force (LDF) model (E. Glueckauf, Trans. Faraday Soc. 51, 1540, 1955) can be used to represent adsorption rate $\left(\frac{\partial \overline{w_i}}{\partial t}\right)$:

$$\rho_b \frac{\partial \overline{w_i}}{\partial t} = k_i\left( c_i - \overline{c_{s_i}} \right) \qquad \text{(A-1)}$$

where $(w_i)$ is the average loading of adsorbate (i), $\rho_b$ is the packed density of the adsorbent in the bed, $c_i$ and $c_{si}$ are average adsorbate gas phase concentrations in the bulk fluid and inside the particle in equilibrium with the adsorbate loading, respectively. The term in brackets is the "concentration driving force." $k_i$ is the mass transfer coefficient which can be represented as a combination of the intrinsic diffusion properties of the adsorbent and adsorbent particle properties as follows:

$$k_i = \frac{15 \varepsilon_p \left( 1 - \varepsilon_b \right) D_{pi}}{r_p^2} \qquad \text{(A-2)}$$

where $D_{pi}$ is the effective pore diffusivity, $\varepsilon_p$ is the void fraction inside the particle, $\varepsilon_b$ is the interparticle bed void fraction (void fraction external to the particles) which for the purposes of this disclosure varies from 0.34 to 0.37, and $r_p$ is the particle radius. The geometry of the macropores is imbedded in the term $D_{pi}$, i.e. a tortuosity factor ($\tau$) is commonly included in the denominator of Equation A-2, but this term has been imbedded in $D_{pi}$ for this disclosure. Equations A-1 and A-2 provide a convenient model to relate the various parameters that effect adsorption rate. While other mechanisms such as micropore diffusion, surface diffusion, film diffusion and bulk phase axial diffusion may influence the mass transfer coefficient, macropore diffusion dominates for many important separations, including air separation using type-X zeolites.

The mass transfer coefficient $k_i$ can be determined by fitting the appropriate model to data obtained from a breakthrough experiment. Since $\varepsilon_p$, $\varepsilon_b$ and $r_p$ can all be determined by measurement, the effective diffusivity $D_{pi}$ is extracted from Equation A-2. This methodology and Equation A-2 clearly distinguish the effects of intrinsic properties from the particle size upon adsorption rate.

Equations A-1 and A-2 represent only one of several ways to characterize adsorption rate. The precise description or definition of adsorption rate is not critical as long as the description is consistently applied and reflects the dominant mechanisms involved for the separations of interest. Such separations include those dominated by equilibrium effects and mass transfer

dominated by macropore diffusion in the particle. The effective diffusivity ($D_{pi}$) is an empirically-determined parameter, and determination must be consistent with the characterization of the adsorption rate.

Since the sorption rate associated with a given adsorbent's internal physical properties has been quantified for the examples herein, the breakthrough experiment employed to obtain the mass transfer rate coefficients and effective diffusivities is briefly described below. One skilled in the art will recognize that variations of this experiment may be used as long as the guiding criteria stated above are followed.

For the process of air separation, a breakthrough test is performed in two steps in which the flow rate, pressure and temperature of the feed gas are the same in both steps. With reference to Figure 1, this process will be described. The first step involves saturation of the adsorbent bed **1** with $O_2$ the least selective component provided via flow meter **2** and line **3**. In the second step, air or a synthetic air mixture containing $N_2$ and $O_2$ is then introduced to bed **1** via flow meter **4** and line **3**. Valve **6** operates in conjunction with flow meter **2** such that pressure of the air or synthetic air is maintained in an external loop until the four port valve **7** connects the air/synthetic air feed to line **3** such that the air/synthetic air flows into bed **1**. The pressure, temperature and composition of the feed mixture in the second step should be representative of that in an adsorption step of an actual process, e.g. 1.5 bar, 300°K and feed air composition. The molar flux was approximately 10 mol/m²s, although this flux may be

varied as required. The pressure is maintained substantially constant in the bed **1** by using a control valve **8** located on the downstream side of the adsorbent bed. The endspace and connecting piping volumes (dead volumes) are designed to be 5% or less than that of the adsorbent bed volume (approximately 20 $cm^3$).

The flow rate and concentration of $O_2$ are continuously and accurately monitored throughout step two via flow meter **9** and oxygen analyzer **10** until the breakthrough of $N_2$ is complete. Flow to analyzer **10** is maintained at a fixed amount via fixed valve **5**. In step two, the more selectively adsorbed $N_2$ displaces the adsorbed $O_2$ already in the bed **1**. As the bed nears saturation with the air mixture, the breakthrough of $N_2$ is reflected in a decrease in the $O_2$ concentration and an increase in overall flow rate of the effluent from the bed. The piping and adsorbent bed are maintained at the same temperature as the feed by immersing them in a thermostat bath controlled at the same temperature as the feed.

A computer model representing the test is then applied to simulate the breakthrough test. A detailed adsorption model, based upon the governing material and energy balances involved in the process, is incorporated. This model uses the same rate expression as that in Equation A-1. The model used here is represented by one dimensional plug flow with negligible axial dispersion. Additional characteristics of the model include: pressure drop (as determined by Darcy's Law or by the Ergun Equation), multicomponent isotherm (as determined by the loading ratio correlation), and adsorption rate (as

determined by the linear driving force). A nonisothermal energy balance was used to account for heat transfer through the wall of the adsorbent bed. Simulations were performed by varying the mass transfer rate coefficients, $k_{N_2}$ and $k_{O_2}$ until the breakthrough effluent flow rate and concentration profiles of the simulation matched those of the experiment.

The prior art has virtually ignored sorption rate effects due to a material's internal physical properties, taking such properties as fixed and invariant. The two exceptions are Moreau et al. (who did not address the significant offsetting effects of high porosity) and Lu et al (who uses forced convection), each of which has been noted in the Background above. With respect to Lu et al, the conditions favoring intraparticle forced convection are not particularly attractive to the present invention in which the intent is to increase pore diffusion within conventional porosity levels while maintaining low bed pressure drop.

The present invention arises in part out of the recognition that the sorption rate, more particularly the effective pore diffusivity, can be manipulated through specific formulation and/or processing of the adsorbent. Examples of such formulations and/or processes include, the variation of binder content and type, the inclusion and subsequent burn-out of fibers having selected dimensions, concentrations and compositions and caustic digestion of the binder. Adsorbents have been produced incorporating such methodologies such that the resulting intrinsic diffusivities are significantly greater than those for conventional, untreated/modified adsorbents. Further, such increases have been achieved while

maintaining the porosity within the desired range of conventional adsorbents. Indeed, the processes and formulations described above, particularly caustic digestion, may even decrease porosity, while increasing the intrinsic diffusivity. Particular parameters for the above are disclosed in co-filed, commonly assigned application D-20,658 (Chao), the contents of which are herein incorporated by reference.

Unexpectedly high gains in process performance were realized when the intrinsic diffusivity was increased. This is evident in Figure 2 where both normalized product recovery and bed size factor (BSF) are shown as functions of effective $N_2$ diffusivity ($D_p$) and mass transfer rate coefficient ($k_{N_2}$). Surprisingly for an equilibrium-dominated separation, there is nearly a 20% improvement in performance when the effective $N_2$ diffusivity (measured using the apparatus of Fig. 1, and at 300K and 1.5 bar) increases from $2.4 \times 10^{-6} m^2/s$ to $1.1 \times 10^{-5} m^2/s$. Further, improvement in process performance diminishes as the rate coefficient is increased beyond some level. As shown in Figure 2, for a 60 second cycle (defined below), improvements in BSF and $O_2$ recovery lessen beyond the mass transfer coefficient $k_{N_2} = 40 s^{-1}$. Therefore it is preferred to maintain the preferred mass transfer coefficient range between $12\ s^{-1} \leq k_{N_2} \leq 40 s^{-1}$. These results were obtained by simulation of a VPSA air separation process for the production of $O_2$ using highly exchanged LiX (2.0) adsorbent having greater than 95% Li. The particle size was held constant along with process conditions while the effective diffusivity was varied for the results shown in Fig. 2.

The desired rate coefficient can be obtained by manipulating the intrinsic properties of the adsorbent and/or the adsorbent particle size. The effect of particle size upon the mass transfer coefficient for various values of the effective $N_2$ diffusivity ($D_p$) is illustrated in Figure 3. Equation A-2 was applied to construct the family of characteristics shown.

It is apparent from the results in Fig. 3 that the larger the intrinsic diffusivity, the larger the particle size that can be used to achieve a desired mass transfer coefficient, i.e. defined by such methods as demonstrated in Figure 2. For example, a desired mass transfer rate coefficient $k_{N_2} = 40s^{-1}$ can be obtained with a 0.85 mm particle size when the effective diffusivity for $N_2$ is $2.4 \times 10^{-6}$ $m^2/s$ or with a 1.95 mm particle size when the intrinsic diffusivity for $N_2$ is $1.2 \times 10^{-5}$ $m^2/s$. We should note that when we refer to particle size ($d_p$), we refer to the average diameter of an adsorbent comprising particles distributed over a range of sizes, as those skilled in the art will recognize.

To demonstrate these concepts further and to bracket process parameters, VPSA air separation process performance was determined for total cycle times of 60s and 15s. The following conditions were maintained: $O_2$ product purity at 90%, adsorption pressure at 1.5 bar, desorption pressure at 0.3 bar and feed temperature at 320°K. A simple eight-step cycle including pressurization, feed, purge, equalization and evacuation was employed. A highly exchanged (>95% Li) LiX adsorbent ($SiO_2/Al_2O_3$ ratio of 2.0) was used as the main adsorbent with bed depths of 1.37m and 0.343m for the 60s and 15s

cycles, respectively. The mass transfer coefficients (determined by methods described previously) for $O_2$ were approximately 35% of those for $N_2$ for LiX adsorbents. The average feed air molar flux was 17 mol/m²s.

The cycle is described by the diagram in Figure 4, while the step times are given in Table I for the 60s cycle. The step times were all shortened by the ratio 15/60 for the 15s cycle.

Table I. 8-Step VPSA Cycle

| Step No. | Description | Duration(s) |
|---|---|---|
| 1.00 | Feed/Make Product | 6.00 |
| 2.00 | Feed/Make Product/Purge | 7.00 |
| 3.00 | Equalization Down | 4.00 |
| 4.00 | Blowdown/Evac. | 13.00 |
| 5.00 | Evacuation | 6.00 |
| 6.00 | Purge/Evac. | 7.00 |
| 7.00 | Equalization Up | 4.00 |
| 8.00 | Repressurization | 13.00 |

A detailed computer model of the process was used to determine the performance at various levels of adsorption rate. The adsorbent bed model equations are similar to those described above for the model of the rate test. The energy balance for the adsorbent is adiabatic, however, in the process model. The different bed pressure drops in each step of the cycle were maintained nearly constant for all process examples.

The simulation results for $O_2$ recovery and BSF are shown in Figure 5a and Figure 5b. As illustrated therein, product recovery drops considerably for $N_2$ mass transfer coefficients $k_{N_2} < 20s^{-1}$ for the 60s cycle, while a

similar decline in performance is apparent for $k_{N_2} < 40s^{-1}$ in the 15s cycle. The BSF results reflect similar conclusions. Likewise, performance gains diminish significantly for $k_{N_2} > 40s^{-1}$ and $k_{N_2} > 80s^{-1}$ for the 60s and 15s cycles, respectively. Increasing the adsorption rate alone nearly doubles the product recovery and halves the BSF, while the shorter cycle alone results in a BSF reduction of more than a factor of three with only a minor penalty in $O_2$ recovery. When combined, the effects of higher adsorption rate and short cycle time lead to a reduction in BSF of more than a factor of six. The values of the $N_2$ rate coefficient ($k_{N2}$) leading to high performance in Figure 5 reflect the process conditions stated: $O_2$ product purity at 90%, adsorption pressure at 1.5 bar, desorption pressure at 0.3 bar and feed temperature at 320°K for a feed composition of air. One skilled in the art will recognize that the same methodology described above can be applied to determine the preferred rate constants for other process conditions.

In general (for cycle times less than about one minute) a mass transfer coefficient $k_{N2} \geq 12\ s^{-1}$ is preferred, with a rate of $k_{N2} \geq 20s^{-1}$ being more preferred. As preferred values of $k_{N2}$ are, in part, dependent upon cycle time (as shown in Fig. 5), for shorter cycle times, greater values of $k_{N2}$ are preferred. Thus for a cycle time of 15 sec, a rate of $k_{N2} \geq 40s^{-1}$ is also preferred, and a value of $k_{N2}$ up to $80s^{-1}$ is acceptable.

Figure 5, which is consistent with the results of Figure 2 and the preferred ranges set forth above, shows the best ranges of adsorption rate for cycle duration

from 15s to 60s. As indicated in Equation A-2, such rates may be achieved by increasing the effective diffusivity ($D_p$) or the intraparticle void fraction ($\varepsilon_p$) and/or by decreasing the particle size. Although each approach has theoretical limits, decreasing particle size or increasing porosity are accompanied by penalties to overall separation performance. Decreased particle size results in increased pressure drop per unit bed length, increased potential for fluidization and greater difficulty in particle retention in the bed as described above.

Intraparticle void fraction **($\varepsilon_p$)** is defined by Equation A-3:

$$\varepsilon_p = \rho_p \nu_i \qquad \text{A-3}$$

where $\rho_p$ is the particle density, and $\nu_i$ is the internal macropore volume per unit mass of particle. $\nu_i$ may be determined by the well-known mercury porosimetry method.

Increasing the porosity or intraparticle void fraction reduces the overall active adsorbent content of the particle resulting in lower particle density. This in turn increases the volume of adsorbent required for a given $N_2$ adsorbate capacity (mol/g). There is a natural tendency for the particle density to decrease as pore volume is increased for a fixed adsorbent composition. Conversely, the pore volume usually decreases when $\rho_p$ increases. This apparent inverse relationship between particle density and macropore volume, while not in constant proportion, tends to restrict the practical

range over which the void fraction $\varepsilon_p$ can be varied for each particular type of adsorbent. Indeed, the intraparticle void fraction ($\varepsilon_p$) of common synthetic zeolites is typically in the rather narrow range of 0.30 to 0.38 (Wankat, P.C., Rate-Controlled Separations, Elsevier Applied Science, 1990, pg. 226).

This range of porosities for zeolites is also related to a physical strength requirement, i.e. adsorbent particles in the bottom of large commercial beds must resist crushing under the weight of thousands of pounds of adsorbent contained in the adsorber vessel. High porosity/low density particles are subject to lower crush strength. Larger internal void fraction ($\varepsilon_p$) also increases the non-selective gas storage volume in the adsorbent bed and thereby decreases the separation capability, i.e. reduces overall product recovery. While at first glance increasing $\varepsilon_p$ appears to be a good way to increase adsorption rate (as indicated by Equation A-2), the offsetting effects in process performance and the potential mechanical difficulties arising from adsorbent particle breakdown make increasing porosity both a limited and an undesirable choice for rate enhancement.

The preferred method for increasing adsorption rate is to increase the effective diffusivities ($D_p$) in the macropore space of the particle. Increased $D_p$ alone results in higher mass transfer coefficients with virtually no offsetting effects in performance nor problems in decreased particle strength. The maximum effective diffusivity is limited, however, to the Maxwell (free space) diffusivity, e.g. for $N_2/O_2$ at 293°K and 1.0

atm, this limit is 2.2 x $10^{-5}$ $m^2/s$ (Hirschfelder, J.O. et al., Molecular Theory of Gases and Liquids, John Wiley & Sons, 1964, pg. 579). This limit is significantly above the effective diffusivities for $N_2/O_2$ in conventional zeolites.

By way of illustration, several examples are provided in which the effective macropore diffusivity has been increased through formulation and/or processing of the aggregated product. The details of the formulation/processing are described in co-pending application D-20,658 (Chao et al), the contents of which are herein incorporated by reference. These results demonstrate that effective diffusivities can be significantly increased in comparison to conventional adsorbents. Such improvement to the intrinsic adsorbent properties can then be applied to great advantage in separation processes as described herein. These examples are in no way limiting, but illustrative only, as one skilled in the art will appreciate that alternative methods for achieving increased effective diffusivity will lead to corresponding improvements in process performance.

Chao (D-20658) has demonstrated various formulations and methods for producing adsorbents with intrinsic diffusivity higher than that of conventional adsorbents. The pore diffusivity of adsorbents can be enhanced by first combining a low amount of binder with zeolite in the bead-forming step followed by caustic digestion (c.d.). The intrinsic rate characteristics of the adsorbent can be improved further by the addition of fiber with subsequent burnout. Not wanting to be restricted to any one method or formulation, the detailed procedure for producing adsorbent S-1 of the invention is

herein described as one example of making such high rate adsorbents. The method of making S-1 involves the four primary steps of bead forming, caustic digestion, ion exchange and calcination as described below.

Bead forming:

2640 gm dry weight of NaKX2.0 (wet weight 4190. gm) zeolite, 360 gm dry weight of the ECCA Tex-611 (wet weight 426 gm) kaolin clay were mulled for 15min. while water was pumped in at a rate of 10 ml/min. The rate of water addition was then decreased to 4 ml/min for 40 min and the mixture was mulled another 20 min. The mulled mixture was then transferred to a DBY-10R Nauta Mixer ( supplied by Hosokawa Micron Powder Systems) and mixed for about one hour. The lumps were broken down to return the mixture to a powder state. Water then was added slowly by an atomizer. As the moisture of the mixture increases, beads start to form. The growing of the beads was stopped by adding dried bonding mix at a time for harvesting the highest yield of 8x12 size beads.

The beads were dried in air overnight and then calcined in a Blue M oven with a dry air purge. The oven temperature was ramped up to 600 C in 2 hours and then held at 600 C for 2 hours during the dry air purge.

Caustic Digestion

1861.8 gm dry weight of calcined NaKX2.0 beads of size 6x16 with 12% binder were used for caustic digestion. To prepare digestion solution, 360 gm of NaOH (9 mole) and 251.1 gm (4.475 mole) KOH was dissolved in 7386 gm of water. To this solution, 320 ml of sacrificial NaKX2.0 beads were added and stirred at 90C for 2 hours. The solution was left to settle and 6397.7 gm supernatant was collected. To this supernatant,

1477.2 ml of water, 72.0 gm of NaOH and 50.2 gm of KOH were added to make up for the discarded caustic. The resulting solution was used as digestion solution.

The beads were loaded into two stainless steel columns of 3 inch diameter and the solution from a common reservoir was recycled through each column at a flow rate of 30 ml/min. and temperature of 88C for 26 hours. After digestion the beads were washed by pumping 40 liter of NaOH solution (pH =12, 88C) through each column. The beads in each column were further washed with 30 liter of NaOH solution (pH =8.5, 88C). The product, NaKX2.0CD, was air-dried and screened to various particle size fractions.

Ion Exchange

694.5 gm dry weight of NaKX2.0CD 8x12 beads were loaded into a 3 inch i.d. glass column. A 10 inch layer of 3mm Pyrex glass beads was placed at the bottom of the column to serve as a preheating zone for the solution. The column was wrapped with a heating tape. The ion exchange solution was first passed through a 15 liter 90 C preheating flask to partially remove any dissolved air to prevent air bubbles from forming that could be subsequently trapped in the column. The hot solution was then pumped into the bottom of the column.

The ion exchange solution was prepared by dissolving 2162 gm LiCl in 80 liter distilled water (0.64M) then LiOH solution was added to adjust pH of solution to 9. The solution was pumped through the column at the speed of 15 ml/min. until ten to twelve times the stoichiometric amount of LiCl, for full Li-exchange of the beads, had been circulated through the column. After the ion exchange was completed, the product was washed with 30 liter of 90 C distilled water at a flow rate of

60 ml/min. The pH of this water was adjusted to 9 by adding LiOH.

Drying and Calcination

The washed product was first air-dried and then dried further in a low temperature oven with ample air purge for 3 hours to bring the moisture content of the beads to about 12-15%. The dried beads were calcined in a Blue M oven with ample dry air purge. The oven temperature was ramped from room temperature to 600 C in two hours and maintained at 600 C for 40 minutes. The sample was removed from the oven at 450 C and placed into a tightly sealed glass jar for cooling.

The $N_2$ mass transfer coefficients and effective diffusivities were determined from breakthrough tests as described above for commercial zeolites available in bead form as 13XHP, 5AMG, LiX(2.5) and LiX(2.3) from UOP of Des Plaines, IL USA. These results are summarized in Table II for reference adsorption conditions of 1.5 bar and 300°K. The effective pore diffusivities are well below the Maxwell diffusivity (approximately $1.5 \times 10^{-5}$ $m^2/s$) at these conditions. The relatively narrow range of effective diffusivities for these commercial materials is reflective of the conventional processing methods for zeolites. The greater range of mass transfer coefficients for these same adsorbents occurs almost entirely from the differences in particle size.

Several different treatments were applied to LiX (2.0) zeolite in order to enhance the effective diffusivities for $N_2$ and $O_2$. As described above, various

clay binder types and contents, and conversion of the binder to zeolite through caustic digestion (c.d.) and the use of a fiber additive with subsequent burnout were all explored. The effects of these treatments are illustrated in Table II for LiX (2.0) adsorbents (S1-S4).

Table II. Summary of Adsorbent Properties

| Sample | Zeolite ($SiO_2/Al_2O_3$) | Binder Content | Other | $\varepsilon_p$ | $D_pN2$ $m^2/s$ | $d_p$ mm | $k_{N2}$ $s^{-1}$ |
|---|---|---|---|---|---|---|---|
| 13X | NaX (2.5) | | | 0.31 | $3.2 \times 10^{-6}$ | 2.1 | 8 |
| 5A MG | NaCaA | | | 0.32 | $3.1 \times 10^{-6}$ | 0.7 | 80 |
| Oxysiv-7 | LiX (2.5) | | | 0.36 | $2.6 \times 10^{-6}$ | 0.55 | 100 |
| S-0 | LiX (2.3) | | | 0.33 | $2.9 \times 10^{-6}$ | 1.9 | 10 |
| S-1 | LiX (2.0) | 12% | c.d. | 0.35 | $5.5 \times 10^{-6}$ | 2.0 | 18 |
| S-2 | LiX (2.0) | 20% | c.d. | 0.27 | $2.0 \times 10^{-6}$ | 2.0 | 5 |
| S-3 | LiX (2.0) | 20% | w/fiber c.d. | 0.32 | $3.4 \times 10^{-6}$ | 2.0 | 10 |
| S-4 | LiX (2.0) | 12% | | 0.35 | $7.9 \times 10^{-7}$ | 2.0 | 2.6 |

Sample S2 represents a zeolite formulation using 20% clay binder subsequently treated by caustic digestion (c.d.) to convert binder to zeolite. The resulting porosity is about 10% lower and the effective diffusivity is about 35% lower for S2 than these same properties for 13X and 5A MG. When the binder content is lowered to 12%, there is a substantial difference in the effective

diffusivity as a result of the caustic digestion and conversion of binder to zeolite. The c.d. sample S1 has an effective diffusivity more than seven times greater than that for sample S4 (no c.d.) with no significant difference in the porosities. Furthermore, the effective diffusivity of sample S1 is nearly 1.9 times greater than the LiX 2.3 adsorbent (S0) - also with only a small change in porosity. In terms of rate coefficient, $k_{N2}$ of S1 is 1.8 to 7.0 times larger than that of any of the other materials of equal particle diameter in Table II. Samples S1-S4 all use the same type of clay binder (kaolin). In samples S1-S3, this binder is converted to zeolite by caustic digestion. Although samples S1-S3 have essentially the same final chemical composition (LiX (2.0)), the intrinsic diffusivities for these samples vary widely due to the different pore structures created as a result of the different formulation and processing steps.

It is evident from these results that substantial increases in effective macropore diffusivity can be obtained through special formulation and processing of the adsorbent. Additionally, these improvements in effective diffusivity are obtained while maintaining the adsorbent particle porosity in the same range as conventional adsorbents. Such increases in this intrinsic rate property of the adsorbent can then be coupled with the proper choice of particle size and process operating conditions to achieve significant process performance advantages - subsequently captured as a reduction in the overall cost of the product.

Once the sorption rate associated with a material's internal physical properties is obtained within the

manufacturing and cost constraints of a given methodology, the results of Figure 5 are combined with the characteristics of Figure 3 to select the particle sizes necessary to achieve the desired mass transfer rate coefficients; i.e. rate coefficients that lead to high process performance and minimum product cost. A value of $D_p$ for $N_2$ of 5.5 x 10-6 $m^2/s$ for a LiX(2.0) (>95% exchanged) adsorbent is selected to illustrate the concept.

Along this $D_p$ characteristic in Figure 3, particle diameters of 1.85mm, 1.3mm and 0.92mm correspond to values of $k_N$, of $20s^{-1}$, $40s^{-1}$ and $80s^{-1}$, respectively. Using a bed of 1.37m depth containing 1.85mm particles as a reference condition for pressure drop, the bed depths for the smaller particle size configurations are now established from the Ergun equation to keep the overall bed pressure drop the same in all three cases. Note that a lower pressure drop could have been chosen as a reference condition. The cycle time was then adjusted to maintain a minimum product purity of 90% $O_2$. In the first three cases the endspace volumes (void space above and below the adsorbent bed inside the vessel) were maintained constant. The results of process simulations are shown in Table III and Figure 6.

Table III Summary of Simulation Results:
Bed Depth/Cycle Time/Particle Size Study

| | (P_3) | (R_2) | (R_3) | (R-3rv) |
|---|---|---|---|---|
| $O_2$ Purity (%) | 90. | 90. | 91. | 90. |
| $O_2$ Recovery | 0.55 | 0.52 | 0.45 | 0.54 |
| Throughput TPDO | 58.00 | 55.00 | 48.00 | 57.00 |
| BSF lb/TPDO | 737.00 | 387.00 | 224.00 | 187.00 |

| | | | | |
|---|---|---|---|---|
| $k_{N2}$ $s^{-1}$ | 20.00 | 40.00 | 80.00 | 80.00 |
| Particle diameter $d_p$, mm | 1.85 | 1.30 | 0.92 | 0.92 |
| bed depth, m | 1.37 | 0.69 | 0.34 | 0.34 |
| Lower endspace void fraction | 0.14 | 0.30 | 0.59 | 0.14 |
| Upper endspace void fraction | 0.18 | 0.38 | 0.76 | 0.18 |
| Cycle time, sec. | 60 | 30 | 15 | 15 |

The first three columns in Table III show the significant reduction (up to 70%) in BSF that is achieved with the shorter cycles enabled by higher mass transfer rates. Unfortunately, the product recovery and throughput decline substantially for the R_3 case with 0.92mm particle diameter and a 0.343m bed depth. This is very undesirable due to the negative impact of reduced recovery upon power consumption.

A major contributor to this problem is the increasing fraction of endspace void volumes relative to bed volume as the bed depth (and bed volume) decrease as shown in Table III, i.e. the upper and lower endspace void fractions increase from 0.18 and 0.14 to 0.76 and 0.59, respectively. A fourth case (R_3rv) was simulated with a reduction in endspace volumes to restore the fractional endspace voids to the same as that in the 1.37m bed depth reference case (P_3). The product recovery and throughput are nearly fully restored. Thus, it is preferred to maintain the void fraction of each of the endspaces at 30% or less than the total adsorbent bed volume. As the effective diffusivity $D_p$ increases, the performance characteristics in Figure 6 shift to the

right, i.e. similar short-cycle performance gains are realized at even larger particle sizes.

The information in Figures 3, 5 and 6 are now combined with the concepts of the invention and the example results to define the preferred particle size and intrinsic diffusivity, i.e. combinations that will result in the highest bed utilization and overall best process performance for various cycle times for VPSA air separation using LiX (2.0). The results are shown in Figure 7 for the range of effective $N_2$ diffusivities $2.4 \times 10^{-6} \leq D_p \leq 1.2 \times 10^{-5}$ m²/s.

From Figure 7, it is evident that the larger the intrinsic diffusivity, and thereby the larger the sorption rate derived from this internal physical property of the adsorbent, the larger the particle size that can be accommodated to achieve a desired performance, e.g. for a cycle time of 30s, a particle size within the range of about 0.85mm to about 2.0mm, preferably between about 1.1 mm and about 1.6 mm will be required depending upon the intrinsic rate properties of the adsorbent. Thus, when the intrinsic diffusivity $D_p = 5.5 \times 10^{-6}$ m²/s, a particle diameter of 1.3mm is recommended for a cycle time of 30s while a particle diameter of 1.75mm is best for 55s cycle.

Considering the results of Figs. 5-7, it is preferred that the rate coefficient ($k_{N2}$) be coupled with a $D_p$ of greater than or equal to $3.5 \times 10^{-6}$ m²/s, preferably $4.0 \times 10^{-6}$ m²/s, more preferably $4.5 \times 10^{-6}$ m²/s.

Although Figure 6 indicates that the highest bed utilizations (lowest BSF) correspond to the shortest cycles, there may be compelling design and cost reasons

to operate above the shortest cycle times, e.g. the lowest product cost may not correspond to the shortest cycle if endspace void volume cannot be controlled in the desired range, valve cycle times may limit the shortest cycle times, etc. For these reasons, Figure 7 provides a guide for these parameters over a significant cycle time range. In general, the bed depth will scale directly with the cycle time as illustrated in Table III.

To accommodate a range of desirable particle sizes: for a cycle time of less than or equal to 80s, the bed depth is preferably less than or equal to about 2.0m; for a cycle time of less than or equal to about 60s, the bed depth is preferably less then or equal to about 1.5m; similarly for a cycle time of less than or equal to 40 seconds, the bed depth is preferably less or equal to 1.2m; and for a cycle time of less than or equal to about 20s, the bed depth is preferably less than or equal to about 0.63m.

As indicated above an object of the present invention is to make significant improvements in adsorbent utilization and product recovery through enhancement of the rate characteristics of the adsorbent - primarily by increasing the effective diffusivity in the macropores of the adsorbent particle. The improved recovery achieved under the conditions of the invention also leads to reduced power consumption per unit of product produced. The invention is preferably directed at equilibrium-based adsorption separation processes with mass transport dominated by intraparticle pore diffusion. While the examples have been directed at air separation using a single main adsorbent, the invention is not

limited to binary mixtures, nor to air as a feed nor to a single main adsorbent.

Further, when more than a single separation is to be achieved, it is contemplated to include one or more adsorbents as main adsorbents. In such a case, each adsorbent would be responsible for a different separation or a different level of the same separation. Multiple mass transfer zones may then be present in the process. An analysis similar to that described above would be performed for each of the adsorbent/adsorbate combinations where overcoming significant mass transfer resistance limitations would lead to overall improvements in process performance. Thus, the properties (particularly those related to the rate of adsorption) of the different adsorbent materials in the main adsorbent zone are selected to maximize all of the separations required of the process. Examples of such processes include the recovery of $H_2$ from $H_2/CO/CO_2/CH_4$ mixtures; prepurification, including the removal of $H_2O$ and $CO_2$ from air, separation of Ar from air or $N_2$ or $O_2$, drying of process streams and the recovery of $CO_2$ from flue gases or from $H_2$ PSA tail gas.

Type X zeolite adsorbents are suggested for air separation, most preferably highly-exchanged LiX as described by Chao (U.S. Pat. No. 4,859,217). Other type X materials with monovalent cations or mixed cations are also applicable to the present invention such as those suggested by Chao (U.S. Pat. No. 5,174,979). The invention is also applicable to any type of equilibrium-selective adsorbent material including, but not limited to, A-zeolite, Y-zeolite, chabazite, mordenite, clinoptilolite and various ion exchanged forms of these,

as well as silica-alumina, alumina, silica, titanium silicates and mixtures thereof.

It should also be clear that the present invention may be practiced with various deployments of adsorbents in the main adsorbent zone, e.g. layers and mixtures of adsorbents of various types or of the same type but with varying adsorption and/or physical characteristics. For example, the enhanced rate concepts of this invention could be applied to the layered beds suggested by Ackley in co-pending application SN 08/837,411, as well as Notaro et al (USP 5,674,311) and Watson et al (USP 5,529,610).

Finally, a further improvement over the basic invention can be obtained by distributing the adsorbents with different rate properties to minimize pressure drop and/or mass transfer zone size. The selection of properties should be made in order to increase the rate of adsorption and minimize the fractional size(s) of the mass transfer zone(s) at the end of the adsorption step.

The present invention teaches a method to improve process performance by reducing mass transfer limitations without incurring any increase in process pressure drop. Bed depth and cycle time are reduced to compensate for increased specific pressure drop (pressure drop per unit depth of adsorbent) when particle size is reduced. There may be cases, however, where either a further reduction in pressure drop is desired and/or where the use of adsorbents with different rate properties is desirable or necessary. In such an embodiment, a poorer sorption rate-quality adsorbent (low mass transfer coefficient) could be used in the equilibrium zone and a higher

sorption rate-quality version of the same adsorbent (high mass transfer coefficient) in the mass transfer zone.

It is further contemplated that the poorer rate-quality material in this latter condition could also be of smaller diameter. This would result in a configuration with regard to particle sizes in the adsorbent bed that is completely opposite to the prior art teachings. Thus when multiple adsorbents with different rate characteristics must be used, maintaining the adsorbent with the largest mass transfer rate coefficient in the mass transfer zone insures the best overall process performance.

Since the mass transfer zone forms initially and develops in what eventually becomes the equilibrium zone (at the end of the adsorption step), the rate of adsorption cannot be too low relative to that in a succeeding layer of adsorbent. This is because the leading edge of the mass transfer zone would erupt from the adsorber before the trailing edge crosses the boundary between the two materials. This would result in a reduced size of the equilibrium zone and increased size of the mass transfer zone and consequently, overall lower product recovery and/or purity.

This condition may be minimized by selecting the adsorbents and the mass transfer coefficients (MTC) of the most selective component such that the size of the mass transfer zone in the adsorbent of the lowest MTC is no more than twice that of the size of the mass transfer zone in the adsorbent of the highest MTC.

The problem may also be solved by distributing the adsorbents in such a way as to achieve a gradual increase in mass transfer coefficients (in contrast to discrete

layers) from the inlet to the outlet of the adsorber. When multiple adsorption zones are contained in the main adsorbent for the purpose of multiple separations, it is appreciated that the concept of mass transfer coefficient gradients (either by discrete layers or by gradual change) can be applied individually to each included separation zone.

The concepts of this invention are not limited to any specific set of process conditions but may be applied over a wide range of process conditions, e.g. temperatures, pressures, feed velocities, etc. It is only necessary to evaluate the rate characteristics of the adsorbent at the process conditions of interest before applying these concepts in order to insure maximum process performance. Likewise, these concepts can be applied to single-bed as well as multi-bed processes operating with subatmospheric (VSA), transatmospheric (VPSA) or superatmospheric (PSA) cycles.

While the examples disclosed in this application use an eight-step cycle, the benefits of the invention may also apply to simpler cycles comprising fewer steps and more complex cycles comprising additional steps.

The enhanced-rate concepts described here are not limited to any particular adsorber configuration and can be effectively applied to axial flow, radial flow, lateral flow, etc. adsorbers. The adsorbent may be constrained or unconstrained within the absorber vessel.

The benefits of the invention may also be obtained in cycles in which the primary product is the more selectively adsorbed component (e.g. $N_2$) or in cycles wherein both the more and less strongly held component are recovered as product.

Specific features of the invention are shown in one or more of the drawings for convenience only, as such feature may be combined with other features in accordance with the invention. Alternative embodiments will be recognized by those skilled in the art and are intended to be included within the scope of the claims.

What is claimed is:

1. A process for the separation of nitrogen from a gas mixture including nitrogen and a less selectively adsorbable component, comprising: contacting said gas mixture in an adsorption zone with an adsorbent that is equilibrium selective for nitrogen over said less selectively adsorbable component and adsorbing said nitrogen on said adsorbent, wherein said adsorbent zone comprises an adsorbent selected from the group consisting of A-zeolite, Y-zeolite, NaX, mixed cation X-zeolite, LiX, chabazite, mordenite, clinoptilolite, silica-alumina, alumina, silica, titanium silicates and mixtures thereof, wherein said adsorbent has a mass transfer coefficient (MTC) for nitrogen of $k_{N2} \geq 12\ s^{-1}$ and an intrinsic diffusivity for $N_2$, when measured at 1.5 bar and 300K, of $D_p \geq 3.5 \times 10^{-6}\ m^2/s$.

2. The process of claim 1 where the mass transfer coefficient for the more selectively adsorbed component is $k_{N2} \geq 20s^{-1}$.

3. The process of claim 1 where the mass transfer coefficient for the more selectively adsorbed component $k_{N2} \geq 40s^{-1}$.

4. The process of claim 1 where the intrinsic diffusivity for $N_2$, when measured at 1.5 bar and 300K, is $D_p \geq 4.0 \times 10^{-6}\ m^2/s$.

5. The process of claim 4 where the mass transfer coefficient for the more selectively adsorbed component is $k_{N2} \geq 20s^{-1}$.

6. The process of claim 4 where the mass transfer coefficient for the more selectively adsorbed component $k_{N2} \geq 40s^{-1}$.

7. The process of claim 1 where the intrinsic diffusivity for $N_2$, when measured at 1.5 bar and 300K, is $D_p > 4.5 \times 10^{-6}$ $m^2/s$.

8. The process of claim 7 where the mass transfer coefficient for the more selectively adsorbed component is $k_{N2} \geq 20s^{-1}$.

9. The process of claim 7 where the mass transfer coefficient for the more selectively adsorbed component $k_{N2} \geq 40s^{-1}$.

10. The process of claim 1 where the less selectively adsorbable component is oxygen.

11. The process of claim 1, wherein said gas mixture is air.

12. The process of claim 1 wherein said process further includes the steps of desorbing said more selectively adsorbable component from said adsorbent such that an adsorption/desorption cycle occurs, and wherein a cycle time to complete said adsorption/desorption cycle is less

than 80s and wherein said adsorbent bed has a bed depth of less than 2.0m.

13. The process in claim 12 where said cycle time is less than 60s and said bed depth is less than 1.5m

14. The process in claim 12 where said cycle time is less than 40s and said bed depth is less than 1.2m.

15. The process in claim 12 where said cycle time is less than 20s and said bed depth is less than 0.63m.

16. The process in claim 12 where the cycle time is less than or equal to 30s and wherein said adsorbent is particulate and has an average particle diameter between 1.0mm and about 2.0mm

17. The process in claim 1 where said adsorbent bed is contained in a vessel, and said vessel has endspace void volumes above and below the adsorbent bed that are each no greater than 30% of a total adsorbent volume.

18. The process in claim 1 where said adsorption zone comprises at least an equilibrium zone and a mass transfer zone, and each zone has a different adsorbent material.

19. The process of claim 18, wherein an adsorbent material selected for the mass transfer zone is selected such that its adsorption rate for the more selectively adsorbed component is greater than the adsorption rate of an adsorbent material used in said equilibrium zone.

20. An adsorbent bed comprising at least an equilibrium zone and a mass transfer zone, wherein each zone has a different adsorbent material, and wherein an adsorbent material selected for the mass transfer zone is selected such that its adsorption rate for a more selectively adsorbed component is greater than the adsorption rate of an adsorbent material used in said the equilibrium zone.

21. The adsorbent bed of claim 20, wherein an adsorbent material selected for the mass transfer zone is selected such that its mass transfer coefficient (MTC) of said more selective component is such that a size of the mass transfer zone in the adsorbent of lowest MTC is no more than twice that of a size of the mass transfer zone in the adsorbent of highest MTC.

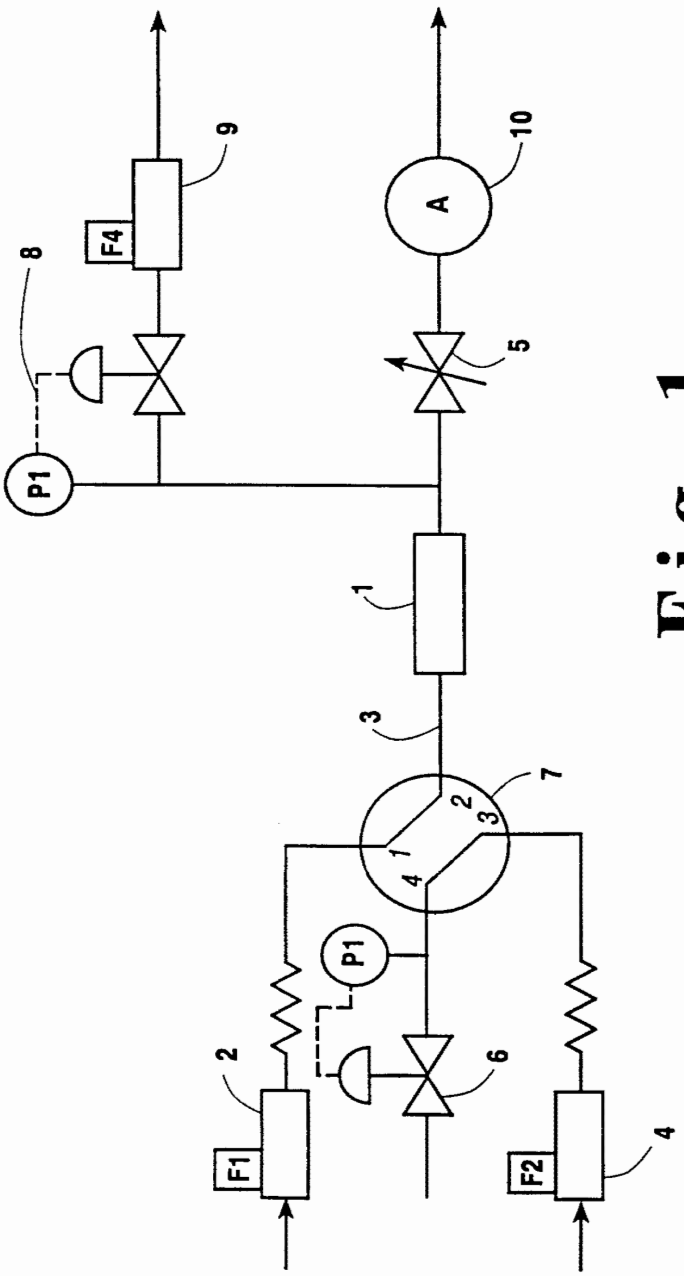



Fig. 1



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GCE Gas Control Equipment Inc. v. VBOX, Inc.
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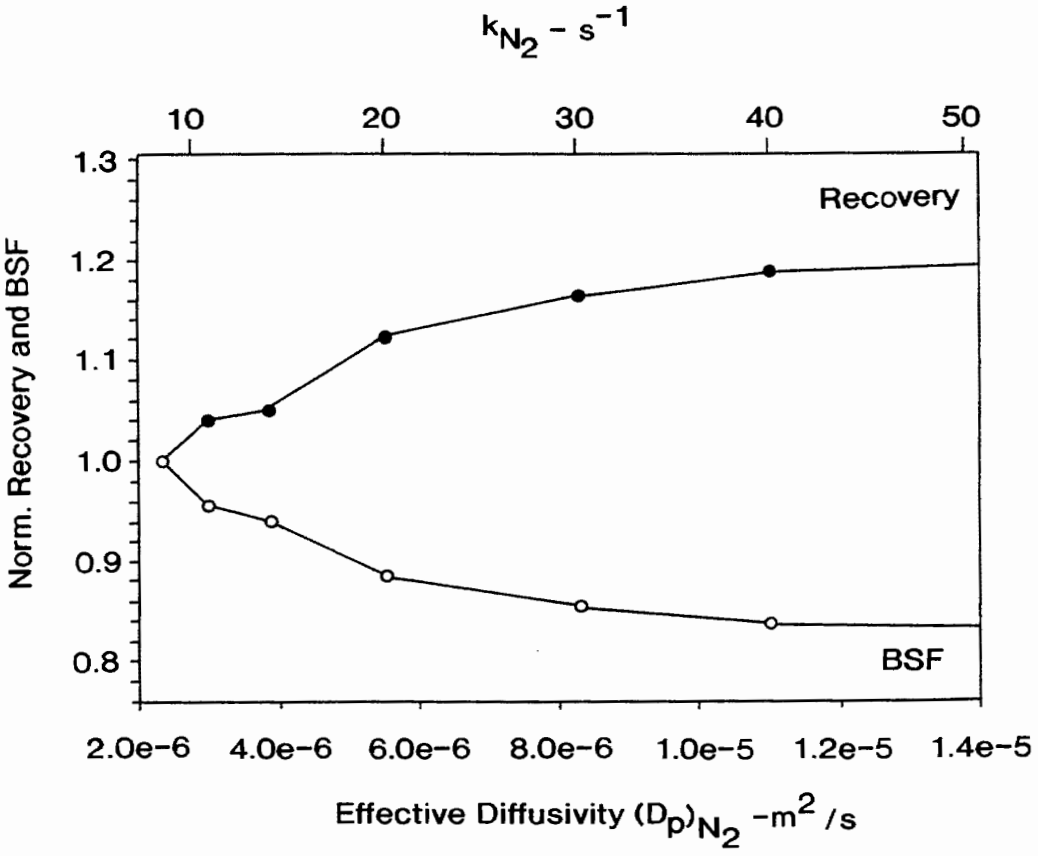



**Fig. 2**



GCE - Exhibit 1071, Page 364
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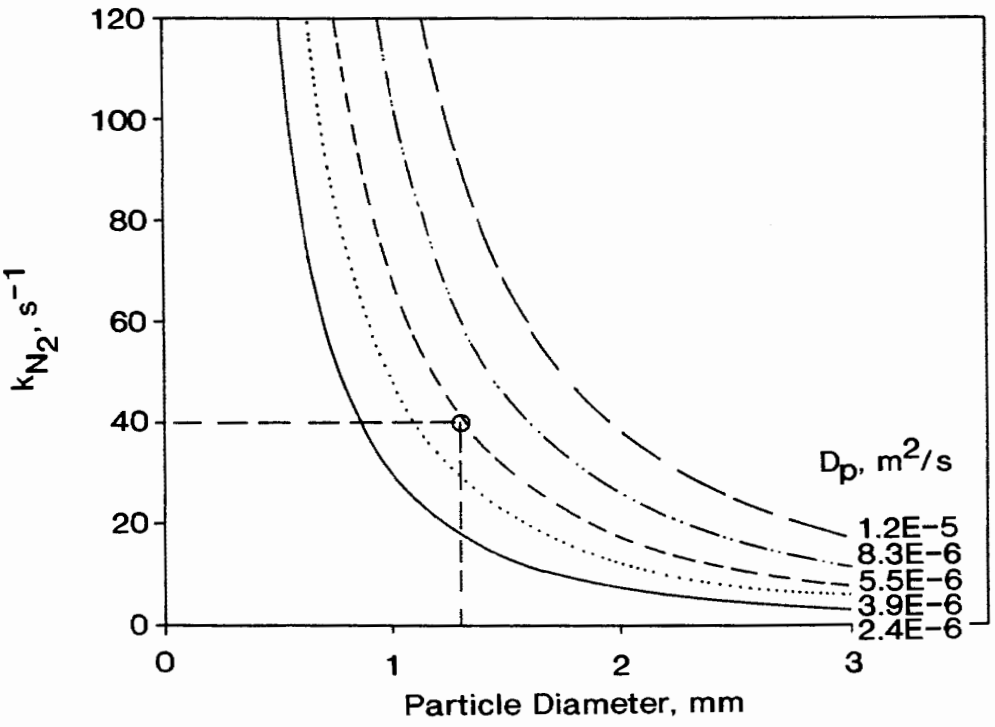



Fig. 3



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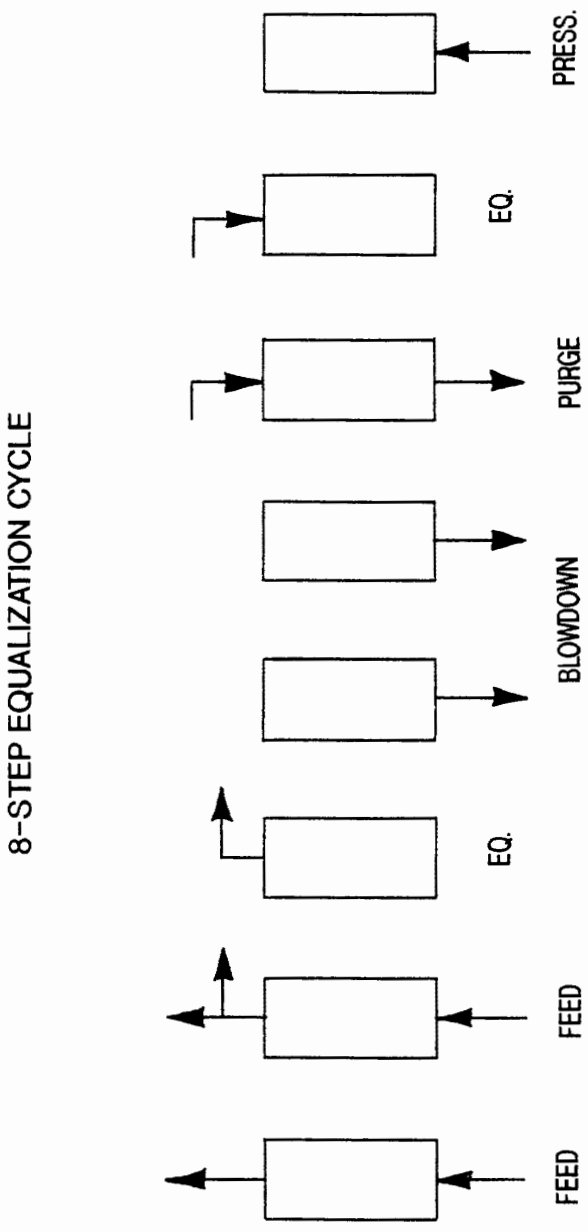



Fig. 4



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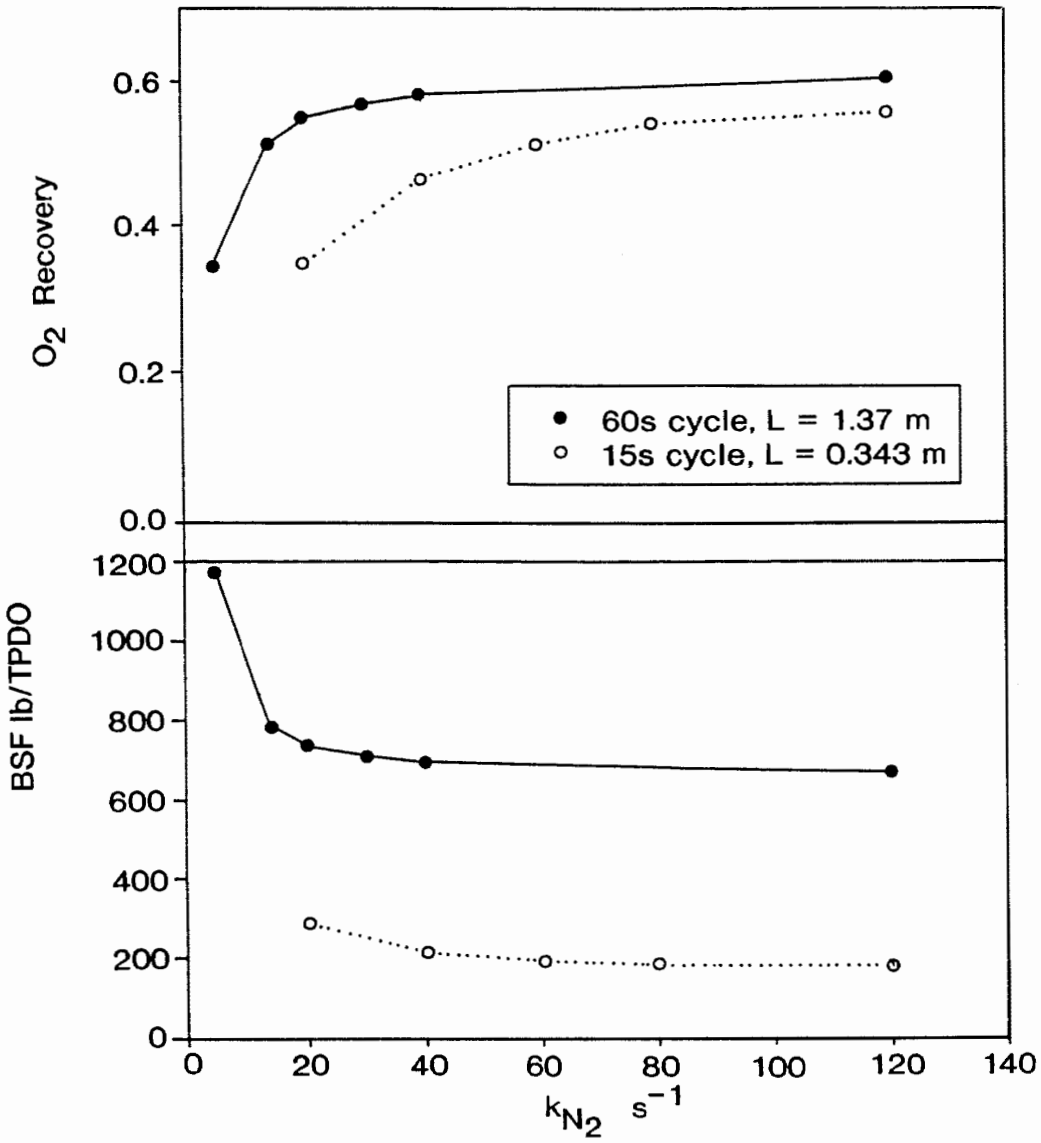



Fig. 5



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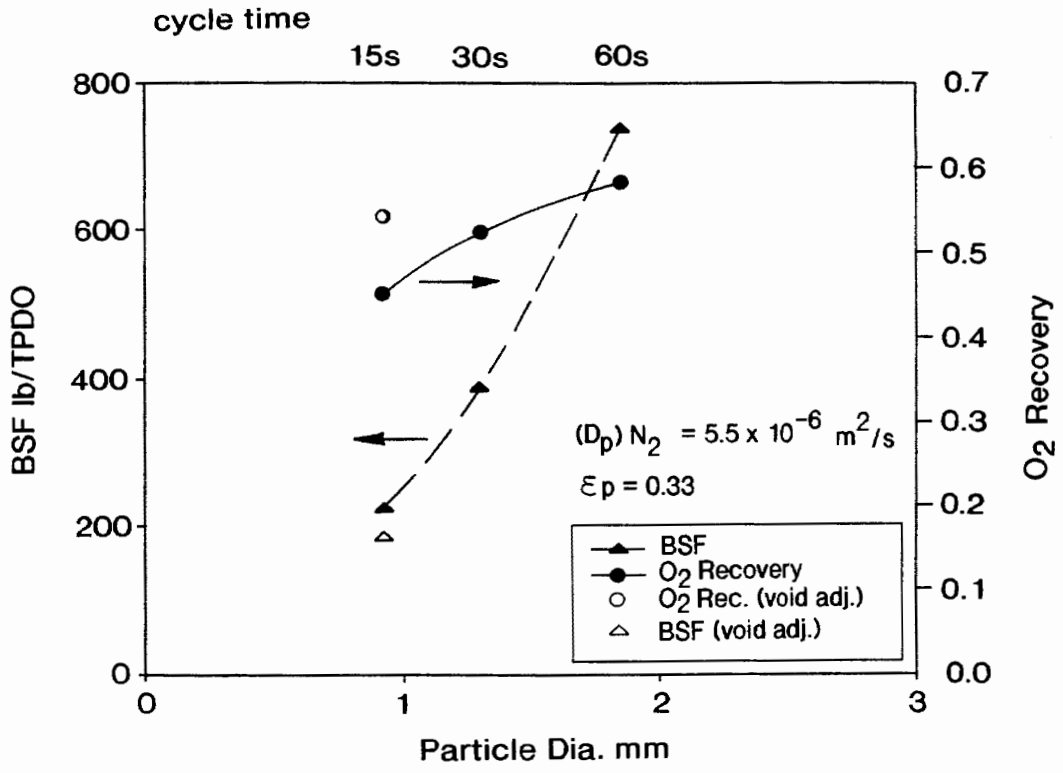



**Fig. 6**



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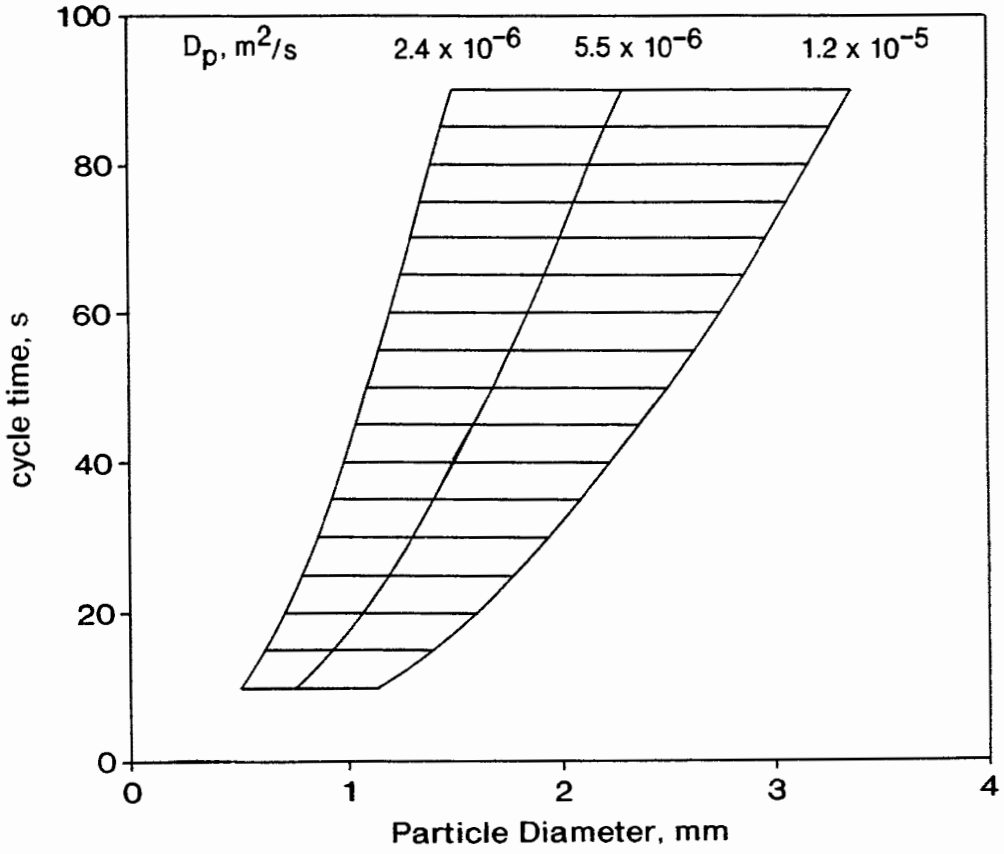



**Fig. 7**



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# INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/04383

A. CLASSIFICATION OF SUBJECT MATTER
IPC(6) :B01D 53/047
US CL :95/96, 130, 902; 96/130, 132, 144
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

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none

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

none

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| A | US 4,859,217 A (CHAO) 22 August 1989 (22.08.89). | 1-21 |
| A | US 5,071,449 A (SIRCAR) 10 December 1991 (10.12.91). | 1-21 |
| A | US 5,122,164 A (HIROOKA ET AL) 16 June 1992 (16.06.92). | 1-21 |
| A | US 5,258,060 A (GAFFNEY ET AL) 02 November 1993 (02.11.93). | 1-21 |
| A | US 5,672,195 A (MOREAU ET AL) 30 September 1997 (30.09.97). | 1-21 |
| A | US 5,711,787 A (NEILL ET AL) 27 January 1998 (27.01.98). | 1-21 |

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

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| Name and mailing address of the ISA/US<br>Commissioner of Patents and Trademarks<br>Box PCT<br>Washington, D.C. 20231<br>Facsimile No. (703) 305-3230 | Authorized officer<br>Robert H. Spitzer<br>Telephone No. (703) 308-3794 |

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| A | US 5,716,427 A (ANDREANI ET AL) 10 February 1998 (10.02.98). | 1-21 |
| A, P | US 5,769,928 A (LEAVITT) 23 June 1998 (23.06.98). | 1-21 |
| A, P | US 5,868,818 A (OGAWA ET AL) 09 February 1999 (09.02.99) | 1-21 |
| A, P | US 5,891,218 A (ROUGE ET AL) 06 April 1999 (06.04.99). | 1-21 |

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(54) Title: PRESSURE SWING ADSORPTION GAS SEPARATION METHOD, USING ADSORBENTS WITH HIGH INTRINSIC DIFFUSIVITY AND LOW PRESSURE RATIOS

(57) Abstract

A gas separation process incorporating the invention combines use of an adsorbent having high intrinsic diffusivity with a low pressure ratio PSA cycle. Further enhancements to the process are derived from the use of fast cycles, shallow beds and small particles – especially in a radial bed configuration. The combination of low pressure ratio, high rate adsorbents and fast cycles has been found to result in an unexpected simultaneous reduction in bed size factor (BSF) and power consumption. These benefits have been achieved while minimizing a decline in product recovery through use of the high rate adsorbent. The net result is a significant reduction in product cost.

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# PRESSURE SWING ADSORPTION GAS SEPARATION METHOD, USING ADSORBENTS WITH HIGH INTRINSIC DIFFUSIVITY AND LOW PRESSURE RATIOS

## FIELD OF THE INVENTION

This invention relates to pressure swing adsorption (PSA) and vacuum pressure swing adsorption (VPSA) methods for gas separation, and more particularly to a method for air separation wherein the cost for the $O_2$ product is reduced by use of a process which employs low pressure ratios and uses adsorbents exhibiting high intrinsic diffusivity.

## BACKGROUND OF THE INVENTION

Significant developments of the vacuum swing adsorption (VSA), PSA and VPSA methods for gas separation have taken place over the past thirty years, with major advances occurring during the last decade. Such processes have also been named subatmospheric, superatmospheric, and transatmospheric, respectively. Unless specifically otherwise noted, PSA will be used below to mean any or all of these processes. Commercialization of these processes can be attributed to improvements in the adsorbents, process cycles and advances in adsorber design.

Highly exchanged lithium molecular sieve adsorbents, as illustrated by Chao in U.S. Pat. No. 4,859,217, are representative of advanced adsorbents for $O_2$ production. Such advanced adsorbents are expensive and represent a significant portion of the capital cost of PSA equipment.

A dominant factor in the total energy requirement of PSA processes is the ratio of adsorption to desorption pressures. Lowering the pressure ratio is a potential method of reducing power consumption.



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Furthermore, a reduction in PSA cycle time has the potential to reduce the amount of adsorbent required. Unfortunately, the usual consequence of both of these strategies is a reduced product (e.g., $O_2$) recovery. Attempts to operate at lower pressure ratios have been accompanied by substantial decreases in adsorbent productivity, e.g. Leavitt, U.S. 5,074,892.

Smolarek, in copending U. S. Patent Application Serial No._____(Attorney docket D-20335), overcomes some of the offsetting effects of low pressure ratio through appropriate selection and operation of vacuum and compression machinery, combined with the improved flow characteristics of radial flow adsorbers. Ackley, et al. in copending U. S. Patent Application Serial No._____(Attorney docket D-20393), have taught the maximizing of product recovery and adsorbent productivity through the use of high intrinsic diffusivity adsorbents in fast cycles.

While the invention to be described below is applicable to a wide range of gas separations, PSA air separation processes aimed at the production of high purity $O_2$ (approximately 88% to 95.7% $O_2$) are of particular interest. Air separation prior art discussed below reflect this $O_2$ purity range.

Advanced adsorbents of the types mentioned above are the result of improvements in equilibrium properties. Improved $N_2$ working capacity and $N_2/O_2$ selectivity of adsorbents have been transformed into large gains in process efficiency - such benefits being obtained at the expense of higher adsorbent cost (Smolarek, et al., Gas Separation Technology, 1990). Lithium-exchanged zeolite adsorbents (LiX), in particular, have had a major impact upon the evolution of PSA air separation processes. The higher $N_2$ capacity and higher $N_2/O_2$ selectivity resulting from



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highly-exchanged LiX zeolites of low $SiO_2/Al_2O_3$ ratio have been more recently exploited for higher performance in air separation PSA processes. Other major improvements to these processes have been the introduction of vacuum for desorption, reduction from three-bed and four-bed processes to two bed cycles, and the use of modified cycle steps such as product pressurization, purge and/or equalization. These and other prior art advances in oxygen production have been summarized by Kumar ("Vacuum Swing Adsorption Process for Oxygen Production - A Historical Perspective," Sep. Sci. Technology, 31: 877-893, 1996).

Improving process efficiency and reducing the cost of the light component product can be accomplished by decreasing the amount of adsorbent required per unit of product (increasing adsorbent productivity) and by increasing the product recovery. The former is generally expressed in terms of bed size factor (BSF) in lbs adsorbent /TPDO (ton per day of contained $O_2$), while the latter is simply the fraction of light component in the feed that is captured as product. Improvement in adsorbents and reduction in cycle time are two primary methods of reducing BSF.

Considerable prior art attention has been focused upon process optimization. Reiss (Chem. Ind. XXXV, p689, 1983) emphasizes the importance of adsorbent qualities and high $O_2$ product recovery upon energy consumption in vacuum processes (VSA) for the oxygen enrichment of air. Reiss has shown that there is a minimum in the specific vacuum pump power characteristic as the desorption pressure is increased for a fixed adsorption pressure. More specifically, power per unit of $O_2$ produced initially decreases with decreasing pressure ratio and then increases such that there is an optimum pressure ratio for minimum specific



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power consumption. Concurrent to the effect of decreasing pressure ratio upon specific pump power, is the uniformly decreasing amount of $O_2$ product or a decrease in the adsorbent productivity.

Smolarek, et al. (Gas Separation Technology, 1990) achieve the objective of lowering unit $O_2$ product cost by reducing both capital cost and power consumption. Process operating parameters were developed around advanced adsorbent characteristics. Bed size was reduced by using shorter cycles, although the optimum cycle time was selected on the basis of the minimum cost. This minimum cost was established as a compromise between decreasing bed size and decreasing process efficiency as cycle time was shortened. It was also shown that two adsorbent beds was optimum. Lower overall power consumption resulted from the combination of increased $O_2$ product recovery, reduced adsorbent inventory and reduced equipment size. A reduction in the optimum pressure ratio was attributed to the advanced adsorbent. The type of adsorbent (LiX), the BSF (10001b/TPDO), and the pressure ratio (6:1), were not originally provided in the publication.

In the prior art, process optimization takes advantage of improved equilibrium adsorbent properties of higher working $N_2$ capacity and higher $N_2/O_2$ selectivity to achieve higher overall product recovery in processes utilizing vacuum desorption. Desorption pressure was increased (pressure ratio decreased) to reduce power consumption. Cycle time was decreased to keep bed size and adsorbent cost in check. Achieving minimum pressure ratio was not a primary objective of the optimization. Indeed, the reduction in pressure ratio was limited by the accompanying increase in bed size and the reduction in product recovery. The lowest pressure ratios corresponding to "optimum performance"



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achieved in these prior art were 5:1 or higher.

Much of the prior art attends to the incremental improvement in process efficiency through cycle step modification. A good example of such improvements is given by Baksh et al. (U.S. 5,518,526).

The potential benefits of low pressure ratios in achieving lower power consumption have generally been limited, due to the offsetting effects of higher BSF and lower product recovery. Although adsorbents with improved equilibrium properties allow process improvement at lower pressure ratios, reductions below a critical or limiting pressure ratio have a more severe impact upon processes incorporating advanced, high cost adsorbents. In other words, the increased adsorbent inventory accompanying lower pressure ratio has a significant impact upon the capital investment of the plant. Such critical or limiting pressure ratios were defined theoretically by Kayser and Knaebel (Chem. Eng. Sci. 41,2931, 1986; Chem. Eng. Sci. 44,1, 1989) for 5A and 13X adsorbents. The $O_2$ recovery/pressure ratio characteristics are relatively flat at higher pressure ratios (nearly constant recovery), but show a steep decline in recovery below the critical pressure ratio. The critical pressure ratio depends upon the adsorbent type and upon process operating conditions and these limits have not been well defined in practical applications. Nevertheless, the reduced recovery trends have generally discouraged PSA $O_2$ process operation at pressure ratios below about 4:1.

More recently, Rege and Yang (Ind. Eng. Chem. Res., 36: 5358-5365, 1997) presented limits for LiX zeolite and revealed an $O_2$ recovery/pressure ratio characteristic for LiX similar to those defined earlier by others for 13X and 5A adsorbents. The theoretical results of Rege and Yang suggest pressure ratios as low



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as 2:1 with little penalty in $O_2$ recovery for vacuum swing processes. They attribute this performance to the superior equilibrium properties of the adsorbent and indicate the lowest optimum BSF for their cycle to be 18kg/kg$O_2$ hr (1500 lb/TPDO). Adsorbent bed pressure drop and adsorbent diffusional resistance are neglected in the theoretical model. Power consumption was not considered in the analyses.

Leavitt (U.S. 5,074,892) proposed low pressure ratio $O_2$ production cycles in the range of 1.4 to 4.0 for adsorbents with advanced equilibrium adsorption properties, e.g. LiX, caustic digested NaX. Leavitt's primary motivation was to reduce overall process costs by reducing power consumption. Leavitt noted the importance of high $N_2$ working capacity and high $N_2/O_2$ selectivity of the adsorbent and indicated the need to achieve relatively high product recovery at the low pressure ratios in order to limit the growth in BSF. Larger amounts of purge were suggested at low pressure ratio to partially offset the lower working capacity for $N_2$. While impressive reductions in power consumption were indicated, BSF increased substantially as pressure ratio was decreased. Leavitt did not consider the effect of adsorbent intrinsic diffusivity upon process performance.

Smolarek (in copending U. S. Patent Application Serial No._____(Attorney docket D-20335) has proposed a two-bed VPSA $O_2$ cycle using a single-stage vacuum device. The adsorption pressure is in the range of 1.3 to 1.6 atm, while the desorption pressure level is between 0.4 and 0.55 atm. The preferred pressure ratio is in the range 2.75 to 3.0. A radial flow adsorber is also utilized to provide optimum flow distribution and minimal pressure drop. The higher desorption pressure increases the molar throughput and



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reduces the pressure differential across the vacuum pump, resulting in the ability to select simplified (single-stage) and less costly vacuum equipment. The lower pressure ratio results in a reduction in product recovery that, in turn, requires a higher feed input for an equivalent amount of product, i.e. compared to a higher pressure ratio cycle. Cycle time is reduced, but is limited in order to avoid introducing additional inefficiencies into the process in order to keep the BSF from increasing significantly. Smolarek claims no increase in BSF compared to the higher pressure ratio reference. The effects of adsorbent properties, high adsorbent rate characteristics in particular, upon process performance have not been addressed in the teachings of Smolarek.

Reducing cycle time is a key strategy to reducing adsorbent inventory and adsorbent cost at any pressure ratio. This is even more important for low pressure ratio cycles. While shorter cycles lead to shorter beds and higher adsorbent utilization, product recovery suffers unless adsorption rate is increased. This phenomena can be ideally characterized in terms of the size of the mass transfer zone (MTZ), i.e. the mass transfer zone becomes an increasing fraction of the adsorbent bed as the bed depth decreases. Since the adsorbent utilization with respect to the heavy gaseous component is much lower in the MTZ than in the equilibrium zone, working capacity declines as this fraction increases. When the resistance to mass transfer is dominated by pore diffusion, a decrease in adsorbent particle size leads to faster rates of adsorption and smaller mass transfer zones. Unfortunately, pressure drop across the adsorbent bed increases with decreasing particle size.



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Armond et al. (UK Pat. Appl. GB 2091121A, 1982) demonstrated a short-cycle (<45 s)/low pressure ratio (3.0) air separation process using 5A molecular sieve. This cycle was super-atmospheric, operating with a desorption pressure near ambient. Armond apparently achieved relatively small adsorbent inventory by using very small particles (0.5 to 1.2mm diameter) to facilitate a short cycle time. However, pressure drop through the bed (48 kPa/m) was quite high as was the power consumption, 0.7 kWhr/$sm^3$ $O_2$ (20 kW/TPDO). The high power consumption was presumably the result of low product recovery.

Ackley et al. in copending U. S. Patent Application Serial No._____(Attorney docket D-20393) have described improved processes utilizing advanced adsorbents with high intrinsic diffusivities relative to conventional adsorbents. Increased $O_2$ product recovery was demonstrated by increasing the rates of adsorption/desorption to create higher $N_2$ mass transfer coefficients at a fixed pressure ratio. This concept was then applied to achieve very short cycles and very low BSF while affecting only minimal decrease in product recovery.

Notaro, et al. in copending U. S. Patent Application Serial No._____(Attorney docket D-20270 ) describe a PSA air separation process, wherein the adsorbent is selected on the basis of related combinations of intrinsic rate and equilibrium properties.

Accordingly, it is a principal object of the invention to reduce product cost, reduce power consumption and increase adsorbent productivity of high performance adsorption processes for the separation of gases.



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It is a further object of the invention to provide an improved PSA process for air separation.

SUMMARY OF THE INVENTION

A gas separation process incorporating the invention combines use of an adsorbent having high intrinsic diffusivity with a low pressure ratio PSA cycle. Further enhancements to the process are derived from the use of fast cycles, shallow beds and small particles - especially in a radial bed configuration. The combination of low pressure ratio, high rate adsorbents and fast cycles has been found to result in an unexpected simultaneous reduction in bed size factor (BSF) and power consumption. These benefits have been achieved while minimizing a decline in product recovery through use of the high rate adsorbent. The net result is a significant reduction in product cost.

The high adsorption rate partially offsets the decline in product recovery that accompanies reduced pressure ratio, thus enabling fast cycle operation in shallow beds which affects an unexpected overall decrease in BSF. The present invention couples the effects of mass transfer rates (and the associated particle properties), cycle time and the bed depth to significantly improve gas separation efficiency at low process pressure ratios, i.e. improvements such as an increase in adsorbent productivity (lower BSF) and a decrease in process power consumption.

Both reduced cycle time and reduced pressure ratio cause a decrease in product recovery. This occurs in the former due to the increased fraction of bed devoted to the mass transfer zone and in the latter due to the decrease in selectivity or separation efficiency of the adsorbent. The reduced separation efficiency is substantial in vacuum desorption cycles using advanced



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adsorbents like LiX and where pressure ratio is commonly reduced by raising the desorption pressure. The application of adsorbents of high intrinsic diffusivity significantly minimizes those undesirable effects during process performance, particularly at low pressure ratios.

While this invention has been demonstrated for the case of air separation, the general methodology applies to other gas phase separations that: (1) depend upon differences in equilibrium adsorption selectivity; and (2) in which the mass transfer resistances are dominated by diffusion in the macropores of the adsorbent particles. The methodology is especially applicable to the production of oxygen in PSA processes incorporating $N_2$-selective adsorbents, e.g. type X zeolites or advanced adsorbents such as highly Li-exchanged type X or other monovalent cation-exchanged zeolites. The invention is particularly well suited to the use of adsorbents having high capacity and high selectivity (in combination with high intrinsic diffusivity) for the most selectively adsorbed (heavy) component of the gas mixture to be separated.

The prior art has focused upon increased $O_2$ product recovery and has exploited lower pressure ratios for lower power consumption only to the extent that was inherently allowed by the improved equilibrium properties of advanced adsorbents. Thus, with each new improvement in adsorbent capacity and selectivity, it was found that reasonable product recovery could be achieved at modestly lower pressure ratio. However, the lower working $N_2$ capacity and shorter cycle time dictated by lower pressure ratios results in lower adsorbent productivity (higher BSF). Prior art attempts to counter this effect by reducing cycle time even further resulted in a rapid deterioration in



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product recovery - thereby offsetting the lower power benefits of the low pressure ratio as well as limiting the potential gain in adsorbent productivity from the shortened cycle. The use of smaller particles to inhibit loss of product recovery in faster cycles is limited, in that the adsorbent bed pressure drop increases with decreasing particle size, which in turn negatively effects power consumption.

The present invention achieves higher adsorption rate through higher intrinsic diffusivity without requiring the use of very small particles (e.g. the invention preferably uses particles having an average diameter ($d_p$) $\geq$0.8mm, more preferably $\geq$1mm). However, adsorbent particle size properly selected in accordance with the pore diffusivity can be applied to further enhance the benefits of the new invention.

The invention further focuses upon lowering the product cost. This approach does not demand increased product recovery; rather it demands that the cycle time, bed depth, pressure ratio, flow rate be selected in such a manner as to achieve the lowest product cost. It has been discovered that the potential benefits of low pressure ratio can be more fully exploited by the use of adsorbents modified to have high adsorptive rate (high intrinsic diffusivity), i.e. in contrast to decreasing particle size. And surprisingly, it has been found that adsorbent productivity can be maintained or even increased as pressure ratio is decreased.

## BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a schematic of a VPSA system adapted to perform the invention hereof.



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Fig. 2 is a schematic showing one possible set of cycle steps for carrying out the invention.

Fig. 3 is a cross-sectional view of a radial bed adsorber that is particularly adapted to use with the invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

Initially, a description will be given of adsorbents that are preferred for use with a PSA process incorporating the invention. Thereafter, an overall description of the use of the preferred adsorbents in a PSA process will be considered, followed by specific examples of tests that have been run and a detailed description of a PSA system and the process steps that are performed therein.

This invention employs the unexpected results that arise from the combining of low pressure ratio PSA cycles with high adsorbent intrinsic diffusivity. The benefits of the invention may be realized in subatmospheric, transatmospheric and superatmospheric pressure ratio cycles and potentially for any bulk gas separation. The teachings are not limited to advanced adsorbents, although the benefits are likely to be most attractive for such high equilibrium performance materials. The benefits of combining low pressure ratio with high adsorbent intrinsic diffusivity, short cycles and shallow beds in PSA air separation are as follows:

- reduced power consumption
- increased adsorbent productivity (reduced BSF)
- reduced vessel size
- equivalent or reduced system pressure drop



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- reduced product cost.

The above benefits occur because the integrated effects of the high rate adsorbent used at low pressure ratio with short cycles, shallow beds and simplified compression equipment more than offsets the lower product recovery that accompanies lower pressure ratios. In fact, product recovery may be traded for a lower pressure ratio until a minimum product cost is achieved.

An adsorbent's pore structure may be manipulated through modification of adsorbent processing steps, e.g. zeolite synthesis, ion exchange, calcination, drying, etc. However, zeolite properties are very sensitive to small changes in manufacturing variables and such changes are often damaging to the adsorbent. As a result, zeolite manufacture is subject to rather strict control of process variables.

Commercial zeolite adsorbents shown in Table 1, such as 13XHP, 5AMG, LiX (2.5) and LiX (2.3), available as beads from UOP of Des Plaines, Ill. USA, were all found to have $N_2$ pore diffusivity ($D_p$) in a rather narrow range ($2.6 \times 10^{-6}$ to $3.2 \times 10^{-6}$ $m^2/s$) - as shown in Table 1, where the $N_2$ pore diffusivity is determined from a breakthrough experiment conducted with air at 1.5 bar, 300K. Porosity ($\varepsilon_p$) of these same materials falls within the range of 0.30 to 0.38 for conventional zeolites.



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Table 1: Comparison of Conventional and High Intrinsic Diffusivity Adsorbents

| Designation | Zeolite ($SiO_2/Al_2O_3$) | $\varepsilon_p$ | $d_p$ mm | $D_{p_{N_2}}$ $m^2/s$ |
|---|---|---|---|---|
| 13X | NaX(2.5) | 0.314 | 2.1 | $3.2 \times 10^{-6}$ |
| 5AMG | NaCaA | 0.315 | 0.7 | $3.1 \times 10^{-6}$ |
| Oxysiv-7 | LiX(2.5) | 0.356 | 0.55 | $2.6 \times 10^{-6}$ |
| N-1A | LiX(2.3) | 0.352 | 1.9 | $2.9 \times 10^{-6}$ |
| Z-0 | LiX(2.0) | 0.345 | 1.6 | $3.0 \times 10^{-6}$ |
| Z-1 | LiX(2.0) c.d. | 0.347 | 2.0 | $5.5 \times 10^{-6}$ |
| Z-2 | LiX(2.0) c.d. | 0.379 | 1.25 | $4.2 \times 10^{-6}$ |

Pore diffusivity = $D_p$

Porosity = $\varepsilon_p$

Caustic Digested = c.d.

Particle diameter = $d_p$

Adsorbents with improved intrinsic diffusivity have been produced according to the teachings of Chao in copending Application Serial No._____ (attorney docket D-20658), herein incorporated by reference. Two such samples (Z-1 and Z-2) are shown in Table 1 and represent a minimum of 30% to 70% improvement in pore diffusivity over that of conventional adsorbents. Sample Z-0 is an advanced LiX (2.0) adsorbent (as described by Chao in U.S. Pat. No. 4,859,217) with conventional pore diffusivity and



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equilibrium properties and used below in VPSA performance comparisons.

Chao in U.S. Patent Application Serial No_____ (attorney docket D-20658) has demonstrated various formulations and methods for producing adsorbents with intrinsic diffusivities higher than those of conventional adsorbents. The pore diffusivities of adsorbents can be enhanced by first combining a low amount of binder with zeolite in the bead-forming step, followed by caustic digestion (i.e., "c.d."). The intrinsic rate characteristics of the adsorbent can be improved further by the addition of fiber or submicron latex particulate with subsequent burn-out. Not wanting to be restricted to any one method or formulation, the detailed procedure for producing adsorbent Z-2 of the invention is herein described as one example of making such high rate adsorbents. The method of making Z-2 involves four primary steps of: bead forming, caustic digestion, ion exchange and calcination as described below.

Bead forming:

2640 gm dry weight of NaKX2.0 (wet weight 4190 gm) zeolite, 360 gm dry weight of the ECCA Tex-611 (wet weight 429.1 gm) kaolin clay and 210 gm corn starch were mulled for 15 minutes while water was pumped in at a rate of 10 ml/min. The rate of water addition was then decreased to 4 ml/min for 100 min and the mixture was mulled another 35 min. The mulled mixture was then transferred to a DBY-10R Nauta Mixer (supplied by Hosokawa Micron Powder Systems) and mixed for about one hour. The lumps were broken down to return the mixture to a powder state. Water was then added slowly by an atomizer. As the moisture of the mixture increased, beads started to form. The growth of the beads was



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stopped by adding dried bonding mix at the point when the highest yield of 8x12 size beads could be harvested.

The beads were dried in air overnight and then calcined in a Blue M oven with a dry air purge. The oven temperature was ramped up to 600 C in 2 hours then held at 600 C for 2 hours during the air purge.

Caustic digestion

1861.8 gm dry weight of calcined NaKX2.0 beads of size 6x16 with 12% binder were used for caustic digestion. To prepare digestion solution, 360 gm of NaOH (9 mole) and 251.1 gm (4.475 mole) KOH was dissolved in 7386 gm of water. To this solution, 320 ml of sacrificial NaKX2.0 beads were added and stirred at 90C for 2 hours. The solution was left to settle and 6397.7 gm supernatant was collected. To this supernatant, 1477.2 ml of water, 72.0 gm of NaOH and 50.2 gm of KOH were added to make up for the discarded caustic. The resulting solution was used as digestion solution.

The beads were loaded into two stainless steel columns of 3 inch diameter and the solution from a common reservoir was recycled through each column, at a flow rate of 30 ml/min. and temperature of 88C for 26 hours. After digestion the beads were washed by pumping 40 liter of NaOH solution (pH=12, 88C) through each column. The beads in each column were further washed with 30 liter NaOH solution (pH=8.5, 88C). The product, NaKX2.0CD, was air-dried and screened to various particle size fractions.

Ion exchange

694.5 gm dry weight of NaKX2.0CD 8x12 beads were loaded into a 3 inch i.d. glass column. A 10 inch



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layer of 3 mm Pyrex glass beads was placed at the bottom of the column to serve as a preheating zone for the solution. The column was wrapped with a heating tape. The ion exchange solution was first passed through a 15 liter 90 C preheating flask to partially remove any dissolved air to prevent air bubbles from forming that could be subsequently trapped in the column. The hot solution was then pumped into the bottom of the column.

The ion exchange solution was prepared by dissolving 2162 gm LiCl in 80 liter distilled water (0.64M) then LiOH solution was added to adjust pH of solution to 9. The solution was pumped through the column at the speed of 15 ml/min. until ten to twelve times the stochiometric amount of LiCl, for full Li exchange of the beads, had been circulated through the column. After the ion exchange was completed, the product was washed with 30 liter of 90 C distilled water at a flow rate of 60 ml/min. The pH of this water was adjusted to 9 by adding LiOH.

Drying and Calcination

The washed product was first air-dried and then dried further in a low temperature oven with ample air purge for 3 hours to bring the moisture of the beads to about 12-15%. The dried beads were calcined in a Blue M oven with ample dry air purge. The oven temperature was ramped from room temperature to 600 C in two hours and maintained at 600 C for 40 minutes. The sample was removed from the oven at 450 C and placed into a tightly sealed glass jar for cooling.

The above procedure was repeated several times in order to produce a sufficient quantity of zeolite for pilot plant testing (approximately 25 lbs.). The various production batches were blended prior to



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loading into the pilot plant adsorber vessels. Properties of the adsorbent were determined using the blended mixture.

The effective $N_2$ and $O_2$ diffusivity were determined at 1.5 bar and 300K using a combination of breakthrough experiment and detailed modeling, all of which are familiar to one skilled in the art. The effective $O_2$ diffusivity determined for the adsorbents in Table 1 is approximately 35% of the effective $N_2$ diffusivity. PSA improvements in this invention have been correlated to the effective $N_2$ diffusivity. Details of the breakthrough experiment and empirical method for determining diffusivity are provided by Ackley et al. in copending Patent Application Serial No,_____ (Attorney Docket D-20393). In addition to the above-described adsorbents, one skilled in the art will appreciate that alternative adsorbents with increased pore diffusivity can be applied in a manner similar to that described herein to achieve corresponding improvements in process performance.

The terms pore diffusivity, effective diffusivity and intrinsic diffusivity are used interchangeably herein. By the term "intrinsic diffusivity" is meant the transport property that is due to the intrinsic characteristics of the adsorbent particle including, but not limited to the structure, size, shape and length, etc. of the macropores. The term "macropores" is intended to include all of the intra-particle void volume (that volume which establishes porosity) that is typically penetrated in a standard Hg porosimetry test for zeolites. Ideally, a material's intrinsic or effective diffusivity is independent of the particle size.

PSA/ADSORBENT SYSTEM CONSIDERATIONS



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Adsorbents may be deployed by this invention in one or more distinct adsorption zones, e.g. pretreatment and main adsorbent zones. One or more adsorbents may be contained in each zone, and the zones do not have to be contained in the same adsorbent vessel. The pretreatment zone is located nearest the feed inlet and its purpose is to remove any undesirable contaminants from the feed stream. Typical contaminants in air separation are water and carbon dioxide. Those skilled in the art will appreciate the use of zeolites, activated alumina, silica gel as well as other appropriate adsorbents in the pretreatment zone. The main adsorbent zone is positioned downstream of the pretreatment zone (relative to the flow through the bed during the adsorption step) and contains adsorbent(s) selective for the primary heavy component(s) in the feed. The pretreatment zone may be excluded if there are no contaminants in the feed stream.

The PSA Processes described herein are those in which the separation of at least two components of a gas phase mixture is affected by differences in equilibrium adsorption capacities of the components in the main adsorbent, i.e. at least one component in the mixture is more selectively adsorbed at equilibrium in comparison to the adsorption of the other components. The invention uses adsorbents that have higher intrinsic diffusion rates than conventional adsorbents.

In equilibrium separations, a gas mixture is passed through a bed of adsorbent particles and the more strongly-adsorbed gas component (heavy) is retained, while the other components (light) emerge from the exit of the adsorber. At the beginning of the adsorption step, a mass transfer zone forms and moves through the bed. Nearly all of the adsorption occurs



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within this zone. The concentration of the gas to be removed decreases from its concentration in the feed mixture to a very low value over the length of this zone. In some separation processes, this zone quickly reaches a constant length (usually significantly smaller than the overall depth of adsorbent bed) and moves through the bed at a constant speed. If relatively high purity light product is desired, the adsorption step must be stopped (and subsequently followed by a regeneration step) when the front of the zone just begins to erupt at the bed exit. At this instant, the bed contains the mass transfer zone near the exit and the remainder of the bed is fully saturated with the more strongly held component in equilibrium with the feed concentration of this component.

The part of a bed located between the inlet of the main adsorption zone and the rear of the mass transfer zone is known as the "equilibrium zone". If the bed is made shorter than the length of the mass transfer zone, then the component to be removed will break through the bed immediately at the beginning of the adsorption step. The overall working capacity of the adsorbent for the heavy component is greatest when the fractional size of the mass transfer zone is kept small relative to the total size of the bed, i.e. most of the bed is saturated (equilibrium zone) at the end of the adsorption step. Faster cycles require shorter beds resulting in an increase in the fractional size of the mass transfer zone. The use of adsorbents of high intrinsic diffusivity counters this effect and enables the use of fast cycles while avoiding an increase in the fractional size of the mass transfer zone.

The size of the mass transfer zone is influenced by the particle size and the rate of diffusion of gas



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into the particle. In many cases, the greatest resistance to this diffusion is in the macropores of the particles, i.e. the gas molecules must travel through the narrow and crooked void passages inside the particle to reach the active surfaces of the adsorbent. If the particle size is reduced, this transfer occurs much more rapidly (since the path length is shortened) - resulting in a shorter mass transfer zone. There are both limitations and disadvantages to this approach as small particles lead to increased pressure drop per unit bed length, difficulty in particle retention in the bed and an increased tendency to fluidize. This approach is further limited in that it ignores the possibility of achieving process performance improvements by increasing intraparticle diffusivity directly without any reduction in particle size.

The pressure drop across the adsorbent mass in a fixed bed adsorber is dependent upon the gas velocity through the bed, the size of particles in the bed, the density of packing of the particles and the bed depth. The relationship amongst these variables is established by the well-known Ergun Equation (Chem. Engng. Progress, 1952), which is a widely used to determine the pressure loss across a fixed adsorbent bed. Simply, the pressure drop increases for smaller particles, deeper beds, higher gas flows and a denser packing.

While the particular adsorber depends upon the characteristics of the separation to be performed, adsorbent bed pressure-drop less than or equal to 0.25 psi/ft (56mbar/m) and bed depths of 4.0ft (1.2m) to 6.0ft (1.8m) have been quite common in $O_2$ production, using $N_2$-selective adsorbents, as well as in other conventional PSA processes. To compensate for the higher pressure drops resulting from reduced particle



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size, and to minimize the increase in power and tendency to fluidize, it is necessary to decrease the bed depth and/or the flow velocity through the bed. These changes lead to a reduced recovery and trade-off in bed utilization for a fixed particle size, i.e. shorter beds necessitate faster cycles leading to reduced recovery and possibly some improvement in bed utilization, although reduced velocity counters this increase in bed utilization (for a fixed inlet area) due to the resulting lower feed throughput.

Although this latter problem can be countered by increasing the flow area, there are practical limits to the size of adsorber vessels - particularly in the case of conventional cylindrical, packed adsorbers with axial flow.

Ackley et al. (copending application Serial No._____ (Attorney docket D-20393) is hereafter incorporated in its entirety by reference, and teaches a method to improve VPSA process efficiency (maximize product recovery) and to reduce product cost at a moderate pressure ratio (5:1), using fast cycles and shallow beds. The Ackley et al. method is practiced using high mass transfer rates achieved with modified adsorbents with high intrinsic pore diffusivity. Examples of such adsorbents are defined by Chao as described in copending application Serial No. _____(D-20,658), the contents of which are incorporated herein by reference.

Particle size may also be selected in combination with high intrinsic diffusivity to tailor mass transfer rates for high product recovery. While this guidance applies to any process pressure ratio, Ackley et al. do not teach the integration of these concepts with low pressure ratio. In particular, there is no indication that reducing pressure ratio could be achieved in



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conjunction with these teachings without suffering the usual expected loss in adsorbent productivity.

Smolarek (D-20,335, copending application Serial No 08/964293, the contents of which are incorporated herein by reference), teaches the importance of the desorption pressure level, bed flow and pressure drop and the use of simplified vacuum equipment in minimizing the drop in product recovery accompanying low process pressure ratio. This integration of simplified equipment and low pressure ratio leads to reduced product cost. Smolarek also notes the limitations of reducing cycle time without "engendering additional inefficiencies..." in the process. Smolarek teaches no particular adsorbents nor high rate adsorbents.

Examples: VPSA Performance Comparisons - $O_2$ Production

A series of pilot plant tests were performed with adsorbents Z-0 and Z-2 (described in Table 1 above) to demonstrate the benefits of the invention for VPSA air separation. The adsorption pressure was maintained at about 1.5 bar in all tests, while the desorption pressure was varied to achieve pressure ratios of approximately 5.1, 3.3 and 2.6. Note that the term "pressure ratio" defines the ratio of the adsorption to the desorption process pressures, when measured at the end of adsorption and desorption, respectively.

The pilot plant consisted of two beds operating out of phase and with a cycle and steps similar to that described by Baksh et al. in U.S. Patent 5,518,526, to be described in detail below. The performance results for production of $O_2$ at 90% purity are summarized in Table 2 below.



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| Test No. | Adsorbent | Pressure Ratio | Cyc. Time s | bed ht m | $O_2$ Rec. % | BSF norm. | Power norm. |
|---|---|---|---|---|---|---|---|
| 1 | Z-0 | 5.3 | 45.0 | 1.0 | 71.5 | 1.0 | 1.0 |
| 2 | Z-0 | 3.4 | 33.0 | 1.0 | 60.5 | 0.97 | 0.97 |
| 3 | Z-2 | 5.0 | 41.5 | 1.0 | 74.5 | 0.78 | 0.99 |
| 4 | Z-2 | 3.1 | 24.5 | 1.0 | 58.5 | 0.78 | 0.91 |
| 5 | Z-2 | 2.6 | 20.5 | 1.0 | 51.0 | 0.92 | 0.88 |
| 6 | Z-2 | 3.3 | 25.0 | 0.9 | 60.0 | 0.76 | 0.90 |

Table 2.

The improvement in process performance achieved by substituting a high rate adsorbent Z-2 for adsorbent Z-0 can be evaluated from the results of Tests 1 and 3 for beds of 1.0m depth. The product recovery increased from 71.5% to 74.5%, the specific power (total process power /unit $O_2$ product) decreased by only about 1% and there was a large decrease (22%) in BSF for the process using Z-2 adsorbent. Bed size factor (BSF) and total process power have been normalized to the values obtained in Test 1.

The decrease in BSF occurs as a result of the combined effects of increased product recovery, decreased cycle time, the lower density of Z-2 (about 8% lower) compared to Z-0 and the higher throughput of feed gas that is possible with a high rate adsorbent.

The significant reduction in $O_2$ product recovery (71.5% to 60.5%) accompanying a reduction in pressure ratio from 5.3 to 3.4 is demonstrated by the results of Tests 1 and 2 for an advanced adsorbent with conventional diffusivity (Z-0). These tests were



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conducted with an adsorbent bed depth of approximately 1.0m. The lower $N_2$ working capacity resulting from the lower pressure ratio necessitated the shorter cycle time. The BSF decreased slightly in contrast to the significant increases in BSF reported by Leavitt (U.S. 5,074,892). A modest decrease in power is also realized, but this is limited due to the large reduction in product recovery. Test 2 performance is representative of processes taught by Smolarek et al. (copending application Serial No. 08/964293).

The same reduction in pressure ratio was imposed for the high rate adsorbent Z-2 between Tests 3 and 4. Here again, the reduction in $O_2$ product recovery is substantial (74.5% to 58.5%), although in this case the BSF remains unchanged due to the beneficial effects of the high-rate adsorbent. The ability to maintain the BSF as the pressure ratio decreases is the result of the much shorter cycle time that can be achieved with the adsorbent of higher intrinsic diffusivity. The penalty in $O_2$ product recovery as cycle time decreases is minimized by substituting the high-rate adsorbent as is evident by comparing the results of Test 2 with those of Test 4 in Table 2. The high intrinsic diffusivity of adsorbent Z-2 also enabled the benefits of lower pressure ratio to be more fully exploited by reducing the power consumption of the process.

Comparing results from Test 1 and Test 4, a 9% reduction in specific power was achieved from the combination of low pressure ratio and high $N_2$ diffusivity. The 22% reduction in BSF for Test 4 (compared to Test 1) results from the combination of factors cited above in addition to the fact that the Z-2 adsorbent has approximately 12% higher $N_2$ working capacity compared to adsorbent Z-0 at a 3.1 pressure ratio. This working capacity difference results from



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the conversion of binder to zeolite in the caustic digestion process step. This higher $N_2$ working capacity will not necessarily occur in other treatment strategies used to achieve high intrinsic rate. Indeed, while advantageous, such higher working capacity is not essential to the practice of the basic invention.

Pressure ratio was reduced further from 3.1 to 2.6 for adsorbent Z-2. The results from Test 5 show that $O_2$ product recovery decreased to 51.0% (from 58.5% at a pressure ratio of 3.1 in Test 4), while BSF increased substantially and power continued to decrease compared to the results in Test 4. Here the potential benefits of lower pressure ratio are diminished due to the overwhelming reduction in product recovery, i.e. the pressure ratio has been reduced too much for this bed depth and adsorbent diffusivity. The use of an adsorbent with intrinsic diffusivity higher than that of adsorbent Z-2, e.g. adsorbent Z-1, would have preserved the desired simultaneous reduction of BSF and power at pressure ratios lower than 3.1. Nevertheless, the performance in Test 5 at a pressure ratio of 2.6 represents a substantial benefit in both power and BSF compared to the performance at the pressure ratio of 5.3 in Test 1.

The above results demonstrate the advantages of combining low pressure ratio with high adsorbent intrinsic diffusivity, while maintaining a constant bed depth. Cycle time was decreased only as much as necessary to compensate for the reduced $N_2$ working capacity of the adsorbent due to the lower pressure ratio. Low pressure ratio may be further exploited to gain additional performance advantage by employing faster cycles in shorter beds, i.e. provided the intrinsic diffusivity of the adsorbent is high enough.



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Test 6 was performed with a bed depth of 0.9m at the pressure ratio of 3.3. Surprisingly, the product recovery increased from 58.5% to 60.0% while BSF and power continued to decrease, i.e. compared to the results for Test 4 at about the same pressure ratio. These improvements derive from the faster cycle / shallower bed and are partially due to lower bed pressure drop and improved flow characteristics- the combination of which is enabled by the high-rate characteristics of the adsorbent.

The results of Table 2. are not intended to represent a complete definition of desirable conditions in terms of cycle time, bed depth and pressure ratio for adsorbent Z-2. Shorter beds and faster cycles may still produce additional advantages for this adsorbent at a pressure ratio of 3.3. Furthermore, lower pressure ratios (< 3:1) can be combined with adsorbents of even higher intrinsic diffusivity (such as Z-1 in Table 1.) to achieve additional performance improvements over those obtained with adsorbent Z-2.

Thus, higher intrinsic diffusivity allows both improved performance at a fixed pressure ratio and/or extension to lower pressure ratios to achieve even lower product cost.

The substantial reductions in BSF and specific power consumption represented by the results of Tests 4, 5 and 6, using adsorbent Z-2, provide significant reduction in the cost of $O_2$ product - particularly when coupled with the simplifications in vacuum equipment (e.g. single-stage vacuum pump) possible at the lower pressure ratios. The possible higher cost of the adsorbent due to specialized processing to achieve the higher intrinsic diffusivity, is expected to be more than offset by the lower BSF. The lower product recovery experienced at lower pressure ratios results



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in higher air compression costs for a given plant capacity. These additional costs are more than offset by the savings in the cost of evacuation power and equipment at these lower pressure ratios due to the higher suction pressure in the process.

This invention represents a significant advance in air separation PSA performance in that processes represented by Tests 4 and 6 simultaneously achieve BSF < 500 lb/TPDO and power consumption < 7.5 kW/TPDO.

It is evident from the air separation examples above that minimum product cost can be achieved by tailoring the pressure ratio, cycle time and bed depth for a specific intrinsic diffusivity. The best combination of conditions will vary for adsorbents with different intrinsic diffusivities. Furthermore, this invention may be applied to other gas separations to affect lower product costs through lower power consumption by implementing lower pressure ratios. Thus, the exact combination of process conditions for minimum cost depends upon the adsorbent properties and the separation of interest.

For PSA air separation, the preferred pressure ratio range is from about 2.0 to 5.0 for adsorbents with an intrinsic $N_2$ diffusivity equal to or greater than $3.5 \times 10^{-6}$ $m^2/s$. A more preferred range of pressure ratios is 1.5 to 3.5 for adsorbents with a diffusivity equal to or greater than $4.5 \times 10^{-6}$ $m^2/s$.

VPSA PROCESS/SYSTEM DESCRIPTION

Referring now to Figs. 1 and 2, the operation of a VPSA system in accordance with the invention will be described. Referring to Fig. 1, a VPSA system 10 includes a feed air inlet 12 that enables feed air to enter through inlet filter 14 and inlet silencer 16. Feed air blower 18 compresses the air for delivery to



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the system via discharge silencer 20 and conduit 22. During periods of unload, the feed blower is vented via valve 24.

Air enters adsorbers 26 and 28 via conduit 30 and open valves 32 and 34. Waste $N_2$ and contaminants are removed from the adsorber via conduit 42 and vacuum pump 44. Waste gas is silenced before venting by silencer 46. During periods of unload, vacuum pump 44 is recirculated through open valve 48.

Product reflux steps and product make is accomplished via conduit 50 and valves 52 and 54. Product make is conducted through conduit 56 and valve 58. Final product storage is contained within surge tank 60 and delivered to use point 62 via valve 64 and conduit 66. Each of adsorber beds 26 and 28 includes a high rate adsorbent that is selective for $N_2$, assuming that VPSA system is to be used for $O_2$ production.

The process steps performed by VPSA System 10 during implementation of the invention will be described in conjunction with the process step diagram shown in Fig. 2. Table 3 indicates the elapsed cycle time, start pressure and end pressure for each step of the representative cycle. One skilled in the art will recognize that the essential elements of the invention can be practiced using other cycle configurations. For the purpose of the cycle description below, the "bottom" of the vessel means the feed inlet while the "top" of the vessel is the product withdrawal point. Note that while adsorber bed 26 undergoes steps 1-6, adsorber bed 28 undergoes steps 7-12.



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TABLE 3.

SINGLE STAGE CYCLE

| Step description | Step time seconds | Start Pressure psia | End Pressure psia |
|---|---|---|---|
| *Step #1 Raising pressure feed with overlap equalization | 2.0 | 8.0 | 14.5 |
| Step #2 Raising pressure feed with overlap product pressurization | 2.0 | 14.5 | 19.0 |
| Step #3 Raising pressure feed | 2.0 | 19.0 | 22.0 |
| Step#4 Constant pressure feed with Make-product | 2.0 | 22.0 | 23.0 |
| Step #5 Constant pressure feed with Make-product and purge | 4.0 | 23.0 | 23.0 |
| Step #6 Falling pressure equalization | 2.0 | 23.0 | 19.0 |
| ----------half cycle---------- | | | |
| Step #7 Falling pressure evacuation with overlap equalization | 2.0 | 19.0 | 13.0 |
| Steps #8 & #9&#10 Failing pressure evacuation | 6.0 | 13.0 | 7.0 |
| Step #11 Constant pressure evacuation with Oxygen purge | 4.0 | 7.0 | 7.0 |
| Step #12 Raising pressure evacuation with overlap equalization | 2.0 | 7.0 | 8.0 |



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Hereafter further details are given of each of the steps listed in Table 3 and illustrated in Fig. 3.

Step #1:

Raising pressure feed with overlap equalization:

This step initiates the feed air pressurization period. Air is fed to the bottom of adsorber bed 26 (for example) from compressor 18 and via conduit 22. The pressure raises rapidly within adsorber bed 26 from about 8.0 psia to about 14.5 psia. The step is 2 seconds in duration. Oxygen-rich equalization gas is simultaneously introduced into the top of adsorber bed 26 from adsorber bed 28 during this step.

Step #2

Raising pressure feed with overlap product pressurization:

This step continues the feed air pressurization period. Air is fed to the bottom of adsorber bed 26 from compressor 18. The pressure continues to rise during this step from 14.5 psia to about 19.0 psia, the step is 2 seconds in duration. Oxygen repressurization gas taken from the product surge tank 60 is simultaneously introduced into the top of adsorber bed 26 during this step.

Step #3

Raising pressure feed:

Feed air only is introduced into adsorber bed 26 and the top of the vessel is closed. The pressure rises from 19.0 to about 22.0 psia during this 2 second step. Feed air is supplied by compressor 18 during this step.

Steps #4-#5



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Constant pressure feed with make-product:

Feed air is introduced into the bottom of adsorber bed 26 while oxygen product is removed from the top. The pressure remains relatively constant during this 6 second period at 22.0 to 23.0 psia. The feed air is supplied by compressor 18. The oxygen product is supplied to oxygen surge tank 60 as well as to adsorber bed 28 as oxygen purge during step 5. The purity of the oxygen product remains relatively constant (90%) during the product make steps.

Step #6

Falling pressure equalization:

The residual oxygen product at the top of adsorber bed 26 is withdrawn during this step from the top of the vessel. There is no flow from the bottom of adsorber bed 26. The vessel pressure falls from 23.0 to about 19.0 psia during this 2 second step. Compressor 18 is vented during this step.

Step #7

Falling pressure evacuation with overlap equalization:

Waste nitrogen is removed from the bottom of adsorber bed 26 through vacuum pump 44. The pressure falls from 19.0 psia to about 13.0 psia during this 2 second step. The oxygen concentration starts at about air purity and falls rapidly. The equalization falling step continues as the oxygen-rich gas is removed from the top of adsorber bed 26.

Steps #8-#10

Falling pressure evacuation:

Waste nitrogen is removed from the bottom of adsorber bed 26 through the vacuum pump 44. The



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pressure falls from 13.0 psia to about 7.0 psia during this 6 second period. The top end of adsorber bed 26 is closed during this step. The oxygen concentration in the waste gas reaches its minimum at the end of step 10.

Step #11

Constant pressure evacuation with oxygen purge:

The minimum evacuation pressure is reached and oxygen purge is introduced to the top of adsorber 26. The pressure remains constant during this 4 second step at 7.0 psia due to the matching of the purge flow to the evacuation flow.

Step #12

Raising pressure evacuation with overlap equalization:

Vacuum pump 44 continues to remove waste gas from the bottom of adsorber bed 26 while oxygen equalization gas is added to the top thereof. The pressure rises during this step because the oxygen equalization flow is larger than the evacuation flow. The pressure raises from 7.0 to about 8.0 psia during the 2 second

The cycle described above is illustrative only, and the essential features of the invention can be practiced using other adsorptive cycles.

Radial Bed Configuration

The VPSA method of the invention is particularly suited to use with a radial bed structure due to the low pressure ratios that are employed. Such a radial bed configuration is shown in Fig. 3. Feed and waste gas is supplied through a conduit 80 to a radial flow distribution assembly 82 where the inlet gas flows to the outside walls of vessel 84.



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The gas, which now is uniformly distributed in the lower head 86, is supplied to adsorption bed 88 via outside vertical flow paths 90, flowing upwardly through straight or tapered flow passages. The gas then flows through adsorber bed 88 in an inward radial manner. The gas exiting at the product end of adsorber bed 88 is collected inside vertical flow paths 92 and flows downward. The product gas is collected in a conical collection assembly 94 at the bottom of vessel 84. The collected product gas exits the vessel through conduit 96, contained within feed conduit 80. Alternatively, conduit 96 may be oriented such that the product gas is withdrawn at the top of the vessel in Figure 3.

Vessel flow distribution is critical to successful operation of a VPSA process and a major contributor to flow distribution is the channel pressure differential between the feed and product ends of the adsorber. This pressure differential is a combination of frictional pressure losses and velocity-head changes in the flowing gases. These two effects tend to cancel when flow is entering a channel, and are additive when flow is exiting a channel. The degree of cancellation and addition is affected by the internal geometry of the chamber, i.e. through design of tapered channels. All VPSA processes by nature reverse the gas flow direction periodically to accomplish the subsequent adsorption and desorption process steps.

SUMMARY

The main features of the invention are as follows:

1) low pressure ratio combined with an adsorbent of high pore diffusivity;

2) low pressure ratio combined with an adsorbent of high pore diffusivity, such that BSF < 500 lb/TPDO



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and total power < 7.5 kW/TPDO, more preferably such that BSF < 300 lb/TPDO and total power < 7.0 kW/TPDO;

3) shallow beds combined with 1) above, followed by shallow beds and shorter cycles combined with 1) above, and with increasingly preferred combinations of bed depth/cycle time of <1.2m/<40s, <0.9m/<30s and <0.6m/ <20s;

4) single stage vacuum device combined with 1) above;

5) radial flow adsorber vessel combined with 1) or with 4) above;

6) a pressure drop across the adsorbent bed not exceeding 1.5 psi in desorption and adsorption is preferred, and a pressure drop across the adsorbent bed not exceeding 1.0 psi in desorption and adsorption is most preferred in combination with 1) above;

7) particle size tailored to the intrinsic diffusivity to maximize mass transfer rate without suffering significant increase in pressure drop combined with 1) above, the most preferred particle size range is for an average particle diameter of about 0.8mm to about 1.6mm;

8) adsorbents with enhanced capacity and/or selectivity combined with 1) above.

The fundamental object of the invention is to reduce product cost by combining adsorbents with high intrinsic diffusivity with low pressure ratios in PSA processes. The invention is directed at equilibrium-based adsorption separation processes with mass transport dominated by intraparticle pore diffusion.

While the examples have been directed at air separation using a single main adsorbent, the invention is not limited to binary mixtures, nor to air as a feed nor to a single main adsorbent. When more than a single separation is to be achieved, it is feasible to



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include one or more adsorbents as main adsorbents - each adsorbent responsible for a different separation or a different level of the same separation. Thus, the properties (particularly those related to the rate of adsorption) of the different adsorbent materials in the main adsorbent zone are selected to maximize all of the separations required of the process. Examples of such processes include the recovery of $H_2$ from $H_2/CO/CO_2/CH_4$ mixtures, the removal of $H_2O$ and $CO_2$ from air, separation of Ar from air or $N_2$ or $O_2$, drying of process streams and the recovery of $CO_2$ from flue gases or from $H_2$ PSA tail gas.

The invention may be applied to adsorption processes intended to recover either the light or the heavy product or both. In all cases, the pore diffusivities of the key adsorbates are to be maximized. The benefits of low pressure ratio are likely to be greatest in bulk separations in which the heavy component is also the major component of the feed gas, particularly in processes incorporating vacuum desorption. Appropriate matching of desorption pressures to suction pressures of existing vacuum equipment provides a means of simplifying and reducing the cost of the equipment, e.g. by reducing the number of compression stages, eliminating aftercooling, etc.

Air separation using $N_2$ -selective adsorbents is a prime example where lower pressure ratio represents a substantial potential reduction in power consumption.

Type X zeolite adsorbents are suggested for air separation, most preferably highly-exchanged LiX as described by Chao (cited above). Type X, type A, and naturally occurring zeolites containing monovalent, multivalent or mixed cations are also applicable to the present invention when appropriately produced to achieve high intrinsic pore diffusivity.



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It should also be clear that the present invention may be practiced with various deployments of adsorbents in the main adsorbent zone, e.g. layers and mixtures of adsorbents of various types or of the same type but with varying adsorption and/or physical characteristics. For example, the low pressure ratio/high diffusivity concepts of this invention can be applied to the layered beds suggested by Ackley in copending U.S. Patent Application Serial No. 837,411 (attorney docket D-20347). While the invention has been described for adsorbent zones consisting of a fixed bed of adsorbent particles, the invention may be practiced using alternative adsorbent deployment configurations, e.g., monoliths, adsorbent agglomerates dispersed on a fibrous substrate, etc.

Additional improvements may be achieved by appropriate selection of adsorbent particle size in conjunction with the adsorbent intrinsic diffusivity and combining in a low pressure ratio cycle. For air separation and for the preferred pressure ratio ranges and $N_2$ intrinsic diffusivities noted above, the preferred average particle size (diameter) is between 0.8mm and 1.6mm.

This latter issue can be addressed best using radial flow, where the flexibility in selecting flow area, bed depth and flow distribution is greatest in regard to controlling pressure drop at acceptable levels. Radial flow adsorbers provide inherent constraint of the adsorbent such that fluidization can be avoided when using small particles.

This invention applies generally to a full range of cycle steps and process conditions, e.g. temperatures, pressures, feed velocity, etc. Likewise, its concepts can be applied to single-bed as well as multi-bed processes operating with subatmospheric



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(VSA), transatmospheric (VPSA) or superatmospheric (PSA) cycles. The concepts described here are not limited to any particular adsorber configuration and can be effectively applied to axial flow, radial flow, lateral flow, etc. adsorbers. The adsorbent may be constrained or unconstrained within the adsorber vessel.

It should be understood that the foregoing description is only illustrative of the invention. Various alternatives and modifications can be devised by those skilled in the art without departing from the invention. Accordingly, the present invention is intended to embrace all such alternatives, modifications and variances which fall within the scope of the appended claims.



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CLAIMS

1. A pressure swing adsorption method comprising repetitions of a cycle of N steps, said method adapted to separate components of a gas mixture into at least a first component and a second component by selective adsorption of said first component into a bed of adsorbent, said method comprising the steps of:

a) during adsorption steps of a cycle, raising a pressure of a feed of said gas to said bed to an adsorption pressure so as to enable adsorption of said first component by said adsorbent, said adsorbent exhibiting an intrinsic diffusivity for said first component that is equal to or greater than $3.5 \times 10^{-6}$ $m^2/sec$;

b) during desorption steps of said cycle, depressurizing said bed to a desorption pressure so as to desorb said first component from said adsorbent, a ratio of pressures of said adsorption pressure to said desorption pressure falling within a preferred range of less than 5.0.

2. The pressure swing absorption method as recited in claim 1, wherein said ratio of pressures falls within a more preferred range of about 3.5 to 5.

3. The pressure swing adsorption method as recited in claim 1, wherein said intrinsic diffusivity of said adsorbent for said first component is equal to or greater than $4.0 \times 10^{-6}$ $m^2/sec$ and said ratio of pressures falls within a preferred range of about 1.5 to 3.5.



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4. The pressure swing adsorption method as recited in claim 3, wherein a lowest pressure during a cycle is within 6 to 9 psia and a highest pressure during said cycle is within about 18 to 24 psia.

5. The pressure swing adsorption method as recited in claim 3, wherein said gas is air, said first component is nitrogen and said second component is oxygen.

6. The pressure swing adsorption method as recited in claim 1, wherein said adsorbent is a particulate and has an average particle diameter between 0.8mm and about 1.6mm.

7. The pressure swing adsorption method as recited in claim 1, wherein said adsorbent is a type X zeolite with a $SiO_2/Al_2O_3$ ratio less than or equal to 2.5 and exchanged with Li (>70%).

8. The pressure swing adsorption method as recited in claim 1, wherein said bed of adsorbent has a dimension, in a direction of flow of said gas through said bed of adsorbent, of less than about 1.2m and said method performs steps a) and b) in a time of less than about 40 seconds.

9. The pressure swing adsorption method as recited in claim 1, wherein said bed of adsorbent has a dimension, in a direction of flow of said gas through said bed of adsorbent, of less than about 0.9m and said method performs steps a) and b) in a time of less than about 30 seconds.



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10. The pressure swing adsorption method as recited in claim 1, wherein said bed of adsorbent has a dimension, in a direction of flow of said gas through said bed of adsorbent, of less than about 0.6m and said method performs steps a) and b) in a time of less than about 20 seconds.

11. A pressure swing adsorption system for performing a gas separation method comprising repetitions of a cycle of N steps, said separation method for separating components of a gas mixture into at least a first component and a second component by selective adsorption of said first component into a bed of adsorbent particles, said system comprising:

an adsorbent that is selective for said first component, said adsorbent exhibiting an intrinsic diffusivity for said first component that is equal to or greater than $3.5 \times 10^{-6}$ $m^2/sec$, said system exhibiting both a reduction in bed size factor (BSF) and specific power consumption relative to a pressure swing adsorption system incorporating adsorbents of lower intrinsic diffusivity; and

control means for controlling a ratio of adsorption pressure to desorption pressure across said bed within a preferred range of less than about 5.0.

12. The system as recited in claim 11 wherein said control means controls said ratio to fall within a more preferred range of about 3.5 to 5.

13. The system as recited in claim 11, wherein said control means controls a pressure drop across said



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bed during said method so as to not exceed about 1.0 psi during desorption and during adsorption.

14. The system as recited in claim 11, wherein said control means controls a pressure drop across said bed during said method so as to not exceed about 1.5 psi during desorption and during adsorption.

15. The system as recited in claim 11, wherein said intrinsic diffusivity of said adsorbent particles for said first component is equal to or greater than $4.5 \times 10^{-6}$ $m^2$/sec and said control means controls said ratio to fall within a preferred range of about 1.5 to 3.5.

16. The system as recited in claim 11, wherein said bed is arranged in a radial annular configuration, and said gas mixture exhibits a flow pattern that is transverse to said annular configuration.

17. The system as recited in claim 16, further comprising:

a single stage vacuum pump coupled to said bed for withdrawing an adsorbed component from said bed during a desorption phase of said gas separation method.

18. The system as recited in claim 11, wherein said gas is air, said first component is nitrogen and said second component is oxygen.

19. The system as recited in claim 18, wherein the system exhibits a bed size factor < 500lb/TPDO and a specific power consumption < 7.5 kW/TPDO.



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20. The system as recited in claim 11, wherein said adsorbent is a particulate and has an average particle diameter of about 0.8mm and about 1.6mm.

21. The pressure swing adsorption method as recited in claim 11, wherein said adsorbent is a type X zeolite with a $SiO_2/Al_2O_3$ ratio less than or equal to 2.5 and exchanged with Li (>70%).

22. The system as recited in claim 11, wherein said bed of adsorbent has a dimension, in a direction of flow of said gas through said bed of adsorbent, of less than about 1.2m and said control means controls said system to perform adsorption and desorption steps in a time of less than about 40 seconds.

23. The system as recited in claim 11, wherein said bed of adsorbent has a dimension, in a direction of flow of said gas through said bed of adsorbent, of less than about 0.9m and said control means controls said system to perform adsorption and desorption steps in a time of less than about 30 seconds.

24. The system as recited in claim 11, wherein said bed of adsorbent has a dimension, in a direction of flow of said gas through said bed of adsorbent, of less than about 0.6m and said control means controls said system to perform adsorption and desorption steps in a time of less than about 20 seconds.



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SUBSTITUTE SHEET (RULE 26)

# Fig. 1

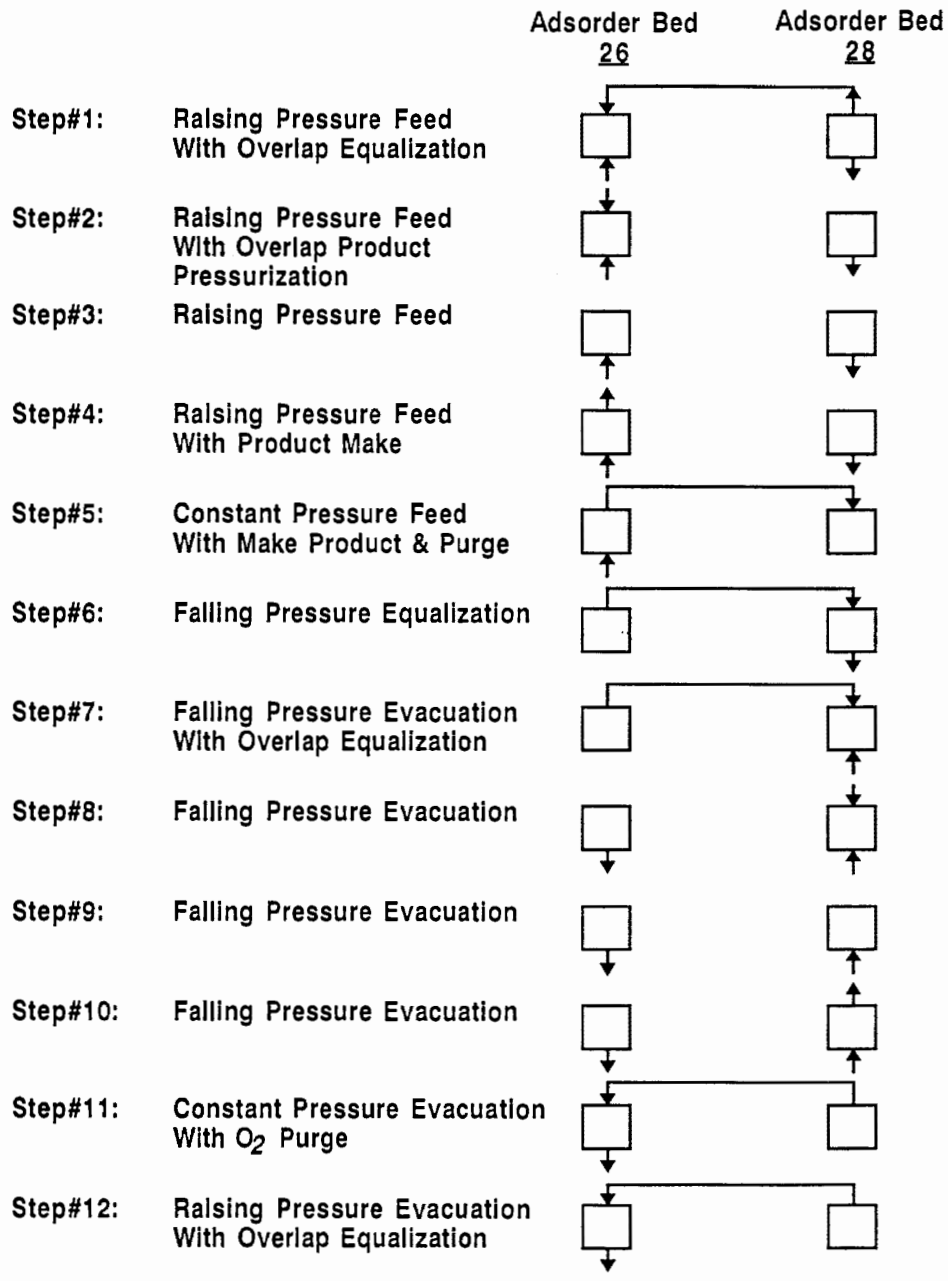



**Fig. 2**



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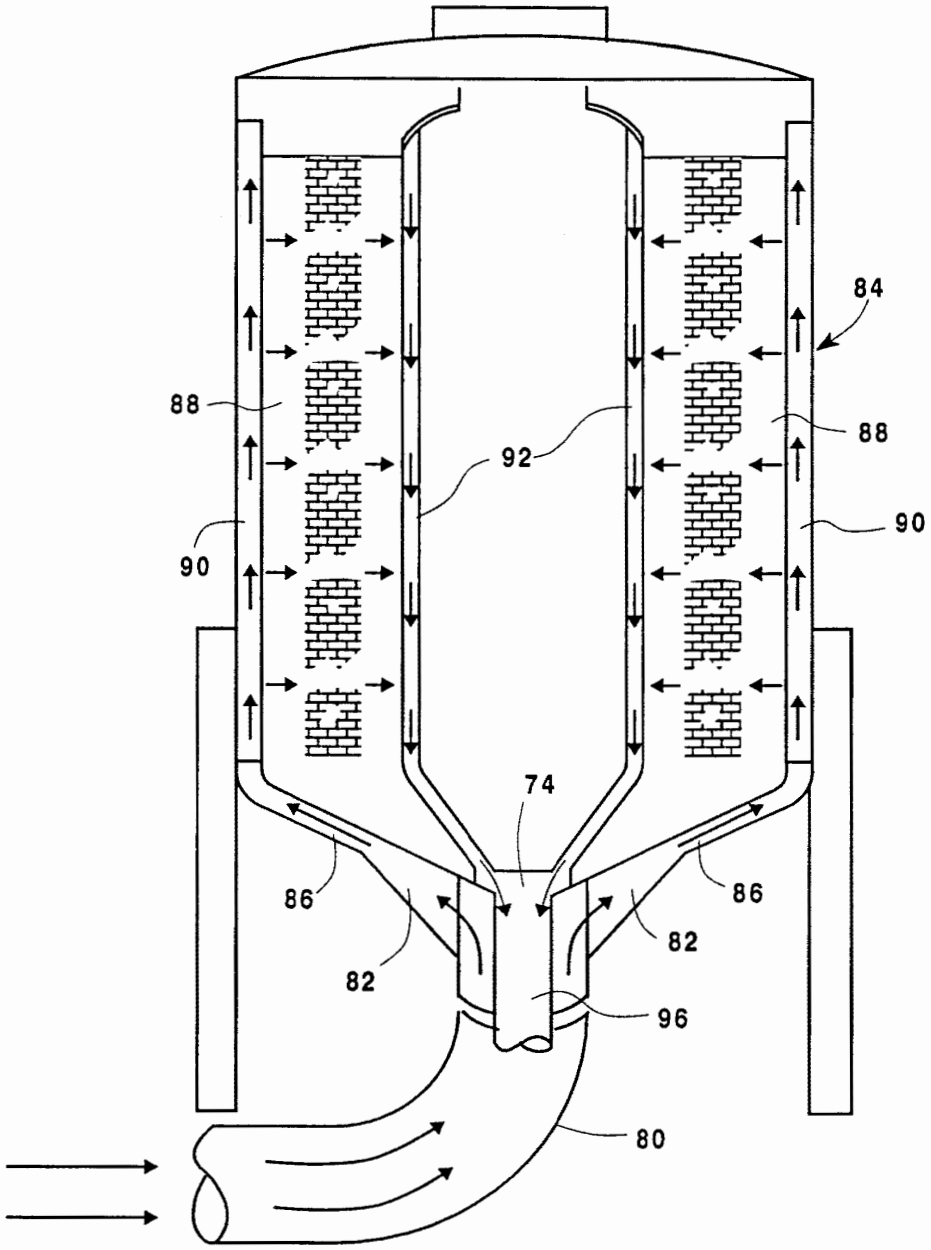



Fig. 3



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# INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/04384

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(6) :B01D 53/053
US CL :95/96, 130, 902; 96/113, 114, 130, 144
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)

U.S. : 95/96-98, 100-105, 130, 902; 96/108, 113, 114, 130, 143,144

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

none

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

none

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| A | US 4,859,217 A (CHAO) 22 August 1989 (22.08.89). | 1-24 |
| A | US 5,071,449 A (SIRCAR) 10 December 1991 (10.12.91). | 1-24 |
| A | US 5,122,164 A (HIROOKA ET AL) 16 June 1992 (16.06.92). | 1-24 |
| A | US 5,258,060 A (GAFFNEY ET AL) 02 November 1993 (02.11.93). | 1-24 |
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| A | US 5,711,787 A (NEILL ET AL) 27 January 1998 (27.01.98). | 1-24 |

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

| | | | |
|---|---|---|---|
| * | Special categories of cited documents: | "T" | later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention |
| "A" | document defining the general state of the art which is not considered to be of particular relevance | "X" | document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone |
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| "O" | document referring to an oral disclosure, use, exhibition or other means | | |
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| Date of the actual completion of the international search | Date of mailing of the international search report |
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**INTERNATIONAL SEARCH REPORT**

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PCT/US99/04384

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
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| A, P | US 5,868,818 A (OGAWA ET AL) 09 February 1999 (09.02.99). | 1-24 |
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Form PCT/ISA/210 (continuation of second sheet)(July 1992)★

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## (54) Method for separation of gaseous mixtures of oxygen, argon and nitrogen

(57) The method according to the invention consists in separation of gaseous mixture of oxygen, argon and nitrogen by a method of variable-pressure adsorption PSA on molecular carbon or zeolite sieves, selectively adsorbing oxygen from argon and nitrogen, by selective adsorption of oxygen at increasing pressure with simultaneous carrying of argon fraction away to the buffer tank, rinsing of argon out with pure oxygen under elevated pressure, higher than the final pressure of filling, with simultaneous carrying of argon away through the buffer tank and desorption of pure oxygen at the pressure near to the atmospheric pressure.

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## Description

**[0001]** The object of the invention is a method for separation of gaseous mixtures of oxygen, argon and nitrogen by variable pressure adsorption on molecular carbon or zeolite sieves in order to produce high purity oxygen.

**[0002]** Development of technologies of variable pressure adsorption, commonly called PSA, allows effective oxygen production in a small scale. The known PSA technologies of air separation on molecular zeolite sieves allow to produce oxygen of the purity at most 95.5% of $O_2$ by volume and the remaining impurity is then only argon. Generally in oxygen generators, where air separation is run on molecular zeolite sieves of NaX, CaX or CaA type, there is obtained oxygen of the purity 95.0 ÷ 95.3% by volume of $O_2$, containing 4.5% of argon and 0.2 - 0.5% of nitrogen by volume.

**[0003]** Oxygen of such purity can be used in the majority of oxygen processes but it cannot be used for cutting steel, steel sheets of high thickness. It cannot be used for automatic steel sheet cutting. For such purposes it is possible to use oxygen of the purity at least 99.8 ÷ 99.9% by volume of $O_2$, preferably of the purity 99.99% by volume of $O_2$.

**[0004]** Oxygen of high purity, above 99.2% by volume of $O_2$ can be produced of oxygen of the purity not lower than 95.0% by volume of $O_2$ containing 0.5% by volume of $N_2$ and 4.5% by volume of Ar, preferably of oxygen of the purity 95.5% by volume of $O_2$ polluted only with argon, produced by air separation on molecular zeolite sieves, applying known PSA technologies of air separation and next by separation of oxygen of such purity by a method of variable pressure adsorption on molecular carbon or zeolite sieves, selectively adsorbing oxygen from argon and nitrogen in a kineticly selective process of variable pressure adsorption. Up to now by this method it has been possible to produce oxygen of the purity higher than 99.7% by volume of $O_2$.

**[0005]** According to the US patent no. 4.190.424, the method for high purity oxygen production consists in the fact that oxygen containing typically 90.0% by volume of $O_2$, 4,5% by volume of Ar and 5.5% by volume of $N_2$, obtained in the PSA process on molecular zeolite sieves, is exposed to purification up to the purity of 99.7% by volume of $O_2$ in the second PSA stage on molecular carbon sieves by selective adsorption of oxygen from nitrogen and argon. There is used a molecular carbon sieve of the pore size 3 - 10 x $10^{-1}$ nm. According to the US patent no.4.813.979 high purity oxygen is obtained from oxygen 95.0% by volume in the PSA process on a molecular carbon sieve selectively adsorbing argon. The process of oxygen purification consists in the fact that in a double-adsober installation, the carbon bed is filled with oxygen and next oxygen is let under elevated pressure through the bed and purified oxygen is received at the other end when in the second adsorber there is run desorption of adsorbed argon by counter-current pressure reduction. The adsorbers work alternatively in cycles lasting 3 - 9 seconds. The solution can be applied for purification of small oxygen streams, for instance for medical purposes.

**[0006]** According to the US patent no. 5.226.933, for oxygen purification there is proposed application of silver mordenite Ag (18)M selectively adsorbing argon from oxygen. From oxygen containing 95.0% by volume of $O_2$ and 5% by volume of Ar there is obtained oxygen of the purity 99.7% by volume. The process requires high pressure and gives low efficiency of oxygen production.

**[0007]** According to the European application 0132239, the method for high purity oxygen production consists in selective adsorption of oxygen from argon in a kineticly selective process on natural or modified clinoptylolites.

**[0008]** The US patent no. 5.137.549 presents a method for high purity oxygen production, which is the nearest to the solution presented in this specification. The clue of the proposed method for high purity oxygen production consists in the fact that oxygen of the purity 95.5% by volume polluted only with argon is obtained in the first PSA stage on molecular zeolite sieves. Next oxygen is brought to a buffer tank, wherefrom it is taken to the second PSA stage, containing two adsorbers filled with molecular carbon sieve selectively adsorbing oxygen from argon. The process of oxygen purification is run in the following technological operations:

- filling; to the adsorber there is supplied oxygen of the purity 95.5% by volume, the pressure in the adsorber is elevated to the pressure of adsorption,
- adsorption; through the carbon bed there is let oxygen of the purity 95.5% by volume, from the other end there is carried away gas enriched with unadsorbed argon,
- cocurrent pressure reduction and rinsing out; to the adsorber there is supplied compressed purified oxygen of the purity 99.2% by volume, obtained in the desorption phase, by means of which there is rinsed out oxygen enriched with argon, contained in the sorbent free space. The stream of rinsed oxygen is directed in cocurrent to the second adsorber, due to which the pressure in the second adsorber grows to the intermediate, and the pressure in the first adsorber is reduced,
- counter-current desorption; from the adsorber there is carried out pure oxygen desorbing from the molecular carbon sieve.

**[0009]** Oxygen is compressed and partially recycled to the operation of rinsing out. The process is run alternatively in two adsorbers. When in one adsorber there are run operations of filling and adsorption, in the second adsorber oxygen desorption takes place.



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**[0010]** The described methods for separation of gaseous mixtures of oxygen, argon and nitrogen on molecular carbon sieves or molecular zeolite sieves by the method of variable pressure adsorption PSA in a kineticly selective PSA process do not allow to produce oxygen of the purity higher than 99.7% by volume.

**[0011]** The PSA process according to the American patent no. 5.137.549 allows to obtain oxygen of the purity typically equal to 99.2% ÷ 99.3% by volume of $O_2$, at most 99.5% by volume of $O_2$ with relatively low efficiency of oxygen yield. Such oxygen purity is not satisfactory for automatic waste free cutting of large thickness steel sheets.

**[0012]** The purpose of this invention was elaboration of a method for production of oxygen of the purity above 99.7%.

**[0013]** Unexpectedly it has been found out that joining the filling operation AR1, in which polluted oxygen is supplied to an adsorber with simultaneous carrying away of argon fraction from the other end of the adsorber to a buffer tank causes quick carrying away of argon contained in the final sorbent layers outside the sorbent zone, which reduces the time stay and contact with the sorbent, due to which the quantity of adsorbed argon is reduced and in consequence the purity of obtained oxygen increases. Unexpectedly it has been also found that supplying pure oxygen to the operation of filling with pure oxygen AR2 in cocurrent to the gas flow in the operation AR1 and under the pressure higher than the final pressure in the operation AR1 and simultaneous carrying away of argon fraction from the other end of the adsorber to the buffer tank causes rinsing out of argon adsorbed in the sorbent and after short time of contact with the sorbent - rinsing of argon out from the buffer tank, which gives further improvement of obtained oxygen purity. It has been also found unexpectedly that supplying pure oxygen to the adsorber in the operation of rinsing with pure oxygen R, in cocurrent to the gas flow in the operation AR1, but at the pressure higher than the final pressure in the operation AR1 and simultaneous carrying away of argon to the buffer tank and carrying away of the argon fraction from the buffer tank causes further rinsing of adsorbed argon out of the sorbent and taking it outside the sorbent zone and from the installation in an oxygen stream in the form of the argon fraction. It has been also found out that in the operations of pressure reduction E1 and pressure elevation 1E simultaneously proceeding in two adsorbers it is not necessary to equalize pressures between the joined adsorbers but the pressure in the adsorber should be reduced only to the level in which desorbing oxygen rinses out the argon residue contained in the free sorbent space.

**[0014]** Joining together in sequence the operations AR1, AR2 and R with continuous carrying away the argon fraction to the buffer tank and further carrying away the argon residue from the operation of pressure reduction E1 to the second adsorber causes that before the desorption is started, in the sorbent there is adsorbed actually pure oxygen and in the free space there is also pure oxygen. Eventually in the operation of desorption from the adsorber there is desorbed pure oxygen slightly polluted with argon and the losses of oxygen in the argon fraction are small.

**[0015]** The clue of the invention concerning kineticly selective process of variable pressure adsorption of separation of gaseous mixtures containing oxygen, argon and nitrogen on molecular carbon or zeolite sieves consists in the fact that the process is run in an installation including at least two adsorbers 1A and 1B and a buffer tank of argon fraction and the complete PSA cycle includes the following technological operations proceeding in sequence:

- operation of filling AR1, in which to the adsorber within the period of 30 ÷ 60 s there is supplied oxygen and at the same time the argon fraction is carried away from the adsorber to the buffer tank, the pressure in the adsorber and the buffer tank increases from the pressure 300 ÷ 400 kPa to 500 ÷ 700 kPa,
- operation of filling with pure oxygen AR2, in which to the adsorber within the period of 0 ÷ 15 s, in cocurrent to the gas flow in the operation AR1, there is supplied pure oxygen produced during the desorption operation and next compressed and at the same time the argon fraction if carried away from the adsorber to the buffer tank, the pressure in the adsorber and the buffer tank increases from the pressure 500 ÷ 700 kPa to 600 + 800 kPa,
- operation of rinsing out R, in which to the adsorber 1A or 1B within the period of 10 ÷ 30 s, in cocurrent to the gas flow in the operation AR1, there is supplied pure oxygen produced in the operation of desorption and next compressed, and at the same time the argon fraction is carried away from the adsorber to the buffer tank thereafter the argon fraction is carried away from the buffer tank, while the pressure is kept on a stable level 600 ÷ 800 kPa.
- operation of pressure reduction E1, in which to the adsorber 1A and 1B there is supplied pure oxygen and at the same time from the adsorber within the period of 5 ÷ 20 s, in cocurrent to the gas flow in the operation AR1 gas is carried away and supplied in cocurrent to the second adsorber correspondingly 1B and 1A, in which the operation of pressure increase 1E is run, while the pressure is reduced from 600 ÷ 800 kPa to 400 ÷ 600 kPa, but the pressures between the joined adsorbers do not equalize,
- operation of desorption D, in which from the adsorber within the period 40 ÷ 105 s, in cocurrent to the gas flow in the operation AR1, there is carried away pure oxygen and the pressure in the adsorber is reduced to the pressure 100 ÷ 110 kPa, and in case of oxygen suction by means of a vacuum pump, the pressure is reduced to 10 ÷ 50 kPa,
- operation of pressure elevation 1E, in which to the adsorber 1A or 1B within the period of 5 ÷ 20 s, in cocurrent to the gas flow in the operation AR1, there is supplied gas produced in the operation of pressure reduction E1, and the pressure is increased to 250 ÷ 350 kPa, while the pressures between the joined adsorbers do not equalize,
- operation of reflexive filling RR, in which to the adsorber 1A or 1B within the period 1 ÷ 2 s, in counter-current to the gas flow in the operation AR1, there is supplied argon fraction contained in the buffer tank 2, but the pressure



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between the adsorber and the buffer tank do not equalize,

It is preferable if:

- the ratio of the volume of the buffer tank 2 to the volume of the molecular carbon or zeolite sieve contained in each adsorber 1A or 1B is within the range 0.4 ÷ 0.7,
- the radio of the quantity of pure oxygen recycled to the process in the operations AR2 and R and E1 to the quantity of oxygen introduced with the raw material is within the range 0.6 ÷ 1.2,
- the ratio of pressure increase in total in the operations AR1 and AR2 to the pressure increase only in the operation AR1 is within the range 1.0 ÷ 2.0,
- the time of the complete PSA cycle is 90 ÷ 240 s, while the ratio of time of the operation AR1 to the total time of the operation AR2 and R is 0.7 ÷ 1.4.

**[0016]** The method for separation of gaseous mixtures of oxygen, argon and nitrogen according to the invention is presented on examples and drawings where fig.1 presents a flowsheet of the installation, fig.2 presents a diagram of gas streams flow in the complete PSA cycle, and fig.3 presents pressure course in the adsorber during the complete cycle.

**[0017]** Separation of gaseous mixtures of oxygen, argon and nitrogen is run in a PSA installation consisting of two adsorbers 1A and 1B filled with molecular carbon or zeolite sieve, selectively adsorbing oxygen from argon and nitrogen, and a buffer tank 2. The process of variable pressure adsorption is run by means of controlled valves marked KV1AB ÷ KV6A, KV6B. The installation includes two reflux valves ZZA and ZZB and a hand valve ZR serving for control of the operation of pressure reduction and elevation. The conduit 101 supplies polluted oxygen, the conduit 102 supplies to the installation pure oxygen produced in the desorption phase and next compressed. The conduit 103 supplies the argon fraction to the buffer tank, the conduit 104 carries argon fraction away from the buffer tank. The conduit 105 carries away from the installation pure oxygen obtained in the operation of desorption, which is next compressed and partially recycled to the process.

**[0018]** The double-way valve KV1AB serves for alternating supply to the adsorbers 1A or 1B polluted oxygen from the conduit 101 in the operation AR1 or pure oxygen from the conduit 102 in the operation AR2, R and E1.

**[0019]** The valves KV2A and KV2B are open and they serve for supplying to the adsorbers, respectively 1A and 1B, polluted oxygen in the operations of filling AR1, and for supplying to the adsorbers 1A, 1B, pure oxygen in the operations of filling with pure oxygen AR2 and rinsing out R and pressure reduction E1. The valves KV3A and KV3B serve for carrying away from the adsorbers 1A and 1B the argon fraction through the conduit 103 to the buffer tank 2 in the operations of filling AR1, filling with pure oxygen AR2, rinsing out R and for carrying the argon fraction from the buffer tank 2 to the adsorbers 1A and 1B during the operation of reflux filling RR. The valve KV4B serves for carrying the argon fraction away from the buffer tank 2 through the conduit 104. The valves, respectively KV5A, ZR, ZZB and KV5B, ZR, ZZA serve for performing mutually correlated operations of pressure reduction E1 and pressure elevation 1E. If the valve KV5A is open, oxygen flows from the adsorber 1A through the valve ZR and the valve ZZB to the adsorber 1B and vice versa, when the valve KV5B is open, oxygen flows from the adsorber 1B through the valve ZR and the valve ZZA to the adsorber 1A. The valves KV6A and KV6B serve for carrying desorbing oxygen away from the installation during desorption D. Oxygen is carried away from the installation through the conduit 105 and next compressed and partially recycled to the process.

**[0020]** Fig. 2 presents a diagram of gas streams flow during the cycle of variable pressure adsorption PSA. The symbol 1A and 1B denote the adsorbers, the figure 2 denotes the argon fraction buffer tank. Denotations of the several technological operations are as follows:

- operation of filling - AR1
- operation of filling with pure oxygen - AR2
- operation of rinsing out - R
- operation of pressure reduction - E1
- operation of desorption - D
- operation of pressure elevation - 1E
- operation of reflux filling - RR

**[0021]** Fig. 3 presents the course of pressure P in a single adsorber during the PSA cycle depending upon the time of the cycle t and several technological operations performed in sequence: AR1, AR2, R, E1, D, 1E and RR.

**[0022]** The process of separation of gaseous mixtures of oxygen, argon and nitrogen by the method of variable pressure adsorption according to this invention proceeds as follows:



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- in the operation of filling AR1 - within the time of 30 ÷ 60 s to the adsorber 1A or 1B, by the conduit 101 through the properly set valve KV1AB and the open valve, respectively KV2A or KV2B, there is supplied oxygen polluted with argon and nitrogen, at the same time from the other end of the adsorber through the open valve, respectively KV3A or KV3B, by the conduit 103 there is carried away the argon fraction to the buffer tank 2, in contact with the sorbent, adsorption of oxygen takes place, the gas of the elevated argon content is carried quickly outside the zone of adsorption, due to which argon adsorbs in a reduced degree; the pressure in the adsorber and the buffer tank increases from 300 ÷ 400 kPa to 500 ÷ 700 kPa,
- in the operation of filling with pure oxygen AR2 - within the time of 0 ÷ 15 s to the adsorber 1A or 1B by the conduit 102 through the properly set valve KV1AB and the open valve, respectively KV2A or KV2B, in cocurrent to the gas flow in the operation AR1, there is supplied pure oxygen produced in the operation of desorption D and next compressed, while from the other end of the adsorber 1A or 1B through the open valve, respectively KV3A or KV3B by the conduit 103 there is carried away the argon fraction from the buffer tank 2 and the pressure in the adsorber and in the buffer tank increases from 500 ÷ 700 kPa to 600 ÷ 800 kPa, thereafter adsorbed argon is rinsed out and carried away in the stream of oxygen to the buffer tank 2.
- in the operation of rinsing out R - within the time of 10 ÷ 30 s to the adsorber 1A or 1B by the conduit 102 through the properly set valve KV1AB and the open valve KV2A or KV2B there is supplied pure oxygen produced in the operation of desorption D and next compressed, and from the other end of the adsorber 1A or 1B through the open valve, respectively KV3A or KV3B. by the conduit 103 there is carried away the argon fraction to the buffer tank 2, and at the same time from the buffer tank 2 through the open valve KV4AB by the conduit 104 the argon fraction is carried away outside the installation, the pressure in the adsorber 1A or 1B and in the buffer tank is kept at the stable level 500 ÷ 700 kPa, oxygen being carried away rinses out adsorbed argon and argon being in the free space of the sorbent; argon is carried away in the stream of oxygen in the form of the argon fraction,
- in the operation of pressure reduction E1 within the time of 5 ÷ 20 s to the adsorber 1A or 1B there is supplied pure oxygen through the suitably set valve KV1AB and the open valve, respectively KV2A or KV2B, and from the adsorber 1A or 1B through the open valves, respectively KV5A, ZR, ZZB or KV5B, ZR, ZZA, in cocurrent to the gas flow in the operation AR1 there is carried away desorbing oxygen which rinses out the argon residue and supplied in cocurrent to the gas flow in the operation AR1 to the second adsorber in which there is performed the operation of pressure elevation 1E, the pressure in the adsorber 1A or 1B is reduced from 600 - 800 kPa to 400 - 600 kPa, but the pressure between the joined adsorbers do not equalize, desorbing oxygen with a small content of argon is introduced in total to the second adsorber,
- In the operation of desorption D - within the time 40 ÷ 105 s from the adsorber 1A or 1B through the open valve, respectively KV6A or KV6B, by the conduit 105 there is carried away desorbing pure oxygen, the pressure in the adsorber is reduced to atmospheric and in case of oxygen suction by means of a vacuum pump, it is reduced to 20 ÷ 50 kPa, oxygen is next compressed to the required pressure and it is partially recycled to the process by the conduit 102,
- in the operation of reflux filling RR - within the time 1 ÷ 2 s from the buffer tank 2 through the open valve KV2A or KV2B there is carried away the argon fraction respectively to the adsorber 1A or 1B, the pressure in the adsorber increases to 300 ÷ 400 kPa, the pressures between the adsorbers 1A or 1B and the buffer tank do not equalize.

For appropriate performance of the process of variable pressure adsorption by the method according to the invention it is important to meet several process conditions. The purity of produced oxygen is influenced by:

- the ratio of the volume of the buffer tank 2 to the volume of the molecular carbon or zeolite sieve packed into each of two adsorbers 1A and 1B,
- the ratio of the quantity of pure oxygen recycled to the process in the operations AR2 and R to the content of oxygen contained in the raw material introduced to the installation in the operation AR1,
- the ratio of the increase of pressures in total in the operation AR1 and AR2 to the increase of pressure in the operation AR1,
- the time periods of several technological operations lasting, particularly the ratio of time of AR1 operation lasting to the sum of the time of the operations AR2 and R.

It is favourable for the process of variable pressure adsorption by the method according to the invention to be run after fulfilling the following conditions:

- the radio of the volume of the buffer tank 2 to the volume of the molecular carbon or zeolite sieve packed into each of the adsorbers 1A and 1B should be within the range 0.4 ÷ 0.7, too low ratio of these volumes does no allow to remove the argon fraction effectively from the sorbent zone during the operation AR1, too high ratio causes unfavourable oxygen losses,



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- the ratio of pure oxygen recycled to the process in the operations AR2, R and E1 to the content of oxygen introduced with the raw material should be within the range 0.6 - 1.2, too low content of recycled oxygen does not allow to achieve oxygen of high purity, too high content of oxygen causes losses during the operation of rinsing out without considerable improvement of obtained oxygen purity,
- the ratio of total pressures increase in the operation AR1 and AR2 to the pressure in the operation AR1 should be within 1.0 ÷ 2.0, preferably within the range 1.4 ÷ 1.5, too high ratio of the pressures brings about unnecessary energy consumption connected with compressing recycled oxygen, the higher ratio of these pressures is, the stronger is rinsing out of argon adsorbed in the sorbent,
- the time of the whole cycle of variable pressure adsorption run according to the invention should be within the range 90 ÷ 240 s, too short time of the cycle does not allow to use effectively the adsorption volume of the sorbent towards oxygen because of reduced rate of oxygen diffusion in the sorbent, too long time of cycle lasting causes pollution of the sorbent with argon, thus the produced oxygen purity decreases, it is favourable when the ratio of the time of the operation AR1 to the sum of time periods of the operations AR2 and R is with the range 0.7 ÷ 1.4, longer time of contact between the sorbent and high purity oxygen causes better rinsing of argon out of the sorbent.

**Example 1 - Comparative**

**[0023]** In the installation including two adsorbers 1A and 1B filled with molecular carbon sieve MSC - 3A of Takeda, 100 kg in each adsorber, and a buffer tank of the volume 0.065 $m^3$, there is run separation of the mixture of oxygen, argon and nitrogen of the following contents: $O_2$ content - 95% by volume, Ar content - 4.5% by volume, $N_2$ content - 0.5% by volume. The process is run according to the US patent no. 5.137.549 applying the following parameters of the PSA cycle:

- in the operation of filling the pressure in the adsorber 1A or 1B is elevated from 280 kPa to 550 kPa and polluted oxygen is supplied to the adsorber for the time of 50 s,
- in the operation of adsorption to the adsorber 1A or 1B there is supplied polluted oxygen under the pressure 550 kPa for 35 s and at the other end the argon fraction is received,
- in the operation of cocurrent pressure reduction and rinsing out to the adsorber 1A or 1B for 18 s there is supplied pure oxygen from the operation of desorption, at the same time the pressure is reduced to 450 kPa,
- in the operation of desorption lasting for 85 s from the adsorber 1A or 1B there is carried away pure oxygen which is next compressed and partially recycled to the process,
- in the operation of pressure elevation the pressure is elevated with the oxygen from the operation of cocurrent pressure reduction and rinsing out to the value of 280 kPa.

**[0024]** In the installation there is separated 37.0 $m^3$/hour of polluted oxygen. From the installation there is received 9.2 $m^3$/hour of the argon fraction. In the desorption phase there is received 41.7 $m^3$/hour of pure oxygen. To the process there is recycled 13.9 $m^3$/hour of pure oxygen. In the result from the installation there is obtained 27.8 $m^3$/hour of pure oxygen. Oxygen yield efficiency calculated as the content to the content of oxygen obtained in the product to the content of oxygen introduced with the raw material is 75.0%. In the process there is obtained oxygen of the purity 99.3 ÷ 99.5% by volume measured by means of an analyser with a high-temperature zirconium probe. The values of flow rates correspond to the pressure 101.3 kPa and to the temperature 273°K.

**Example 2**

**[0025]** In the installation of Example 1 there is separated polluted oxygen containing 95% by volume of $O_2$, 4.5% by volume of Ar and 0.5% by volume of $N_2$ according to the invention. The parameters of the process are presented in Table 1.

Table 1.

| Parameters of the PSA process | | | |
|---|---|---|---|
| Operation Symbol | Operation time | Initial pressure | Final pressure |
| | S | kPa | kPa |
| AR1 | 50 | 300 | 550 |
| AR2 | 12 | 550 | 650 |
| R | 25 | 650 | 650 |



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Table 1. (continued)

| Parameters of the PSA process | | | |
|---|---|---|---|
| Operation Symbol | Operation time | Initial pressure | Final pressure |
| E1 | 15 | 650 | 460 |
| D | 87 | 460 | 105 |
| 1E | 15 | 105 | 270 |
| RR | 1 | 270 | 300 |

**[0026]** In the installation there is separated 36.5 $m^3$/hour of polluted oxygen and carried away 9.5 $m^3$/hour of the argon fraction. During desorption from the installation there is received 52.9 $m^3$/hour of pure oxygen. After compression to the installation there is recycled 25.8 $m^3$/hour of pure oxygen, from the installation there is carried away 27.1 $m^3$/hour of pure oxygen. Oxygen yield efficiency is 78%. In the process there is obtained oxygen of the purity 99.8 - 99.9% by volume of $O_2$ measured by means of an analyser with a high-temperature zirconium probe.

**Example 3**

**[0027]** In the installation as in Examples 1 and 2 there is run separation of a mixture consisting of oxygen and argon of the following composition: $O_2$ content - 99.5%, argon content - 4.5%. The same parameters as in Example 2 are used. To the installation there is supplied 36.0 $m^3$/hour of polluted oxygen and received 9.5 $m^3$/hour of the argon fraction. During desorption from the installation there is received 53.3 $m^3$/hour of pure oxygen. To the process there is recycled 26.8 $m^3$/hour of pure oxygen and eventually from the installation there is obtained 26.5 $m^3$/hour of pure oxygen. The oxygen yield efficiency is 77.9%. In the process there is obtained oxygen of the purity 99.9 - 100% by volume measured by means of a high-temperature zirconium probe.

**Claims**

1. Method for separation of gas mixtures of oxygen, argon and nitrogen by a method of variable-pressure adsorption in kineticly selective PSA process on molecular carbon or zeolite sieves wherein the PSA process is run in an installation including at least two adsorbers (1A) and (1B) and a buffer tank of the argon fraction (2) and the full PSA cycle comprises the following technological operations proceeding in sequence:

   - operation of filling (AR1) in which to the adsorber (1A) or (1B) within the time of 30 ÷ 60s there is supplied polluted oxygen and at the same time from the adsorber (1A) or (1B) there is carried away the argon fraction from the buffer tank, while the pressure in the adsorber and the buffer tank increases from 300 ÷ 400 kPa to 500 ÷ 700 kPa.

   - operation of filling with pure oxygen (AR2) in which to the adsorber within the time 0 ÷ 15 s in cocurrent to the gas flow in the operation (AR) theres is supplied pure oxygen produced in the operation of desorption and next compressed, while at the same time from the adsorber (1A) or (1B) to the buffer tank (2) there is supplied argon fraction, while the pressure in the adsorber (1A) or (1B) increases from 500 ÷ 700 kPa to 600 ÷ 800 kPa,

   - operation of rinsing out (R) in which to the adsorber (1A) or (1B) within the time 10 ÷ 30 s, in cocurrent to the gas flow of the operation (AR1), there is supplied pure oxygen produced in the operation of desorption and next compressed, while at the same time from the adsorber (1A) or (1B) to the buffer tank (2) there is carried away argon fraction and at the same time from the argon fraction is carried away from the buffer tank (2), while the pressure is kept on a stable level of 600 ÷ 800 kPA,

   - operation of pressure reduction (E1) in which to the adsorber (1A) or (1B) there is supplied pure oxygen and at the same time from the adsorber (1A) or (1B) within the time of 5 ÷ 20 s, in cocurrent to the gas flow in the operation (AR1) there is carried away gas and supplied in cocurrent to the second adsorber, respectively (1B) or (1A), in which the operation of pressure elevation (1E) is run, while the pressure is reduced from 600 ÷ 800 kPa to 400 ÷ 600 kPa, but the pressures between the adsorbers do not equalize,



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- operation of desorption (D) in which from the adsorber within the time of 40 ÷ 105 s, in counter-current to the gas flow in the operation (AR1) there is carried away desorbing pure oxygen and the pressure in the adsorber is reduced to the pressure 100 ÷ 110 kPa, and in case of oxygen suction by means of a vacuum pump, the pressure is reduced to 10 ÷ 50 kPa,

- operation of pressure elevation (1E) in which to the adsorber (1A) or (1B) within the time of 5 ÷ 20 s, in cocurrent to the gas flow in the operation (AR1) there is supplied gas produced in the operation of pressure reduction (E1), and the pressure increases from 250 ÷ 350 kPa but the pressures between the adsorbers do not equalize,

- operation of reflux filling (RR) in which from the adsorber (1A) or (1B) within the time 1 ÷ 2 s, in counter-current to the gas flow in the operation (AR1) there is supplied argon fraction contained in the buffer tank (2) but the pressures between the adsorbers (1A) or (1B) and the buffer tank (2) do not equalize.

2. Method according to Claim 1 wherein the ratio of the volume of the buffer tank (2) to the volume of the molecular carbon or zeolite sieve contained in the adsorber (1A) or (1B) is within the range 0.4 ÷ 0.7.

3. Method according to Claim 1 wherein the ratio of pure oxygen recycled to the process in the operations (AR1) and (R), and (E1) to the content of oxygen introduced with the raw material is with the range 0.6 ÷ 1.2.

4. Method according to Claim 1 wherein the ratio of the pressure increase in total in the operations (AR1) and (AR2) to the pressure increase only in the operation (AR1) is with the range 1.0 ÷ 2.0.

5. Method according to Claim 1 wherein the lasting time of the full PSA cycle is 90 ÷ 240 s, while the ratio of the lasting time of the operation (AR1) to the sum of the lasting time of the operation (AR1) and (R) is 0.7 ÷ 1.4.



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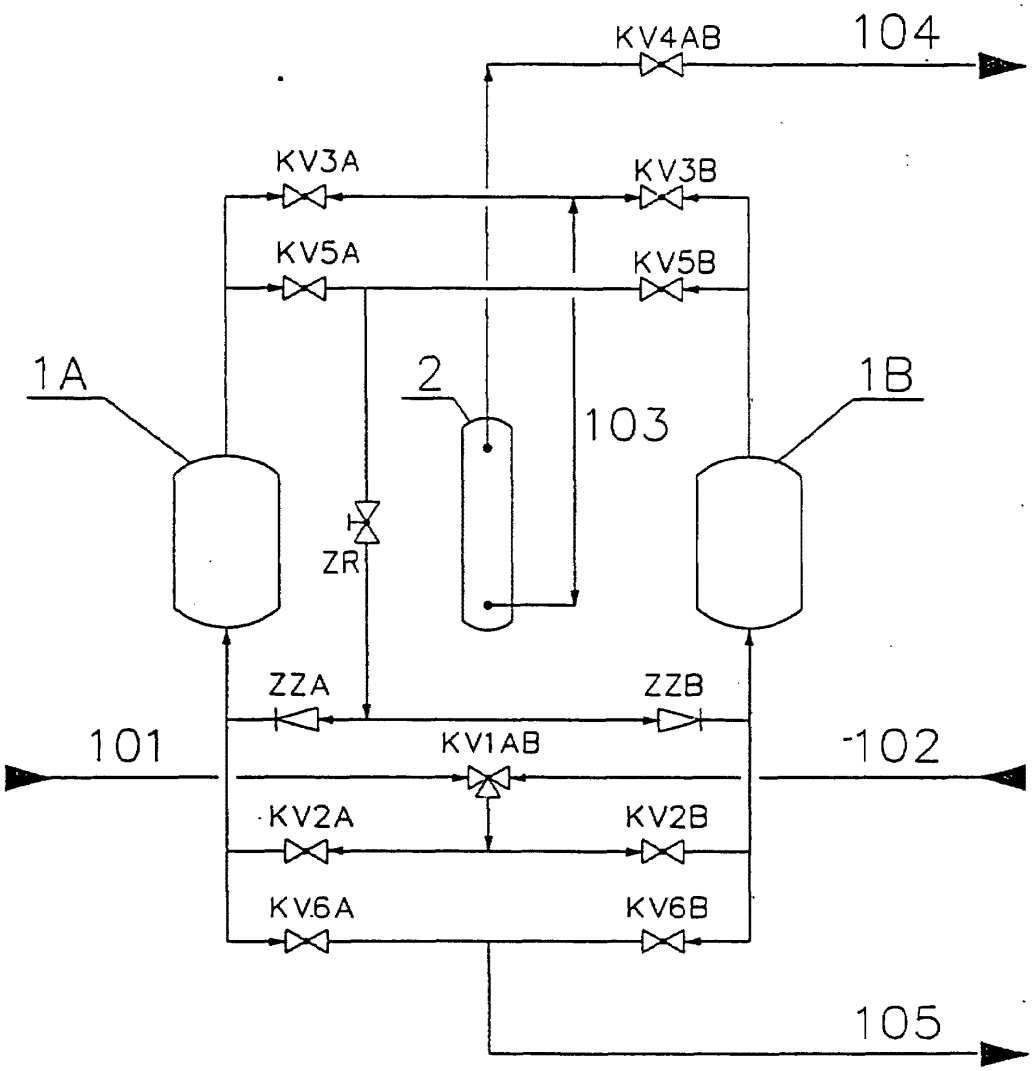



Fig. 1



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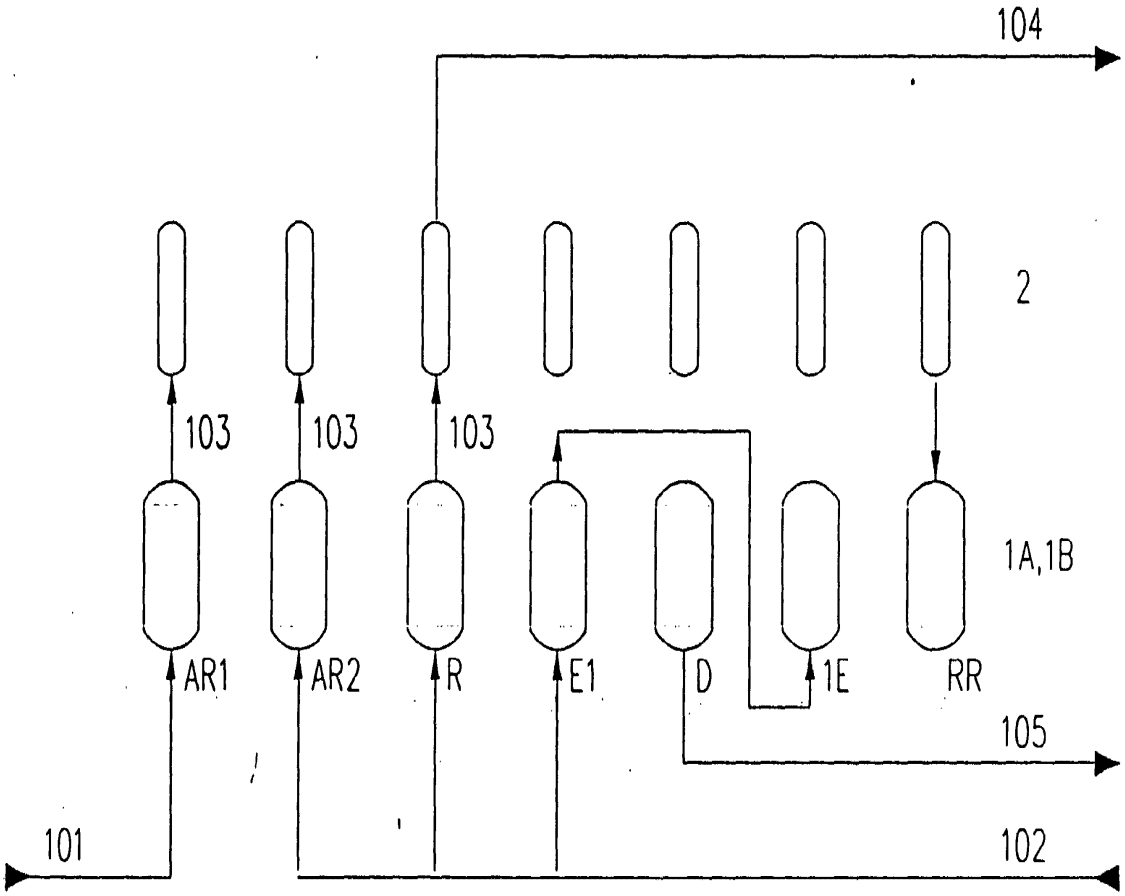



Fig. 2



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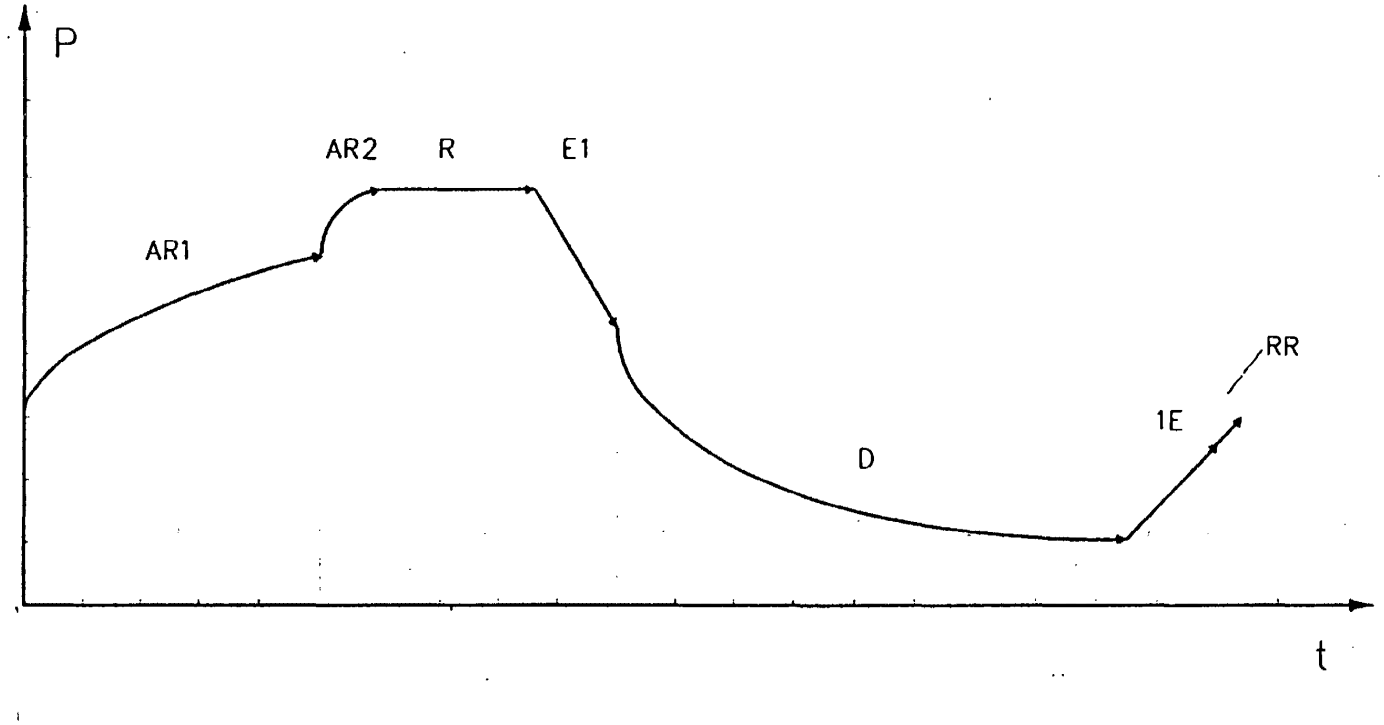



Fig. 3



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# EUROPEAN SEARCH REPORT

Application Number
EP 00 11 9469

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B01D
C01B
F25J

The present search report has been drawn up for all claims

| Place of search | Date of completion of the search | Examiner |
|---|---|---|
| BERLIN | 20 June 2001 | Cubas Alcaraz, J |

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X : particularly relevant if taken alone
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## ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 00 11 9469

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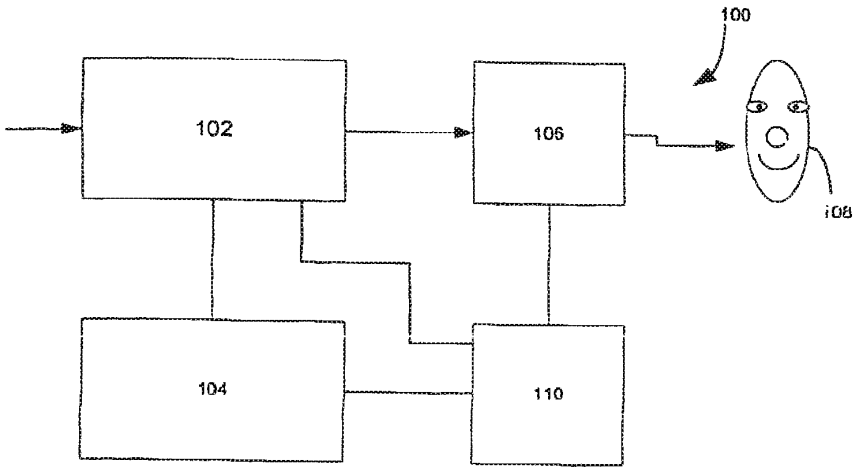
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(54) **Title:** PORTABLE OXYGEN CONCENTRATION SYSTEM




(57) **Abstract:** A portable oxygen concentrator system (100) adapted to be readily transported by a user includes a rechargeable energy source (104) and a concentrator (114) powered by the energy source (104). The concentrator (114) converts ambient air into concentrated oxygen gas for the user and includes a plurality of adsorption beds (300) and a rotary valve assembly (310). The rotary valve assembly (310) is relatively rotatable with respect to the plurality of adsorption beds (300) to provide valving action for selectively transferring fluids through the plurality of absorption beds (300) for converting ambient air into concentrated oxygen gas for the user. The ratio of adiabatic power to oxygen flow for the concentrator (114) is in the range of 6.2 W/LPM to 23.0 W/LPM.

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*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

# DESCRIPTION

# PORTABLE OXYGEN CONCENTRATION SYSTEM

## Background of the Invention

**[0001]** The field of this invention relates, in general, to oxygen concentrators and, in particular, to portable oxygen concentration systems for ambulatory respiratory patients that allow them to lead normal and productive lives.

**[0002]** There is a burgeoning need for home and ambulatory oxygen. Supplemental oxygen is necessary for patients suffering from lung disorders; for example, pulmonary fibrosis, sarcoidosis, or occupational lung disease. For such patients, oxygen therapy is an increasingly beneficial, life-giving development. While not a cure for lung disease, supplemental oxygen increases blood oxygenation, which reverses hypoxemia. This therapy prevents long-term effects of oxygen deficiency on organ systems -- in particular, the heart, brain and kidneys.

**[0003]** Oxygen treatment is also prescribed for Chronic Obstructive Pulmonary Disease (COPD), which afflicts about six-hundred million people in the U.S., and for other ailments that weaken the respiratory system, such as heart disease and AIDS. Supplemental oxygen therapy is also prescribed for asthma and emphysema.

**[0004]** The normal prescription for COPD patients requires supplemental oxygen flow via nasal cannula or mask twenty four hours per day. The average patient prescription is two liters per minute of high concentration oxygen to increase the oxygen level of the total air inspired by the patient from the normal 21% to about 40%. While the average oxygen flow requirement is two liters per minute, the average oxygen concentrator has a capacity of four to six liters of oxygen per minute. This extra capacity is occasionally necessary for certain patients who have developed more severe problems, are not generally able to leave the home (as ambulatory patients) and do not require a portable oxygen supply.

**[0005]** There are currently three modalities for supplemental medical oxygen: high pressure gas cylinders, cryogenic liquid in vacuum insulated containers or thermos bottles commonly called "dewars," and oxygen concentrators. Some patients require in-home oxygen only while others require in-home as well as ambulatory oxygen depending on their prescription. All three modalities are used for in-home use, although oxygen concentrators are preferred because they do not require dewar refilling or exchange

of empty cylinders with full ones. Home oxygen concentrators, however, do have their drawbacks. They consume relatively large amounts of electricity (350-400 Watts), are relatively large (about the size of a night stand), are relatively heavy (weight about 50 lbs.), emit quite a bit of heat, and are relatively noisy.

**[0006]** Only small high pressure gas bottles and small liquid dewars are truly portable enough to be used for ambulatory needs (outside the home). Either modality may be used for both in-home and ambulatory use or may be combined with an oxygen concentrator which would provide in-home use.

**[0007]** As described below, the current oxygen-supplying methods and devices have proven cumbersome and unwieldy and there has been a long-felt need for an improved portable device for supplying oxygen to the user.

**[0008]** For people who need to have oxygen and operate away from an oxygen-generating or oxygen-storage source such as a stationary oxygen system (or even a portable system which cannot be readily transported), the two most prescribed options generally available to patients are: (a) to carry with them small cylinders typically in a wheeled stroller; and (b) to carry portable containers typically on a shoulder sling. Both these gaseous oxygen and liquid oxygen options have substantial drawbacks, but from a medical view, both have the ability to increase the productive life of a patient.

**[0009]** The major drawback of the gaseous oxygen option is that the small cylinders of gaseous oxygen can only provide gas for a short duration. Another drawback is that a patient's high-pressure gaseous oxygen cylinders are not allowed in some locations such as airplanes because of safety considerations. A further drawback of the gaseous oxygen option is the refill requirement for oxygen once the oxygen has been depleted from the cylinder. These small gas cylinders must be picked up and refilled by the home care provider at a specialized facility. This requires regular visits to a patient's home by a provider and a substantial investment in small cylinders by the provider because so many are left at the patient's home and refilling facility. Although it is technically possible to refill these cylinders in the patient's home using a commercial oxygen concentrator that extracts oxygen from the air, this task would typically require an on-site oxygen compressor to boost the output pressure of the concentrator to a high level in order to fill the cylinders. Some disadvantages of common on-site oxygen compressors are that they are expensive, loud and emit a lot of heat. Additionally, attempting to



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compress the oxygen in pressurized canisters in the home is potentially dangerous, especially for untrained people.

**[0010]** This approach of course presents several safety concerns for in-home use. For example, in order to put enough of this gas in a portable container, it must typically be compressed to high pressure (~2000 psi) Compressing oxygen from 5 psi (the typical output of an oxygen concentrator) to 2000 psi will produce a large amount of heat. (Enough to raise the temperature 165 degrees C per stage based on three adiabatic compression stages with intercooling.) This heat, combined with the oxygen which becomes more reactive at higher pressures, sets up a potential combustion hazard in the compressor in the patient's home. Thus, operation of a high-pressure gas system in the patient's home is dangerous and not a practical solution.

**[0011]** The convenience and safety issues are not the only drawbacks of this compressed oxygen approach. Another drawback is that the compressors or pressure boosters needed are costly because they require special care and materials needed for high pressure oxygen compatibility.

**[0012]** Turning now to the liquid oxygen storage option, its main drawback is that it requires a base reservoir -- a stationary reservoir base unit within the patient's home about the size of a standard beer keg -- which may be refilled about once a week from an outside source. Liquid oxygen can then be transferred from the patient's base unit to a portable dewar, which can be used by the ambulatory patient. Also, with the liquid oxygen option, there is substantial waste, as a certain amount of oxygen is lost during the transfer to the portable containers and from evaporation. It is estimated that 20% of the entire contents of the base cylinder will be lost in the course of two weeks because of losses in transfer and normal evaporation. These units will typically boil dry over a period of 30 to 60 days even if no oxygen is withdrawn.

**[0013]** Home refilling systems that produce liquid oxygen and have the capability of refilling portable liquid oxygen dewars have been proposed. However, these devices require the user to perform the task of refilling bottles and add tens of dollars per month to the user's electric bill, which is not reimbursable.

**[0014]** There are other complications with these portable high-pressure cylinders and liquid dewars. Typically, supplemental oxygen is supplied to the patient by a home care provider, in exchange for which the provider receives a fixed monetary payment from



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insurance companies or Medicare regardless of the modality. Oxygen concentrators are preferred by the provider as the least expensive option for supplying the patient's at-home needs. For outside the home use, however, only small high-pressure gas bottles and small liquid dewars are portable enough to be used for ambulatory needs. Either one of these two modalities may be used for both in-home and ambulatory use or may be combined with an oxygen concentrator, which would provide in-home use. In either case, the home care provider must make costly weekly or biweekly trips to the patient's home to replenish the oxygen. One of the objects of this invention is to eliminate these costly milk runs."

**[0015]** So-called "portable" oxygen concentrators are commercially available for providing patients with gaseous oxygen by converting ambient air into concentrated gaseous oxygen. However, these devices are only "portable" in the sense that they are capable of being transported to another point of use via an automobile or an airplane. One of these devices is packaged in a suitcase and is billed as a transportable machine rather than a truly portable oxygen concentrator. The device weighs about 37 lbs. without battery and requires 135 Watts of power at a 2 LPM (liters per minute) oxygen flow rate. Operation from an automobile battery is possible when in route in a car, but operation from a separate battery is impractical. Another device is a 3 LPM concentrator mounted on its own cart. It weighs 22 lbs. without battery and also requires about 135 Watts of power. A further device weighs about 28 lbs. without battery and has a similar flow rate and power requirements to the above devices. Even without a battery, these devices are too heavy for the average ambulatory respiratory patient. With the weight of a battery, these prior art devices are not "portable" in the true sense of the word because they can not be readily transported from one point to another. Because these devices have relatively large power consumption requirements, they also require a sizable battery.

**[0016]** Further, in addition to the weight and power consumption problems with the above oxygen concentrators, none of these prior art concentrators are particularly quiet. They produce noise levels similar to those produced by a home concentrator. In fact, one of these devices specifie's noise production at 60 dBA (decibels), about twice the noise of a home concentrator. Consequently, none of these so-called "portable" oxygen concentrators are suitable for use in environments where low noise is especially important, e.g., restaurants, libraries, churches and theatres.



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**[0017]** Thus, a long-felt need exists for a truly "portable" oxygen concentration system that eliminates the need for high-pressure gas cylinders and liquid dewars, the constant refilling/replacing requirements associated with high-pressure gas cylinders and liquid dewars, and the need for a separate home oxygen concentration system for ambulatory respiratory patients. A truly "portable" oxygen concentration system would be light enough so that, even with a battery, an average ambulatory respiratory patient could carry the device. Inherently the device would have to be designed to have relatively low power consumption requirements so that a light-weight battery pack or other energy source could be used. Further, the device should be small enough so that it can be conveniently carried by the user, emit a relatively low amount of noise and should only emit a small amount of heat.

Summary of Invention

**[0018]** An aspect of the present invention involves a portable oxygen concentrator system adapted to be readily transported by a user. The portable oxygen concentrator system includes a rechargeable energy source and a concentrator powered by the energy source. The concentrator converts ambient air into concentrated oxygen gas for the user and includes a plurality of adsorption beds and a rotary valve assembly. The rotary valve assembly is relatively rotatable with respect to the plurality of adsorption beds to provide valving action for selectively transferring fluids through the plurality of adsorption beds for converting ambient air into concentrated oxygen gas for the user. The ratio of adiabatic power to oxygen flow for the concentrator is in the range of 6.2 W/LPM to 23.0 W/LPM.

**[0019]** Another aspect of the invention involves a rotary valve assembly for a pressure swing adsorption system having a plurality of adsorption beds. The rotary valve assembly includes a valve port plate and a rotary valve shoe having respective engaged surfaces and are relatively rotatable about a common center of rotation to provide valving action for selectively transferring fluids therethrough. The valve port plate includes at least two ports interconnected with at least two adsorption beds. The rotary valve shoe includes a second valve surface opposite the engaged surface with at least one equalization passage to register with the at least two ports of the port plate to equalize pressure between the at least two adsorption beds.



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**[0020]** Other and further objects, features, aspects, and advantages of the present inventions will become better understood with the following detailed description of the accompanying drawings.

Brief Description of the Drawings

**[0021]** Figure 1 is a block diagram of a portable oxygen concentration system constructed in accordance with an embodiment of the invention;

**[0022]** Figure 2 is a block diagram of a portable oxygen concentration system constructed in accordance with another embodiment of the invention, and illustrates, in particular, an embodiment of an air separation device;

**[0023]** Figure 3A is a perspective, cut-away view of an embodiment of a concentrator that may be used with the portable oxygen concentration system.

**[0024]** Figure 3B is a perspective, exploded view of the concentrator illustrated in Figure 3A.

**[0025]** Figure 4 is a top perspective view of an embodiment of a top manifold and multiple adsorption beds that may be used with the concentrator illustrated in Figures 3A and 3B.

**[0026]** Figures 5A and 5B are a bottom plan view and a top plan view respectively of an embodiment of a rotary valve shoe that may be used with the concentrator illustrated in Figures 3A and 3B.

**[0027]** Figure 6A is a top plan view of an embodiment of a valve port plate that may be used with the concentrator illustrated in Figures 3A and 3B.

**[0028]** Figure 6B is a flow chart of an exemplary process cycle for the concentraotor illustrated in Figures 3A and 3B.

**[0029]** Figures 7A and 7B are a top plan view and a bottom plan view respectively of an embodiment of a media retention cap that may be used with the concentrator illustrated in Figures 3A and 3B.

**[0030]** Figures 8A and 8B are a top perspective, exploded view and a bottom perspective, exploded view respectively of an embodiment of a rotary valve assembly including a centering pin that may be used with the concentrator illustrated in Figures 3A and 3B.

**[0031]** Figures 9A and 9B are a bottom perspective, exploded view and a top perspective, exploded view respectively of an embodiment of a rotary valve assembly



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including a centering ring that may be used with the concentrator illustrated in Figures 3A and 3B.

**[0032]** Figure 10A is a bottom perspective view of an embodiment of a rotary valve shoe, a motor drive, and a pair of elastic chain links that may be used with the concentrator illustrated in Figures 3A and 3B.

**[0033]** Figures 10B and 10C are a top perspective, exploded view and a bottom perspective, exploded view respectively of the rotary valve shoe, motor drive, and pair of elastic chain links illustrated in Figure 10A.

**[0034]** Figure 11 is a table of experimental data for a portable oxygen concentration system including the concentrator illustrated in Figures 3A and 3B.

**[0035]** Figure 12 is a schematic illustration of a further embodiment of the portable oxygen concentration system and an embodiment of a cradle for use with the portable oxygen concentration system;

**[0036]** Figure 13 is a block diagram of the one or more sensors that may be used with an embodiment of the portable oxygen concentration system;

**[0037]** Figure 14 is a block diagram of the one or more components that may be controlled by the control unit of the portable oxygen concentration system;

**[0038]** Figure 15 is a block diagram of a portable oxygen concentration system constructed in accordance with additional embodiment of the invention; and

**[0039]** Figure 16 is a schematic illustration of another embodiment of a portable oxygen concentration system including a high-pressure reservoir.

Detailed Description of the Preferred Embodiment

I. Portable Oxygen Concentration System

**[0040]** With reference to Figure 1, a portable oxygen concentration system, indicated generally by the reference numeral 100, constructed in accordance with an embodiment of the invention will now be described. The oxygen concentration system 100 includes an air separation device such as an oxygen gas generator 102 that separates concentrated oxygen gas from ambient air, an energy source such as rechargeable battery, battery pack, or fuel cell 104 that powers at least a portion of the oxygen gas generator 102, one or more output sensors 106 used to sense one or more conditions of the user 108, environment, etc. to determine the oxygen output needed by the user or required from the system 100, and a control unit 110 linked to the output sensor 106, the air separation



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device 102, and the energy source 104 to control the operation of the air separation device 102 in response to the one or more conditions sensed by the one or more output sensors 106.

**[0041]** In an alternative embodiment, the system 100 may not include the one or more output sensors 106 coupled to the control unit 110. In this embodiment, conditions of the system 100 such as flow rate, oxygen concentration level, etc. may be constant for the system or may be manually controllable. For example, the system 100 may include a user interface 111 (Figure 14) that allows the user, provider, doctor, etc. to enter information, e.g., prescription oxygen level, flow rate, etc. to control the oxygen output of the system 100.

**[0042]** Each element of the system 100 will now be described in more detail.

A. Air Separation Device

**[0043]** With reference to Figure 2, the air separation device is preferably an oxygen generator 102 generally including a pump such as a compressor 112 and an oxygen concentrator 114 (OC), which may be integrated.

**[0044]** The oxygen generator 102 may also include one or more of the elements described below and shown within the segmented boundary line in Figure 2. Ambient air may be drawn through an inlet muffler 116 by the compressor 112. The compressor 112 may be driven by one or more DC motors 118 (M) that run off of DC electrical current supplied by the rechargeable battery 104 (RB). The motor 118 also preferably drives the cooling fan part of the heat exchanger 120. A variable-speed controller (VSC) or compressor motor speed controller 119, which is described in more detail below, may be integral with or separate from the control unit 110 (CU) and is preferably coupled to the motor 118 for conserving electricity consumption. The compressor 112 delivers the air under pressure to the concentrator 114.

**[0045]** In a preferred embodiment, at a maximum speed air is delivered to the concentrator 114 at 7.3 psig nominal and may range from 5.3 to 12.1 psig. At maximum speed, the flow rate of feed is a minimum of 23.8 SLPM at inlet conditions of 14.696 psi absolute, 70 degrees F, 50% relative humidity.

**[0046]** A heat exchanger 120 may be located between the compressor 112 and the concentrator 114 to cool or heat the air to a desired temperature before entering the concentrator 114, a filter (not shown) may be located between the compressor 112 and the



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concentrator 114 to remove any impurities from the supply air, and a pressure transducer 122 may be located between the compressor 112 and the, concentrator 114 to get a pressure reading of the air flow entering the concentrator 114.

**[0047]** The concentrator 114 separates oxygen gas from air for eventual delivery to the user 108 in a well-known manner. One or more of the following components may be located in a supply line 121 between the concentrator 114 and the user 108: a pressure sensor 123, a temperature sensor 125, a pump 127, a low-pressure reservoir 129, a supply valve 160, a flow and purity sensor 131, and a conservation device 190. As used herein, supply line 121 refers to the tubing, connectors, etc. used to connect the components in the line. The pump 127 may be driven by the motor 118. The oxygen (gas may be stored in the low-pressure reservoir 129 and delivered therefrom via the supply line 121 to the user 108. The supply valve 160 may be used to control the delivery of oxygen gas from the low-pressure reservoir 129 to the user 108 at atmospheric pressure.

**[0048]** Exhaust gas may also be dispelled from the concentrator 114. In a preferred embodiment of the invention, a vacuum generator 124 (V), which may also be driven by the motor 118 and integrated with the compressor 112, draws exhaust gas from the concentrator 114 to improve the recovery and productivity of the concentrator 114. The exhaust gas may exit the system 100 through an exhaust muffler 126. A pressure transducer 128 may be located between the concentrator 114 and the vacuum generator 124 to get a pressure reading of the exhaust flow from the concentrator 114. At maximum rated speed and a flow rate of 20.8 SLPM, the pressure at the vacuum side is preferably -5.9 psig nominal and may range from -8.8 to -4.4 psig.

1. Compressor/Variable Speed Controller

**[0049]** Example of compressor technologies that may be used for the compressor 112 include, but not by way of limitation, rotary vane, linear piston with wrist pin, linear piston without wrist pin, nutating disc, scroll, rolling piston, diaphragm pumps, and acoustic. Preferably the compressor 112 and vacuum generator 124 are integrated with the motor 118 and are oil-less, preventing the possibility of oil or grease from entering the air flow path.

**[0050]** The compressor 112 preferably includes, at a minimum, a 3:1 speed ratio, with a low speed of at least 1,000 rpm and a 15,000 hour operating life when run at full speed. Operating temperature surrounding the compressor/motor system is preferably



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32 to 122 degrees F. Storage temperature is preferably -4 to 140 degree F. Relative humidity is preferably 5 to 95% RH noncondensing. Voltage for the compressor 112 is preferably 12 V DC or 24V DC and the electrical power requirements are preferably less than 100 W at full speed and rated flow/nominal pressure and less than 40 W at 1/3 speed and 1/3 flow at rated pressure. A shaft mounted fan or blower may be incorporated with the compressor 112 for compressor cooling and possible complete system cooling. Preferably, the maximum sound pressure level of the compressor 112 may be 46 dBA at a maximum rated speed and flow/pressure and 36 dBA at 1/3 rated speed. Preferably the compressor 112 weighs less than 3.5 pounds.

**[0051]** It is desirable for the compressor 112 to run at a variety of speeds; provide the required vacuum/pressure levels and flow rates, emit little noise and vibration, emit little heat, be small, not be heavy, and consume little power.

**[0052]** The variable-speed controller 119 is important for reducing the power consumption requirements of the compressor 112 on the rechargeable battery 104 or other energy source. With a variable-speed controller, the speed of the compressor 112 may be varied with the activity level of the user, metabolic condition of the user, environmental condition, or other condition indicative of the oxygen needs of the user as determined through the one or more output sensors 106.

**[0053]** For example, the variable-speed controller may decrease the speed of the motor 118 when it is determined that the oxygen requirements of the user 108 are relatively low, e.g., when the user is sitting, sleeping, at lower elevations, etc., and increased when it is determined that the oxygen requirements of the user 108 are relatively high or higher, e.g., when the user stands, when the user is active, when the user is at higher elevations, etc. This helps to conserve the life of the battery 104, reduce the weight and size of the battery 104, and reduce the compressor wear rate, improving its reliability.

**[0054]** One of the inventors of the present invention was a co-inventor on a variable-speed controller in the past that regulated the compressor speed to operate the compressor only at the speed and power needed to deliver oxygen at the user's prescribed flow rate.

**[0055]** The variable-speed controller 119 allows the compressor 112 to operate at a low average rate, typically the average rate or speed will be between full speed and 1/6



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full speed of the compressor 112, resulting in an increase in battery life, decrease in battery size and weight, and decrease in compressor noise and emitted heat.

2. Concentrator

**[0056]** In a preferred embodiment, the concentrator 114 is an Advanced Technology Fractionator (ATF) that may be used for medical and industrial applications. The ATF may implement a pressure swing adsorption (PSA) process, a vacuum pressure swing adsorption (VPSA) process, a rapid PSA process, a very rapid PSA process or other process. If a PSA process is implemented, the concentrator preferably includes a rotary valve assembly to control air flow through multiple sieve beds. In an alternative, less preferred embodiment, the concentrator may include a non-rotating valve mechanism to control air flow through multiple sieve beds. The sieve beds may be tapered so that they have larger diameter where gaseous flow enters the beds and a smaller diameter where gaseous flow exits the beds. Tapering the sieve beds in this manner requires less sieve material and less flow to obtain the same output.

**[0057]** Although an ATF concentrator 114 is used in a preferred embodiment, it will be readily apparent to those skilled in the art that other types of concentrators or air-separation devices may be used such as, but not by way of limitation, membrane separation types and electrochemical cells (hot or cold). If other types of concentrators or air-separation devices are used, it will be readily apparent to those skilled in the art that some aspects described herein may change accordingly. For example, if the air-separation device is a membrane separation type, pumps other than a compressor may be used to move air through the system.

**[0058]** The ATF preferably used is significantly smaller that ATFs designed in the past. The inventors of the present invention recognized that reducing the size of the ATF concentrator 114 not only made the system 100 smaller and more portable, it also improved the recovery percentage, i.e., the percentage of oxygen gas in air that is recovered or produced by the concentrator 114 and the productivity (liters per minute/lb. of sieve material) of the concentrator 114. Reducing the size of the ATF decreases the cycle time for the device. As a result, productivity is increased.

**[0059]** Further, the inventors also determined that finer sieve materials increased recovery rates and productivity. The time constant to adsorb unwanted gases is smaller for finer particles because the fluid path is shorter for the gases than for larger particles. Thus,



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fine sieve materials having small time constants are preferred. The sieve material may be a LithiumX Zeolite that allows for a high exchange of Lithium ions. The bead size may, for example, be 0.2-0.6 mm. In an alternative embodiment, the Zeolite may be in the form of a rigid structure such as an extruded monolith or in the form of rolled up paper. In this embodiment, the Zeolite structure would allow for rapid pressure cycling of the material without introducing significant pressure drop between the feed and product streams.

**[0060]** The size of the concentrator 114 may vary with the flow rate desired. For example, the concentrator 114 may come in a 1.5 Liter per minute (LPM) size, a 2 LPM size, a 2.5 LPM size, a 3 LPM size, etc.

**[0061]** The oxygen gas generator 102 may also include an oxygen source in addition to the concentrator 114 such as, but not by way of limitation, a high-pressure oxygen reservoir, as described in more detail below.

**[0062]** An ATF valve controller 133 may be integral with or separate from the control unit 110 and is coupled with valve electronics in the concentrator 114 for controlling the valve(s) of the concentrator 114.

**[0063]** The concentrator may have one or more of the following energy saving modes: a sleep mode, a conserving mode, and an active mode. Selection of these modes may be done manually by the user 108 or automatically such as through the described one or more sensors 106 and control unit 110.

**[0064]** With reference to Figures 3A and 3B, an embodiment of a concentrator 114 that may be used in the oxygen generator 102 will now be described in more detail. Although the concentrator 114 will be described as separating oxygen from air, it should be noted that the concentrator 114 may be used for other applications such as, but not by way of limitation, air separations for the production of nitrogen, hydrogen purification, water removal from air, and argon concentration from air. As used herein, the term "fluids" includes both gases and liquids.

**[0065]** The concentrator 114 described below includes numerous improvements over previous concentrators that result in increased recovery of the desired component and increased system productivity. Improved recovery is important since it is a measure of the efficiency of the concentrator. As a concentrator's recovery increases, the amount of feed gas required to produce a given amount of product decreases. Thus, a concentrator with higher recovery may require a smaller feed compressor (e.g., for oxygen concentration



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from air) or may be able to more effectively utilize feed gas to recover valuable species (e.g., for hydrogen purification from a reformate stream). Improved productivity is important since an increase in productivity relates directly to the size of the concentrator. Productivity is measured in units of product flow per mass or volume of the concentrator. Thus, a concentrator with higher productivity will be smaller and weigh less than a concentrator that is less productive, resulting in a more attractive product for many applications. Therefore, concentrator improvements in recovery, productivity, or both are advantageous. The specific improvements that lead to improved recovery and productivity are detailed below.

**[0066]** The concentrator 114 includes five adsorption beds 300, each containing a bed of adsorbent material which is selective for a particular molecular species of fluid or contaminant, a rotary valve assembly 310 for selectively transferring fluids through the adsorption beds 300, an integrated tube-assembly and mainifold "manifold" 320, a product tank cover 330, and a valve assembly enclosure 340.

**[0067]** The adsorption beds 300 are preferably straight, elongated, molded, plastic vessels surrounded by the product tank cover 330, which is made of metal, preferably aluminum. The molded, plastic adsorption beds 300 surrounded by the metal cover 330 make for a low-cost design without the detrimental effects of water influx that occur with prior-art plastic housings or covers. Plastic adsorption beds have the inherent problem of the plastic being permeable to water. This allows water to penetrate into the adsorbent material, decreasing the performance of the adsorbent material. Surrounding the plastic adsorption beds 300 with the aluminum cover 330, which also may serve as a product accumulation tank, maintains the low cost of the design and does not sacrifice performance.

**[0068]** Each adsorption bed 300 includes a product end 350 and a feed end 360. With reference additionally to Figure 4, the product ends 350 of the beds 300 communicate with incoming product passages 370 of the manifold 320 through product lines 380 for communication with the rotary valve assembly 310. The feed ends 360 of the beds 300 communicate with outgoing feed passages 390 of the manifold 320 for communication with the rotary valve assembly 310.

**[0069]** The manifold 320 may also include outgoing product passages 400 that communicate the rotary valve assembly 310 with the interior of the product tank 330, an



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incoming feed passage 410 that communicates the rotary valve assembly 310 with a feed pressure line 420, and a vacuum chamber 430 that communicates the rotary valve assembly 310 with a vacuum pressure line 440. A product delivery line 450, which may be the same as the supply line 121 described above with respect to Figure 2, communicates with the interior of the product tank 330. The vacuum pressure line 440 may communicate directly or indirectly with the vacuum generator 124 for drawing exhaust gas from the concentrator 114.

**[0070]** In use, air flows from the compressor 112 to the feed pressure line 420, through the incoming feed passage 410 of the manifold 320. From there, air flows to the rotary valve assembly 310 where it is distributed back through outgoing feed passages 390 of the manifold 320. From there, the feed air flows to the feed ends 360 of the adsorption beds 300. The adsorption beds 300 include adsorbent media that is appropriate for the species that will be adsorbed. For oxygen concentration, it is desirable to have a packed particulate adsorbent material that preferentially adsorbs nitrogen relative to oxygen in the feed air so that oxygen is produced as the non-adsorbed product gas. An adsorbent such as a highly Lithium exchanged X-type Zeolite may be used. A layered adsorbent bed that contains two or more distinct adsorbent materials may also be used. As an example, for oxygen concentration, a layer of activated alumina or silica gel used for water adsorption may be placed near the feed end 360 of the adsorbent beds 300 with a lithium exchanged X-type zeolite used as the majority of the bed toward the product end 350 to adsorb nitrogen. The combination of materials, used correctly, may be more effective than a single type of adsorbent. In an alternative embodiment, the adsorbent may be a structured material and may incorporate both the water adsorbing and nitrogen adsorbing materials.

**[0071]** The resulting product oxygen gas flows towards the products ends 350 of the adsorption beds 300, through the product lines 380, through incoming product passages 370 of the manifold 320, and to the rotary valve assembly 310, where it is distributed back through the manifold 320 via the outgoing product passage 400 and into the product tank 330. From the product tank 330, oxygen gas is supplied to the user 108 through the product delivery line 450 and the supply line 121.

**[0072]** With reference to Figures 3B, 5A, 5B, 6A, 8A, and 8B, an embodiment of the rotary valve assembly 310 will now be described. The rotary valve assembly 310 includes a rotary valve shoe or disk 500 and a valve port plate or disk 510. The rotary



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valve shoe 500 and valve port plate 510 are both preferably circular in construction and made from a durable material such as ceramic, which can be ground to a highly polished flat finish to enable the faces of the valve shoe 500 and port plate 510 to form a fluid-tight seal when pressed together.

**[0073]** With reference specifically to FIG. 5A, the rotary valve shoe 500 has a flat, bottom engagement surface 520 and a smooth cylindrical sidewall 530. The valve shoe 500 has several symmetrical arcuate passages or channels cut into the engagement surface 520, all of which have as their center the geometric center of the circular engagement surface 520. The passages or channels include opposite high-pressure feed channels 540, equalization channels 550, opposite low-pressure exhaust passages 560, circular low-pressure exhaust groove 570 which communicates with exhaust passages 560, opposite product delivery channels 580, opposite purge channels 590, a high-presure central feed passage 600, a first annular vent groove 610, and a second annular vent groove 620.

**[0074]** With reference additionally to Figure 5B, a parallel, top, second valve surface 630 of the rotary valve shoe 500 will now be described. The purge channels 590 of the engagement surface 520 communicate with each other through vertical, cylindrical purge passages 640 and a rainbow-shaped purge groove 650 on the top surface 630. The equalization channels 550 of the engagement surface 520 extend vertically through the valve shoe 500. Pairs of equalization channels 550 communicate through equalization grooves 660 on the top surface 630. The equalization grooves 660 are generally U-shaped and extend around receiving holes 670. Equalization routing via the grooves 660 on the second valve surface 630, in a plane out of and parallel to a plane defined by the engagement surface 520, helps to maintain the relatively small size of the rotary valve shoe 500 while at the same time enabling more complex fluid routing through the valve shoe 500. The equalization grooves allow the secondary valve surface to be used to equalize pressures between adsorption beds 300.

**[0075]** With reference to Figures 3B, 8A, and 8B, a first valve shoe cover 680 is disposed over the second valve surface 630 to isolate the various grooves and passages on the second valve surface 630. Both the first valve shoe cover 680 and the second valve shoe cover 690 include aligned central holes 691, 692, respectively, for communicating the central feed passage 600 with a high-pressure feed fluid chamber formed around the



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periphery of a cylindrical base 693 of the second valve shoe cover 690. The first valve shoe cover 680 also includes a plurality of holes 694 near its periphery for the purpose of maintaining a balance of pressure during operation on either side of the first valve shoe cover 680 between the cylindrical base 693 and the second valve surface 630. Routing the high-pressure feed fluid into the high-pressure feed fluid chamber on the top or backside of the valve shoe 500 causes pressure balancing on the valve shoe 500 that counteracts the pressure force urging the valve shoe 500 away from the port plate 510. A spring or other type of passive sealing mechanism (not shown) may be used to hold the rotary valve shoe 500 against the port plate 510 when the concentrator 114 is not operating.

**[0076]** With reference to Figure 5A, to additionally counteract the pressure force that works to unseat the rotary valve shoe 500 from the port plate 510, the exhaust goove 570 is sized such that, when the concentrator 114 is operated at nominal feed and purge (vacuum) pressures, the sealing force due to the vacuum in the exhaust groove 570 subtantially balances this unseating pressure force. This enables the use of relatively small passive sealing mechanisms, reducing the torque and power required to turn the rotary valve shoe 500 and also reduces the weight and size of the concentrator 114.

**[0077]** With reference to Figure 6A, the valve port plate 510 will now be described in greater detail. The valve port plate 510 has a flat engagement surface 700 that engages the flat engagement surfce 520 of the rotary valve shoe 500 and a smooth cylindrical sidewall 710. With reference additionally to Figure 3B, an underside of the valve port plate 510 is disposed on a manifold gasket 720. The valve port plate 510 includes multiple sets of generally symmetric concentrically disposed ports or openings aligned with openings in the manifold gasket 720 to communicate the ports in the plate 510 with the passages in the manifold 320. The ports extend vertically through the valve port plate 510 in a direction generally perpendicular to the engagement surface 700. In an alternative embodiment, the ports extend vertically through the valve port plate 510 in an angular direction toward the engagement surface 700. Preferably, all of the ports of each concentric set have the same configuration. Each concentric set of ports will now be described in turn.

**[0078]** A first set of eight circular vacuum ports 730 concentrically disposed at a first radius from the geometric center of the valve port plate 510 communicate with the vacuum chamber 430 of the manifold 320 and the exhaust gas grooves 570 of the valve



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shoe 500. In the prefered embodiment, eight ports are used as they allow sufficient gas flow through the valve without significant pressure drop. In an alternative embodiment, a number of ports different from eight could be used.

**[0079]** A second set of five round outgoing feed ports 740 concentrically disposed at a second radius from the geometric center of the valve port plate 510 communicate with outgoing feed passages 390 of the manifold 320, the feed channels 540 of the valve shoe 500, and the vacuum ports 730 via the exhaust passages 560 of the valve shoe 500.

**[0080]** A third set of five generally elliptical incoming product ports 750 concentrically disposed at a third radius from the geometric center of the valve port plate 510 communicate with the incoming product passages 370 of the manifold 320, the equalization channels 550 of the valve shoe 500, the purge channels 590 of the valve shoe 500, and the product delivery channels 580.

**[0081]** A fourth set of five circular outgoing product ports 760 concentrically disposed at a fourth radius from the geometric center of the valve port plate 510 communicate with the outgoing product passages 400 of the manifold 320 and the incoming product ports 750 via the product delivery channels 580.

**[0082]** A fifth set of three circular port plate alignment holes 731 concentrically disposed at a fifth radius from the geometric center of the valve port plate 510 align with alignment pins 321 (Figures 3B, 4) on the manifold 320. The alignment holes 731 ensure the port plate 510 will sit in proper alignment with the manifold 320. In an alternative embodiment, two or more alignment holes located at one or more radiuses from the geometric center of the valve port plate 510 may be aligned with an equal number of alignment pins located at set positions on the manifold 320.

**[0083]** A round central incoming feed port 770 disposed at the geometric center of the valve port plate 510 and the center of rotation of the valve assembly 310 communicates with the incoming feed passage 410 of the manifold 320 and the central feed pasage 600 of the rotary valve shoe 500.

**[0084]** In the rotary valve assembly 310 described above, a maximum of 1 PSI pressure drop occurs through any port of the valve asembly 310 when the system is producing 3 LPM of oxygen product. At lesser flows, the pressure drop is negligible.



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**[0085]** With reference additionally to Figure 6B, a single pressure swing adsorption cycle of the concentrator 114 will now be described. During use, the rotary valve shoe 500 rotates with respect to the valve port plate 510 so that the cycle described below is sequentially and continuously established for each adsorption bed 300. The speed of rotation of the rotary valve shoe 500 with respect to the valve port plate 510 may be varied alone, or in combination with a variable-speed compressor, in order to provide the optimal cycle timing and supply of ambient air for a given production of product. To help the reader gain a better understanding of the invention, the following is a description of what occurs in a single adsorption bed 300 and the rotary valve assembly 310 during a single cycle. It should be noted, with each revolution of the rotary valve shoe 500, the adsorption beds 300 undergo two complete cycles. For each cycle, the steps include: 1) pre-pressurization 774, 2) adsorption 776, 3) first equalization down 778, 4) second equalization down 780, 5) co-current blowdown 782, 6) low-pressure venting 784, 7) counter-current purge and low-pressure venting 786, 8) first equalization up 788, and 9) second equalization up 790. Each of these steps will be described in turn below for an adsorption bed 300.

**[0086]** In the pre-pressurization step 774, air flows from the compressor 112 to the feed pressure line 420, through the incoming feed passage 410 of the manifold 320. From there, air flows through the central incoming feed port 770 of the port plate 510, through the central feed passage 600 and out the feed channels 540 of the valve shoe 500, through the outgoing feed ports 740, and through outgoing feed passages 390 of the manifold 320. From there, the feed air flows to the feed ends 360 of the adsorption beds 300. With reference to Figure 5A, because the feed channel 540 is advanced with respect to the product delivery channel 580 (i.e., initially the feed channel 540 is in communication with outgoing feed port 740 and the product delivery channel 580 is blocked, not in communication with the incoming product port 750), the feed end 360 of the adsorption bed 300 is pressurized with feed gas, i.e., pressurized, prior to the commencement of product delivery. In alternative embodiments, the product end 350 may be pre-pressurized with product gas, or the product end 350 may be pre-pressurized with product gas and the feed end 360 may be pre-pressurized with feed gas.

**[0087]** In the adsorption step 776, because the product delivery channel 580 is in communication with the incoming product port 750, adsorption of Nitrogen occurs in the



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bed 300 and the resulting product oxygen gas flows towards the product ends 350 of the adsorption beds 300, through the product lines 380, and through incoming product passages 370 of the manifold 320. From there, oxygen gas flows through the incoming product port, into and out of the product delivery channel 580, through outgoing product port 760, through the outgoing product passage 400, and into the product tank 330. From the product tank 330, oxygen gas is supplied to the user 108 through the product delivery line 450 and the supply line 121.

**[0088]** In the first equalization-down step 778, the product end 350 of the bed 300, which is at a high pressure, is equalized with the product end of another bed, which is at a low pressure, to bring the product end 350 of the bed 300 to a lower, intermediate pressure. The product ends 350 communicate through the product lines 380, the incoming product passages 370, the incoming product ports 750, the equalization channels 550, and the equalization groove 660. As indicated above, equalization routing via the grooves 660 on the second valve surface 630, in a plane out of and parallel to a plane defined by the engagement surface 520, helps to maintain the relatively small size of the rotary valve shoe 500, in order to keep the torque required to turn the valve shoe 500 as low as possible, while at the same time enabling more complex fluid routing through the valve shoe 500. In this step 778 and the equalization steps 780, 788, 790 to be discussed below, the adsorption beds 300 may be equalized at either the feed end 360, the product end 350, or a combination of the feed end 360 and the product end 350.

**[0089]** In the second equalization-down step 780, the product end 350 of the bed 300, which is at an intermediate pressure, is equalized with the product end of another bed, which is at a lower pressure, to bring the product end 350 of the bed 300 further down to an even lower pressure than in step 778. Similiar to the first equalization-down step 778, the product ends 350 communicate through the product lines 380, the incoming product passages 370, the incoming product ports 750, the equalization channels 550, and the equalization groove 660.

**[0090]** In the co-current blowdown ("CCB") step 782, oxygen enriched gas produced from the product end 350 of the adsorption bed 300 is used to purge a second adsorption bed 300. Gas flows from the product side of the adsorption bed 300, through product line 380, incoming product passage 370, and incoming product port 750. The gas further flows through purge channel 590, purge passage 640, through the purge groove



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650, out the purge passage 640 on the opposite side of the valve shoe 500, through the purge channel 590, through the incoming product port 750, through the incoming product pasasge 370, through the product line 380, and into the product end 350 of adsorption bed 300 to serve as a purge stream. In an alternative embodiment, in this step 782 and the following step 784, co-current blowdown may be replaced with counter-current blowdown.

**[0091]** In the low-pressure venting ("LPV") step 784, the adsorption bed 300 is vented to low pressure through the feed end 360 of the adsorption bed 300. The vacuum in the exhaust groove 570 of the rotary valve shoe 500 communicates with the exhaust passage 560 and the feed end 360 of the adsorption bed 300 (via the outgoing feed port 740 and outgoing feed passage 390) to draw the regeneration exhaust gas out of the adsorption bed 300. The low pressure venting step 784 occurs without introduction of oxygen enriched gas because the exhaust passage 560 is in communication with the outgoing feed port 740 and the purge channel 590 is not in communication with the incoming product port 750.

**[0092]** In the counter-current purge and low-pressure venting ("LPV") step 786, oxygen enriched gas is introduced into the product end 350 of the adsorption bed 300 in the manner described above in step 782 concurrently with the feed end 360 of the adsorption bed 300 being vented to low pressure as was described in the above step 784. Counter-current purge is introduced into the product end 350 of the adsorbent bed 300 through fluid communication with the product end 350 of a second adsorption bed 300. Oxygen enriched gas flows from the product end 350 of the second adsorption bed 300 through the product line 380, incoming product passage 370, incoming product port 750, through purge channel 590, purge passage 640, through the purge groove 650, out the purge passage 640 on the opposite side of the valve shoe 500, through the purge channel 590, through the incoming product port 750, through the incoming product passage 370, through the product line 380, and into the product end 350 of adsorption bed 300. Because the exhaust passage 560 is also in communication with the outgoing feed port 740 during this step 786, oxygen enriched gas flows from the product end 350 to the feed end 360, regenerating the adsorption bed 300. The vacuum in the exhaust groove 570 of the rotary valve shoe 500 communicates with the exhaust passage 560 and the feed end 360 of the adsorption bed 300 (via the outgoing feed port 740 and outgoing feed passage 390) to



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draw the regeneration exhaust gas out of the adsorption bed 300. From the exhaust passage 560, the exhaust gas flows through the vacuum ports 730, into the vacuum chamber 430, and out the vacuum presure line 440. In an alternative embodiment, the vacuum may be replaced with a low-pressure vent that is near atmospheric pressure or another pressure that is low relative to the feed pressure. In another embodiment, product gas from the product tank 330 is used to purge the product end 350 of the adsorbent bed 300.

**[0093]** In the first equalization-up step 788, the product end 350 of the bed 300, which is at a very low pressure, is equalized with the product end of another bed, which is at a high pressure, to bring the adsorption bed 300 to a higher, intermediate pressure. The product ends 350 communicate through the product lines 380, the incoming product passages 370, the incoming product ports 750, the equalization channels 550, and the equalization groove 660.

**[0094]** In the second equalization-up step 790, the product end 350 of the bed 300, which is at an intermediate pressure, is equalized with the product end of another bed, which is at a higher pressure, to bring the product end 350 of the bed 300 further up to an even higher pressure than in step 788. Similiar to the first equalization-down step 778, the product ends 350 communicate through the product lines 380, the incoming product passages 370, the incoming product ports 750, the equalization channels 550, and the equalization groove 660.

**[0095]** It should be noted, in a preferred embodiment, the combined duration of feed steps 774, 776 may be substantially the same as the combined duration of purge steps 782, 784, 786, which may be substantially three times the duration of each equalization step 778, 780, 788, 790. In an alternative embodiment, the relative duration of the feed steps 774, 776, the purge steps 782, 784, 786, and the each equalization step 778, 780, 788, 790 may vary.

**[0096]** After the second equalization-up step 790, a new cycle begins in the adsorption bed 300 starting with the pre-pressurization step 774.

**[0097]** The five-bed concentrator 114 and cycle described above has a number of advantages over other-numbered concentrators and cycles used in the past, some of which are described below. The multiple equalization steps 788, 790 at the product ends 350 and the pre-pressurization step 774 contribute to the pre-pressurization of the



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adsorption beds 300 prior to product delivery. As a result, the beds 300 reach their ultimate pressure (substantially equal to the feed pressure) quickly and thereby allow for maximum utilization of the adsorbent media. Additionally, pre-pressurizing the adsorbent beds 300 allows product to be delivered at substantially the same pressure as the feed, thereby retaining the energy of compression in the stream, which makes the product stream more valuable for use in downstream processes. In an alternative embodiment, pre-pressurizing the beds 300 with product before exposing the feed end 360 of the bed 300 to the feed stream eliminates any pressure drop experienced due to the fluid interaction or fluid communication between two or more adsorbent beds 300 on the feed end 360. Additionally, compared to systems with greater numbers of beds, the use of a 5-bed system, reduces the duration and number of beds that are in fluid communication with the feed chanels 540 at the same time, thereby reducing the propensity for fluid flow between adsorption beds. Since fluid flow between adsorption beds is associated with a reversal of the flow direction in the higher pressure bed (resulting in decreased performance), reduction in this effect is advantageous.

**[0098]** A further advantage of a 5-bed system over many systems is that it includes a small number of adsorption beds 300, allowing the concentrator to be relative small, compact, and light-weight, while delivering sufficient flow and purity and maintaining high oxygen recovery. Other PSA systems, typically those with a small number of adsorption beds, result in deadheading the compressor (resulting in high power use) during a portion of the cycle. Deadheading the compressor eliminates detrimental flow between the feed side 360 of the two or more adsorption beds 300 (as discussed above) but increases system power. The 5-bed system eliminates compressor deadheading and minimizes performance-limiting feed side 360 flow between adsorbent beds 300.

**[0099]** Use of the multiple pressure equalization steps 778, 780, 788, 790 reduces the amount of energy of compression required to operate the concentrator 114. Equalizing the beds 300 conserves high-pressure gas by moving it to another bed 300 rather than venting it to the atmosphere or to a vacuum pump. Because there is a cost associated with pressurizing a gas, conserving the gas provides a savings and improves recovery. Also, because a bed 300 may contain gas enriched with product, usually at the product end 350 of the bed 300, allowing this gas to move into another bed 300, rather than venting it, conserves product and improves recovery. The number of equalizations



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are preferably between one and four. It should be noted, each equalization represents two equalization steps, an equalization-down step and an equalization-up step. Thus, two equalizations means two down equalizations and two up equalizations, or four total equalizations. The same is true for other-number equalizations. In a preferred embodiment, one to four equalizations (two to eight equalization steps) are used in each cycle. In a more preferred embodiment, one to three equalizations (two to six equalization steps) are used in each cycle. In a most preferred embodiment, two equalizations (four equalization steps) are used in each cycle.

**[00100]** In alternative embodiments, the concentrator 114 may have other numbers of adsorption beds 300 based on the concentration of the feed stream, the specific gases to be separated, the pressure swing adsorption cycle, and the operating conditions. For example, but not by way of limitation, there also are advantages to four-bed concentrators and six-bed concentrators. When operating a cycle similar to that described above with a four-bed concentrator, the problem of fluid communication between the feed chanels 540 and more than one adsorption bed (at one instant) is completely eliminated. When the feed-end fluid communication is eliminated, the feed steps 774, 776 occur in a more desirable fashion resulting in improved recovery of the desired product.
The advantages of a six-bed system, compared to a five-bed system, are realized when the pressure-swing cycle described above is modified so that there are three equalization up stages and three equalization down stages instead of two equalization up stages and two equalization down stages. A third equalization is advantageous when the feed gas is available at high pressure. The third equalization conserves compressor energy because it allows the equalized beds to obtain substantially 75% of the feed pressure compared to substantially 67% of the feed pressure when two equalization stages are used. In any PSA cycle, whenever an equalization up occurs, there is a corresponding equalization down. The requirement of matching equalization stages inparts some restrictions on the relative timing of the cycle steps. If, for example, the duration of the feed step is substantially the same as the duration of each equalization step, then a six-bed cycle would provide the required matching of equalization stages.

**[0100]** A number of additional inventive aspects related to the concentrator 114 that increase recovery of a desired component and system productivity will now be described. With reference to Figures 3A, 3B, 7A, and 7B, an embodiment of a media



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retention cap 800 that reduces dead volume in the adsorption beds 300 will now be described. Each media retention cap 800 is located at the product end 350 of the adsorption bed 300 and supports the adsorbent material above the media retention cap 800. A spring 810 located within and below the media retention cap 800 urges the media retention cap 800 upwards to hold the packed bed of adsorbent material firmly in place. The media retention cap 800 has a cylindrical base 820 with first and second annular flanges 830, 840. The second annular flange 840 terminates at its top in a circular rim 850. A top surface 860 of the media retention cap 800 includes a plurality of ribs 870 radiating in a generally sunburst pattern from a central port 880. Adjacent the central port 880, gaps 890 create diffusion zones for purge fluid coming out of the central port 880. The gaps 890 and the radiating ribs 870 cause the purge fluid to be distributed outward from the central port 880, causing a more uniform, improved regeneration of the adsorbent material during a purging step. The radiating ribs 870 also help to channel product gas towards the central port 880 during a product delivery step. In an alternative embodiment, the media retention cap 800 may have a generally non-cylindrical surface to retain media in a generally non-cylindrical adsorbtion bed 300. In a further alternative embodiment, the central port 880 may be located away from the geometric center of the either cylindrical or non-cylindrical media retention cap 800.

**[0101]** With reference to Figure 7B, on the underside of the media retention cap 800, the cydrical base 820 forms an interior chamber in which the spring 810 is disposed. A central port nipple 900 extends from a bottom surface 910 of the media retention cap 800. An end of the product line 380 connects to the central port nipple 900 for communicating the product end 350 of the adsorption bed 300 with the incoming product passage 370 of the manifold 320.

**[0102]** In the past, media retention caps may be held in place with a spring that fits inside and above the cap so that the spring is in the fluid flow path between the bottom of the adsorbent material and any exit port, at the product end 350 of the bed 300. The volume in which the spring is housed represents dead volume in the system. As used herein, "dead volume" is system volume that is compressed and purged, but does not contain adsorbent media. The process of filling this volume with compressed feed and then venting that volume represents wasted feed. The improved media retention cap 800 does not add dead volume to the system because the spring 810 is housed outside of the



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fluid flow path. Elimination of any extra volume within the system results directly in more effective utilization of the feed, and, thus, higher recovery of the desired product.

**[0103]** With reference to Figures 8A and 8B, an embodiment of a centering mechanism for maintaining the rotary valve shoe 500 laterally fixed and centered with respect to the valve port plate 510 will now be described. The centering mechanism may include a centering pin 920 having a hollow cylindrical shape and made of a rigid material. When the engagement surface 520 of the rotary valve shoe 500 is engaged with the engagement surface 700 of the valve port plate 510, the centering pin 920 is partially disposed in the central feed pasage 600 of the rotary valve shoe 500 and the central incoming feed port 770 of the valve port plate 510. In use, the rotary valve shoe 500 rotates around the centering pin 920 and the hollow interior of the centering pin 920 allows high-pressure feed fluid to flow therethrough. The pin 920 maintains the rotating valve shoe in a fixed position relative to the valve port plate 510. In the past, the rotary valve shoe was roughly centered with respect to the valve port plate by the motor that drives the rotary valve shoe. If the rotary valve shoe 500 and the valve port plate 510 are off center with respect to each other, the concentrator 114 will not cycle as intended, inhibiting the productivity, recovery, and efficiency of the concentrator. The precision offered by the centering pin 920 is important when the valve assembly 310 is controlling complex cycles or maintaining very small pressure drops.

**[0104]** With reference to Figures 9A and 9B, a rotary valve assembly constructed in accordance with another embodiment of the invention includes an alternative centering mechanism to maintain the rotating valve shoe 500 in a fixed position relative to the valve port plate 510. A circular centering ring 930 fits snugly over the smooth cylindrical sidewall 530 of the rotary valve shoe 500 and the smooth cylindrical sidewall 710 of the stationary valve port plate 510. The circular ring 930 centers the rotary valve shoe 500 relative to the valve port plate 510 by holding the rotary shoe 500 in a fixed position relative to the port plate 510 while at the same time allowing the rotary valve shoe 500 to rotate.

**[0105]** With reference to Figures 10A-10C, an embodiment of an elastic link for coupling the motor 118 to the valve shoe 500 will now be described. A drive mechanism 940 includes a drive shaft 950, a drive wheel 960, and three (two shown) elastic chain links 970. The drive shaft 950 may be connected to the motor 118 for rotating the drive



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wheel 960. With reference to Figure 10C, a lower side 980 of the drive wheel 960 may include downwardly protruding cylindrical support posts 990. Similarly, with reference to Figure 10B, an upper side 1000 of the second valve shoe cover 690 may include upwardly protruding cylindrical support posts 1010. The elastic chain links 970 are preferably made of semi-rigid, elastic material (such as silicon rubber) and have a generally wrench-shaped configuration. Each elastic chain link 970 includes cylindrical receiving members 1020 with central cylindrical bores 1030. The cylindrical receiving members 1020 are joined by a narrow connecting member 1040. The drive wheel 960 is coupled to the second valve shoe cover 690 through the elastic chain links 970. One receiving member 1020 of each elastic chain link receives the support post 990 of the drive wheel 960 and the other receiving member 1020 receives the support post 1010 of the second valve shoe cover 690. In the past, rigid connections were made between the motor and the rotating valve shoe. These rigid connections caused the rotating valve shoe to be affected by vibration or other non-rotational movement of the motor. The elastic chain links 970 absorb the vibration and non-rotational movement of the motor, preventing this detrimental energy from being imparted to the rotating valve shoe 500.

**[0106]** Figure 11 is a table of experimental data from a concentrator similar to the concentrator 114 shown and described above with respect to Figures 3-10. As shown by this table, the recovery of oxygen from air with the concentrator 114 is 45-71% at about 90% purity. The ratio of adiabatic power (Watts) to oxygen flow (Liters Per Minute) is in the range of 6.2 W/LPM to 23.0 W/LPM. As defined in Marks' Standard Handbook for Mechanical Engineers, Ninth Edition, by Eugene A. Avallone and Theodore Baumeister, the equation for adiabatic power, tanken from the equation from adiabatic work, is as follows:

$$Power = \frac{W}{t} = P_1 V_1 \left( \frac{k}{1-k} \right) \left[ \left( \frac{P_2}{P_1} \right)^{\frac{k-1}{k}} - 1 \right] C$$

Power = Adiabatic Power (Watts)

W = Adiabatic Work (Joule)

t = time (Second)



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$P_1$ = Atmospheric Pressure (psia)

$P_2$ = Compressor / Vacuum pressure (psia)

k = Ratio of Specific Heats = constant = 1.4 (for air)

$V_1$ = Volumetric flow rate at atmospheric pressure (SLPM)

C = Conversion Factor, added by authors for clarity = 0.114871 Watts / psi / LPM

B. Energy Source

**[0107]** With reference additionally to Figure 12, in order to properly function as a lightweight, portable system 100, the system 100 must be energized by a suitable rechargeable energy source. The energy source preferably includes a rechargeable battery 104 of the lithium-ion type. It will be readily apparent to those skilled in the art that'. the system 100 may be powered by a portable energy source other than a lithium-ion battery. For example, a rechargeable or renewable fuel cell may be used. Although the system is generally described as being powered by a rechargeable battery 104, the system 100 may be powered by multiple batteries. Thus, as used herein, the word "battery" includes one or more batteries. Further, the rechargeable battery 104 may be comprised of one or more internal and/or external batteries. The battery 104 or a battery module including the battery 104 is preferably removable from the system 100. The system 100 may use a standard internal battery, a low-cost battery, an extended-operation internal battery, and an external secondary battery in a clip-on module.

**[0108]** The system 100 may have a built-in adapter including battery charging circuitry 130 and one or more plugs 132 configured to allow the system 100 to be powered from a DC power source (e.g., car cigarette lighter adapter) and/or an AC power source (e.g., home or office 110 VAC wall socket) while the battery 104 is simultaneously being charged from the DC or AC power source. The adapter or charger could also be separate accessories. For example, the adapter may be a separate cigarette lighter adapter used to power the system 100 and/or charge the battery 104 in an automobile. A separate AC adapter may be used to convert the AC from an outlet to DC for use by the system 100 and/or charging the battery 104. Another example of an adapter may be an adapter used with wheel chair batteries or other carts.

**[0109]** Alternatively, or in addition, a battery-charging cradle 134 adapted to receive and support the system 100 may have an adapter including battery charging



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circuitry 130 and a plug 132 that also allow the system 100 to be powered while the battery 104 is simultaneously being charged from a DC and/or AC power source.

**[0110]** The system 100 and cradle 134 preferably include corresponding mating sections 138, 140 that allow the system 100 to be easily dropped into and onto the cradle 134 for docking the system 100 with the cradle 134. The mating sections 138, 140 may include corresponding electrical contacts 142, 144 for electrically connecting the system 100 to the cradle 134.

**[0111]** The cradle 134 may be used to recharge and/or power the system 100 in the home, office, automobile, etc. The cradle 134 may be considered part of the system 100 or as a separate accessory for the system 100. The cradle 134 may include one or more additional charging receptacles 146 coupled to the charging circuitry 130 for charging spare battery packs 104. With a charging receptacle 146 and one or more additional battery packs 104, the user can always have a supply of additional fresh, charged batteries 104.

**[0112]** In alternative embodiments, the cradle 134 may come in one or more different sizes to accommodate one or more different types of systems 100.

**[0113]** The cradle 134 and/or system 100 may also include a humidifying mechanism 148 for adding moisture to the air flow in the system 100 through appropriate connections 149. In an alternative embodiment of the invention, the humidifying mechanism 148 may be separate from the system 100 and the cradle 134. If separate from the system 100 and cradle 134, the cradle 134 and/or system 100 may include appropriate communication ports for communicating with the separate humidifying mechanism 148. The cradle 134 may also include a receptacle adapted to receive a separate humidifying mechanism 148 for use with the system 100 when the system 100 is docked at the cradle 134.

**[0114]** The cradle 134 and/or system 100 may also include a telemetry mechanism or modem 151 such as a telephone modem, high-speed cable modem, RF wireless modem or the like for communicating the control unit 110 of the system 100 with one or more remote computers. To this end, the cradle 135 may include a line 153 with a cable adapter or telephone jack plug 155, or a RF antenna 157. In an alternative embodiment of the invention, the telemetry mechanism or modem 151 may be separate from the cradle 134 and to this end, the cradle 134 or system 100 may include one or more



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appropriate communication ports, e.g., a PC port, for directly communicating the telemetry mechanism or modem 151 with the cradle 134 or system 100. For example, the cradle 134 may be adapted to communicate with a computer (at the location of the cradle) that includes the telemetry mechanism or modem 151. The computer may include appropriate software for communicating information described below using the telemetry mechanism or modem 151 with the one or more remote computers.

**[0115]** The telemetry mechanism or modem 151 may be used to communicate physiological information of the user such as, but not by way of limitation, heart rate, oxygen saturation, respiratory rate, blood pressure, EKG, body temperature, inspiratory/expiratory time ratio (I to E ratio) with one or more remote computers. The telemetry mechanism or modem 151 may be used to communicate other types of information such as, but not by way of limitation, oxygen usage, maintenance schedules on the system 100, and battery usage with one or more remote computers.

**[0116]** A user ideally uses the system 100 in its cradle 134 at home, at the office, in the automobile, etc. A user may decide to have more than one cradle, e.g., one at home, one at the office, one in the automobile, or multiple cradles at home, one in each room of choice. For example, if the user has multiple cradles 134 at home, when the user goes from room to room, e.g., from the family room to the bedroom, the user simply lifts the system 100 out of its cradle 134 in one room, and walks to the other room under battery operation. Dropping the system 100 in a different cradle 134 in th destination room restores the electrical connection between the system 100 and the AC power source. Since the system's batteries 104 are constantly charging or charged when located in the cradle 134, excursions outside the home, office, etc. are as simple as going from room to room in the user's home.

**[0117]** Because the system 100 is small and light (2-15 pounds), the system 100 may simply be lifted from the cradle 134 and readily carried, e.g., with a shoulder strap, by an average user to the destination. If the user is unable to carry the system 100, the system 100 may be readily transported to the destination using a cart or other transporting apparatus. For an extended time away from home, office, etc., the user may bring one or more cradles 134 for use at the destination. Alternatively, in the embodiment of the system 100 including the built-in adapter, power may be drawn from power sources such as a car cigarette lighter adapter and/or an AC power outlet available at the destination.



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Further, spare battery Packs 104 may be used for extended periods away from standard power sources.

**[0118]** If the battery pack 104 includes multiple batteries, the system 100 may include a battery sequencing mechanism to conserve battery life as is well known in the cellphone and laptop computer arts.

C. Output Sensor

**[0119]** With reference to Figures 1, 2 and 13, one or more output sensors 106 are used to sense one or more conditions of the user 108, environment, etc. to determine the oxygen flow rate needs of the user and, hence, the oxygen flow rate output requirements for the system 100. A control unit 110 is linked to the one or more output sensors 106 and the oxygen gas generator 102 to control the oxygen generator 102 in response to the condition(s) sensed by the one or more output sensors 106. These output sensors include a pressure sensor 150, a position sensor 152, an acceleration sensor 154, as well as a physiological condition or metabolic sensor 156 and an altitude sensor 158.

**[0120]** The first three sensors 150, 152, 154 (and, in certain circumstances, the physiological condition sensor 156) are activity sensors because these sensors provide a signal representing activity of the user 108. In the delivery of oxygen with a portable oxygen concentration system, it is important to deliver an amount of oxygen gas proportional to the activity level of the user 108 without delivering too much oxygen. Too much oxygen may be harmful for the user 108 and reduces the life of the battery 104. The control unit 110 regulates the oxygen gas generator 102 to control the flow rate of oxygen gas to the user 108 based on the oneor more signals representative of the activity level of the user produced by the one or more sensors 106. For example, if the output sensor(s) 106 indicates that the user 108 has gone from an inactive state to an active state, the control unit 110 may cause the oxygen gas generator 102 to increase the flow rate of oxygen gas to the user 108 and/or may provide a burst of oxygen gas to the user 108 from a high-pressure oxygen reservoir to be described. If the output sensor(s) 106 indicates that the user 108 has gone from an active state to an inactive state, the control unit 110 may cause the oxygen gas generator 102 to reduce the flow rate of oxygen gas to the user.

**[0121]** In an embodiment of the invention, the amount of oxygen gas supplied is controlled by controlling the speed of the compressor motor 118 via the variable-speed controller 119.



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**[0122]** Alternatively, or in addition to the variable-speed controller, the supply of oxygen gas may be controlled by the supply valve 160 located in the supply line 121 between the oxygen gas (generator 102 and the user 108. For example, the supply valve 160 may be movable between at least a first position and a second position, the second position allowing a greater flow of concentrated gaseous oxygen through than the first position. The control unit 110 may cause the supply valve 160 to move from the first position to the second position when one or more of the activity level sensors 152, 154, 156 senses an active level of activity of the user 108. For example, the control unit 110 may include a timer, and when an active level is sensed for a time period exceeding a predetermined timed period, the control unit 110 causes the valve 160 to move from the first position to the second position.

**[0123]** Examples of pressure sensors 150 include, without limitation, a foot switch that indicates when a user is in a standing position compared to a sedentary position, and a seat switch that indicates when a user is in a seated position compared to a standing position.

**[0124]** A pendulum switch is an example of a position sensor 152. For example, a pendulum switch may include a thigh switch positioned pendulously to indicate one mode when the user is standing, i.e., the switch hangs vertically, and another mode when the user seated, i.e., the thigh switch raised to a more horizontal position. A mercury switch may be used as a position sensor.

**[0125]** An acceleration sensor 158 such as an accelerometer is another example of an activity sensor that provides a signal representing activity of the user.

**[0126]** The physiological condition or metabolic sensor 156 may also function as an activity sensor. The physiological condition sensor 156 may be used to monitor one or more physiological conditions of the user for controlling the oxygen gas generator 102 or for other purposes. Examples of physiological conditions that may be monitored with the sensor 156 include, but without limitation, blood oxygen level, heart rate, respiration rate, blood pressure, EKG, body temperature, and I to E ratio. An oximeter is an example of a sensor that is preferably used in the system 100. The oximeter measures the blood oxygen level of the user, upon which oxygen production may be at least partially based.

**[0127]** An altitude sensor 158 is an example of an environmental or ambient condition sensor that may sense an environmental or ambient condition upon which



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control of the supply of oxygen gas to the user may be at least partially based. The altitude sensor 158 may be used alone or in conjunction with any or all of the above sensors, the control unit 110 and the oxygen gas generator 102 to control the supply of oxygen gas to the user in accordance with the sensed altitude or elevation. For example, at higher sensed elevations, where air is less concentrated, the control unit may increase the flow rate of oxygen gas to the user 108 and at lower sensed elevations, where air is more concentrated, the control unit may decrease the flow rate of oxygen gas to the user 108 or maintain it at a control level.

**[0128]** It will be readily apparent to those skilled in the art that one or more additional or different sensors may be used to sense a condition upon which control of the supply of oxygen gas to the user may be at least partially based. Further, any or all of the embodiments described above for regulating the amount of oxygen gas supplied to the user 108, i.e., variable-speed controller 119, supply valve 160, (or alternative embodiments) may be used with the one or more sensors and the control unit 110 to control of the supply of oxygen gas to the user 108.

D. Control Unit

**[0129]** With reference to Figure 14, the control unit 110 may take any well-known form in the art and includes a central microprocessor or CPU 160 in communication with the components of the system described herein via one or more interfaces, controllers, or other electrical circuit elements for controlling and managing the system. The system 100 may include a user interface (Figure 14) as part of the control unit 110 or coupled to the control unit 110 for allowing the user, provider, doctor, etc. to enter information, e.g., prescription oxygen level, flow rate, activity level, etc., to control the system 100.

**[0130]** The main elements of an embodiment of the system 100 have been described above. The following sections describe a number of additional features, one or more of which may be incorporated into the embodiments of the invention described above as one or more separate embodiments of the invention.

II. Conserving Device

**[0131]** With reference to Figure 15, a conserving device or demand device 190 may be incorporated into the system 100 to more efficiently utilize the oxygen produced by the oxygen gas generator 102. During normal respiration, a user 108 inhales for about



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one-third of the time of the inhale/exhale cycle and exhales the other two-thirds of the time. Any oxygen flow provided to the user 108 during exhalation is of no use to the user 108 and, consequently, the additional battery power used to effectively provide this extra oxygen flow is wasted. A conserving device 190 may include a sensor that senses the i nhale/exhale cycle by sensing pressure changes in the cannula 111 or another part of the system 100, and supply oxygen only during the inhale portion or a fraction of the inhale portion of the breathing cycle. For example, because the last bit of air inhaled is of no particular use because it is trapped between the nose and the top of the lungs, the conserving device 190 may be configured to stop oxygen flow prior to the end of inhalation, improving the efficiency of the system 100. Improved efficiency translates into a reduction in the 20size, weight, cost and power requirements of the system 100.

**[0132]** The conserving device 190 may be a stand-alone device in the output line of the system 100, similar to a regulator for scuba diving, or may be coupled to the control unit 110 for controlling the oxygen generator 102 to supply oxygen only during inhalation by the user 108.

**[0133]** The conserving device 190 may include one or more of the sensors described above. For example, the conserving device may include a sensor for monitoring the respiration rate of the user.

**[0134]** The system 100 may also include a special cannula retraction device for retracting the cannula ill when not in use. Further, the cannula 111 may come in different lengths and sizes.

III. High-pressure reservoir

**[0135]** With reference to Figure 16, a high-pressure reservoir 164 may be located in a secondary line 166 for delivering an additional supply of oxygen gas to the user 108 when the oxygen gas generator 102 can not meet the oxygen gas demands of the user 108. Any of the components described below in the secondary line 166 may be coupled to the control unit 110 or a high-pressure reservoir controller 167 (Figure 14) for control thereby. Exemplary situations where this additional oxygen gas need may occur are when a user suddenly goes from an inactive state to an active state, e.g., when getting out of a chair, when the system 100 is turned on, or when the system 100 goes from a conserving mode or sleep mode to an active mode. As used herein, secondary line 166 refers to the tubing, connectors, etc. used to connect the components in the line. A valve



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168 may be controlled by the control unit 110 to allow gaseous oxygen to flow into the secondary line 166. The valve 168 may be configured to allow simultaneous flow to both the supply line 121 and the secondary line 166, flow to only the supply line 121, or flow to only the secondary line 166.

**[0136]** A pump or compressor 168, which is preferably powered by the motor 118, delivers the oxygen gas at a relatively high pressure, e.g., at least approximately 100 psi, to the high-pressure reservoir 164.

**[0137]** An oxygen-producing electrochemical cell 171 may be used in conjunction with or instead of the elements described in the secondary line 166 to supply additional oxygen gas to the user 108. For example the electrochemical cell 171 may be used to deliver oxygen gas at a relatively high pressure to the highpressure reservoir 164.

**[0138]** A pressure sensor 172 is in communication with the high-pressure reservoir 164 and the control unit 110 so that when the pressure in the high-pressure reservoir 164 reaches a certain limit, the control unit 110 causes the valve 168 to direct oxygen to the secondary line 166.

**[0139]** A regulator 174 may be used to control flow and reduce pressure of the oxygen gas to the user 108.

**[0140]** A valve 176 may also be controlled by the control unit110 to allow gaseous oxygen from the high-pressure reservoir 164 to flow into the supply line 121 when the user 108 requires an amount of oxygen gas that cannot be met by the oxygen gas generator 102. The valve 176 may be configured to allow simultaneous flow from the oxygen gas generator 102 and the high-pressure reservoir 164, from only the oxygen gas generator 102, or from only the high-pressure reservoir 164.

**[0141]** The one or more sensors 106 are interrelated with the control unit 110 and the oxygen gas generator 102 so as to supply an amount of oxygen gas equivalent to the oxygen gas needs of the user 108 based at least in part upon one or more conditions sensed by the one or more sensors 106. When the oxygen gas generator 102 cannot meet the oxygen gas demands of the user 108, the control unit 110, based at least in part upon sensing one or more conditions indicative of the oxygen needs of the user, may cause the high-pressure reservoir 164 (via the valve 176) to supply the additional oxygen gas needed.



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**[0142]** In the scenario where the oxygen gas generator 102 is capable of supplying the full oxygen gas needs of the user 108, but is simply turned off or is in a conserving or sleep mode, the period of time that the high-pressure reservoir 164 supplies the oxygen gas, i.e., the period of time that the valve 176 connects the high-pressure reservoir 164 with the supply line 121, is at least as long as the time required for the oxygen gas generator 102 to go from an off or inactive condition to an on or active condition. In another scenario, the control unit 110 may cause oxygen gas to be supplied to the user from the high-pressure reservoir 164 when the demand for gaseous oxygen by the user exceeds the maximum oxygen gas output of the oxygen gas generator 102. Although the high-pressure reservoir 164 is shown and described as being filled by the oxygen gas generator 102, in an alternative embodiment, the high-pressure reservoir 164 may be filled by a source outside or external to the system.

IV. Global Positioning System

**[0143]** With reference back to Figure 12, in an alternative embodiment of the invention, the system 100 may include a global positioning system (GPS) receiver 200 for determining the location of the system 100. The location of the receiver 200 and, hence, the user 108 can be transmitted to a remote computer via the telemetry mechanism or modem 151. This may be desirable for locating the user 108 in the event the user has a health problem, e.g., heart attack, hits a panic button on the system, an alarm is actuated on the system, or for some other reason.

V. Additional Options and Accessories

**[0144]** In addition to the cradle 134, the portable oxygen concentration system 100 may include additional options and accessories. A number of different types of bags and carrying cases such as, but not by way of limitation, a shoulder bag, a backpack, a fanny pack, a front pack, and a split pack in different colors and patterns may be used to transport the system 100 or other system accessories. A cover may be used to shield the system from inclement weather or other environmental damage. The system 100 may also be transported with a rolling trolley/cart, a suit case, or a travel case. The travel case may be designed to carry the system 100 and include enough room to carry the cannulae 111, extra batteries, an adapter, etc. Examples of hooks, straps, holders for holding the system 100 include, but not by way of limitation, hooks for seatbelts in cars, hooks/straps for walkers, hooks/straps, for wheel chairs, hooks/straps for hospital beds, hooks for other



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medical devices such as ventilators, hooks/straps for a golf bag or golf cart, hooks/straps for a bicycle, and a hanging hook. The system 100 may also include one or more alarm options. An alarm of the system 100 may be actuated if, for example, a sensed physiological condition of the user 108 falls outside a pre-defined range. Further, the alarm may include a panic alarm that may be manually actuated by the user 108. The alarm may actuate a buzzer or other sounding device on the system 100 and/or cause a communication to be sent via the telemetry mechanism or modem 151 to another entity, e.g., a doctor, a 911 dispatcher, a caregiver, a family member, etc.

**[0145]** Although this invention has been described in terms of certain preferred embodiments, other embodiments apparent to those of ordinary skill in the art are also within the scope of this invention. Accordingly, the scope of this invention is intended to be defined only by the claims that follow.



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We claim:

1. A portable oxygen concentrator system adapted to be readily transported by a user, comprising:

a rechargeable energy source;

a concentrator powered by said energy source and adapted to convert ambient air into concentrated oxygen gas for said user, the concentrator including a plurality of adsorption beds and a rotary valve assembly, the rotary valve assembly relatively rotatable with respect to the plurality of adsorption beds to provide valving action for selectively transferring fluids through the plurality of adsorption beds for converting ambient air into concentrated oxygen gas for said user,

wherein the ratio of adiabatic power to oxygen flow for the concentrator is in the range of 6.2 W/LPM to 23.0 W/LPM.

2. The portable oxygen concentrator system of claim 1, wherein the concentrator includes exactly five adsorption beds.

3. The portable oxygen concentrator system of claims 1 or 2, wherein the rotary valve assembly includes a valve port plate and a rotary valve shoe having respective engaged surfaces and relatively rotatable about a common center of rotation to provide valving action for selectively transferring fluids therethrough, and a centering mechanism other than a motor to center the rotary valve shoe relative to the valve port plate.

4. The portable oxygen concentrator system of claim 3, wherein the valve port plate and the rotary valve shoe include respective central holes, and the centering mechanism includes a centering pin disposed in the central holes of the valve port plate and rotary valve shoe to center the rotary valve shoe relative to the valve port plate.

5. The portable oxygen concentrator system of claim 3, wherein the valve port plate and rotary valve shoe include cylindrical sidewalls, and the centering mechanism includes a centering ring disposed around the sidewalls of the valve port plate and the rotary valve shoe to center the rotary valve shoe relative to the valve port plate.

6. The portable oxygen concentrator system of any of claims 1-5, wherein the adsorption beds carry adsorbent material, and include a first end, a second end, an adsorbent media disposed in the adsorption beds between the first end and the second end, a media retention cap disposed between the adsorbent media and the second end, a spring



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to urge the media retention cap against the adsorbent material to hold the adsorbent media in place, and the spring not located in a flow path of the adsorption beds.

**7.** The portable oxygen concentrator system of claim 6, wherein the media retention cap includes a surface that contacts the adsorbent media, the surface including a central hole and a plurality of ribs radiating from the central hole.

**8.** The portable oxygen concentrator system of claim 6, wherein the media retention cap includes a bottom base with an interior, and the spring is disposed in the interior of the bottom base.

**9.** The portable oxygen concentrator system of any of claims 1-8, wherein the rotary valve assembly includes a valve port plate and a rotary valve shoe having respective engaged surfaces and relatively rotatable about a common center of rotation to provide valving action for selectively transferring fluids therethrough, and the portable oxygen concentrator system further includes a motor to rotate the rotary valve shoe, and one or more elastic links to couple the motor to the rotary valve shoe.

**10.** The portable oxygen concentrator system of claim 9, wherein the motor includes a drive shaft and a drive wheel with one or more protruding support members, the rotary valve shoe includes one or more protruding support members, and the elastic link connects the one or more protruding support members of the drive wheel with the one or more protruding support members of the rotary valve shoe.

**11.** The portable oxygen concentrator system of any of claims 1-10, wherein the concentrator includes plastic adsorption beds and a metal cover surrounding the plastic adsorption beds.

**12.** The portable oxygen concentrator system of claim 11, wherein the adsorption beds are, elongated, molded, plastic vessels.

**13.** The portable oxygen concentrator system of claim 12, wherein the metal cover is made of aluminum and surrounds the adsorption beds to form a product tank.

**14.** The portable oxygen concentrator system of any of claims 1-13, wherein the rotary valve assembly includes a valve port plate and a rotary valve shoe having respective engaged surfaces and relatively rotatable about a common center of rotation to provide valving action to put the adsorption beds in a pressure swing adsorption cycle, and the pressure swing adsorption cyle includes a number of equalization steps in each adsorption bed ranging from two to eight.



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**15.** The portable oxygen concentrator system of claim 14, wherein the pressure swing adsorption cyle includes a number of equalization steps in each adsorption bed ranging from two to six.

**16.** The portable oxygen concentrator system of claim 14, wherein the pressure swing adsorption cyle includes four equalization steps in each adsorption bed.

**17.** The portable oxygen concentrator system of claim 14, wherein the equalization steps include a first equalization down step, a second equalization down step, a first equalization up step, and a second equalization up step.

**18.** The portable oxygen concentrator system of claim 14, wherein adsorption beds include a feed end and a product end, and equalization occurs between product ends of adsorption beds.

**19.** The portable oxygen concentrator system of any of claims 1-18, further including a variable-speed compressor to supply ambient air to the concentrator.

**20.** The portable oxygen concentrator system of any of claims 1-19, wherein recovery of oxygen from air from the concentrator is 45-71% at about 90% purity.

**21.** The portable oxygen concentrator system of any of claims 1-20, wherein the rotary valve assembly includes a valve port plate and a rotary valve shoe having respective engaged surfaces and relatively rotatable about a common center of rotation to provide valving action for selectively transferring fluids therethrough, said valve port plate having at least two ports interconnected with at least two adsorption beds, and said rotary valve shoe having a second valve surface opposite said engaged surface with at least one equalization passage to register with said at least two ports of the port plate to equalize pressure in said at least two adsorption beds.

**22.** The portable oxygen concentrator system of any of claims 1-21, wherein a pressure drop through the rotary valve assembly is no more than 1 PSI when the concentrator is producing 3 LPM of oxygen gas.

**23.** The portable oxygen concentrator system of any of claims 1-22, wherein the rotary valve assembly includes a valve port plate and a rotary valve shoe having respective engaged surfaces and relatively rotatable about a common center of rotation to provide valving action for selectively transferring fluids therethrough, the rotation speed of the rotary valve shoe with respect to the valve port plate is varied in order to provide desired cycle timing for a given production of product.



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**24.** The portable oxygen concentrator system of claim 23, further including a variable-speed compressor to supply compressed ambient air to the concentrator, and the rotation speed of the rotary valve shoe with respect to the valve port plate is varied in combination with variation in the speed of the variable-speed compressor in order to provide desired cycle timing and desired supply rate of ambient air to the concentrator for a given production of product.

**25.** The portable oxygen concentrator system of any of claims 1-24, wherein the rotary valve assembly includes a valve port plate and a rotary valve shoe having respective engaged surfaces and relatively rotatable about a common center of rotation to provide valving action for selectively transferring fluids therethrough, and the rotary valve shoe includes a vacuum sealing mechanism that counteracts a pressure force working to unseat the rotary valve shoe from the valve port plate.

**26.** A rotary valve assembly for a pressure swing adsorption system having a plurality of adsorption beds, comprising:

a valve port plate and a rotary valve shoe having respective engaged surfaces and relatively rotatable about a common center of rotation to provide valving action for selectively transferring fluids therethrough, said valve port plate having at least two ports interconnected with at least two adsorption beds, and said rotary valve shoe having a second valve surface opposite said engaged surface with at least one equalization passage to register with said at least two ports of the port plate to equalize pressure in said at least two adsorption beds.



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AMENDED CLAIMS

[received by the International Bureau on 07 October 2003 (07.10.2003); original claim 26 cancelled; new claims 27-35 added; remaining claims unchanged (2 pages)]

**24.** The portable oxygen concentrator system of claim 23, further including a variable-speed compressor to supply compressed ambient air to the concentrator, and the rotation speed of the rotary valve shoe with respect to the valve port plate is varied in combination with variation in the speed of the variable-speed compressor in order to provide desired cycle timing and desired supply rate of ambient air to the concentrator for a given production of product.

**25.** The portable oxygen concentrator system of any of claims 1-24, wherein the rotary valve assembly includes a valve port plate and a rotary valve shoe having respective engaged surfaces and relatively rotatable about a common center of rotation to provide valving action for selectively transferring fluids therethrough, and the rotary valve shoe includes a vacuum sealing mechanism that counteracts a pressure force working to unseat the rotary valve shoe from the valve port plate.

**27.** A portable oxygen concentrator system adapted to be readily transported by a user, comprising:

an internal rechargeable energy source;

an air separation device powered by said energy source and adapted to convert ambient air into concentrated oxygen gas for said user, the air separation device including a plurality of adsorber columns and a rotating valve, the rotating valve relatively rotatable with respect to the plurality of adsorber columns to provide valving action for selectively transferring fluids through the plurality of adsorber columns for converting ambient air into concentrated oxygen gas for said user,

wherein the portable oxygen concentrator system weighs 2-15 pounds and the adsorber columns each including a layered adsorbent bed having two or more distinct adsorbent material layers.

**28.** The portable oxygen concentrator system of claim 27, wherein the adsorber columns each include a feed end and a product end, and the two or more distinct adsorbent material layers include at least a water adsorption layer and a nitrogen adsorption layer, the water adsorption layer located closer to the feed end than the nitrogen adsorption layer.

**29.** The portable oxygen concentrator system of claim 28, wherein the water adsorption layer is an activated alumina.

**30.** The portable oxygen concentrator system of claim 28, wherein the water adsorption layer is a silica gel.



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**31.** The portable oxygen concentrator system of any of claims 28-30, wherein the nitrogen adsorption layer is a lithium exchanged X-type zeolite.

**32.** A portable oxygen concentrator system adapted to be readily transported by a user, comprising:

an internal rechargeable energy source;

an air separation device powered by said energy source and adapted to convert ambient air into concentrated oxygen gas for said user, the air separation device including a plurality of adsorber columns each including a feed end and a product end and at least one valve operable with respect to the plurality of adsorber columns to provide valving action for selectively transferring fluids through the plurality of adsorber columns for converting ambient air into concentrated oxygen gas for said user,

wherein the portable oxygen concentrator system weighs 2-15 pounds and the adsorber columns each including a layered adsorbent bed having two or more distinct adsorbent material layers, the two or more distinct adsorbent material layers including at least a water adsorption layer and a nitrogen adsorption layer, the water adsorption layer located closer to the feed end than the nitrogen adsorption layer.

**33.** The portable oxygen concentrator system of claim 32, wherein the water adsorption layer is an activated alumina.

**34.** The portable oxygen concentrator system of claim 32, wherein the water adsorption layer is a silica gel.

**35.** The portable oxygen concentrator system of any of claims 32-34, wherein the nitrogen adsorption layer is a lithium exchanged X-type zeolite.



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**Statement Under PCT Article 19(1)**

New claims have been added to further refine and clarify the claimed invention. Prior pending claims 1-25 have not been amended. Claim 26 has been cancelled.



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IPR2023-00527

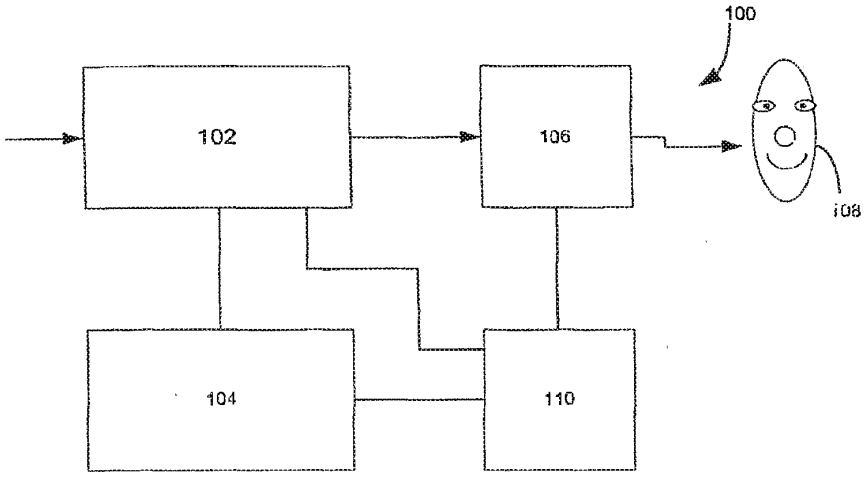



Figure 1

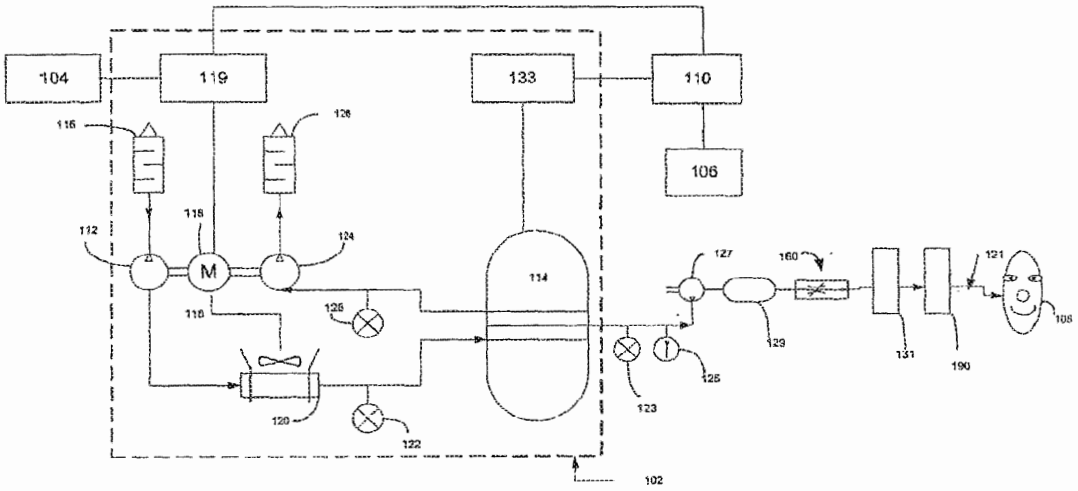

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Figure 2

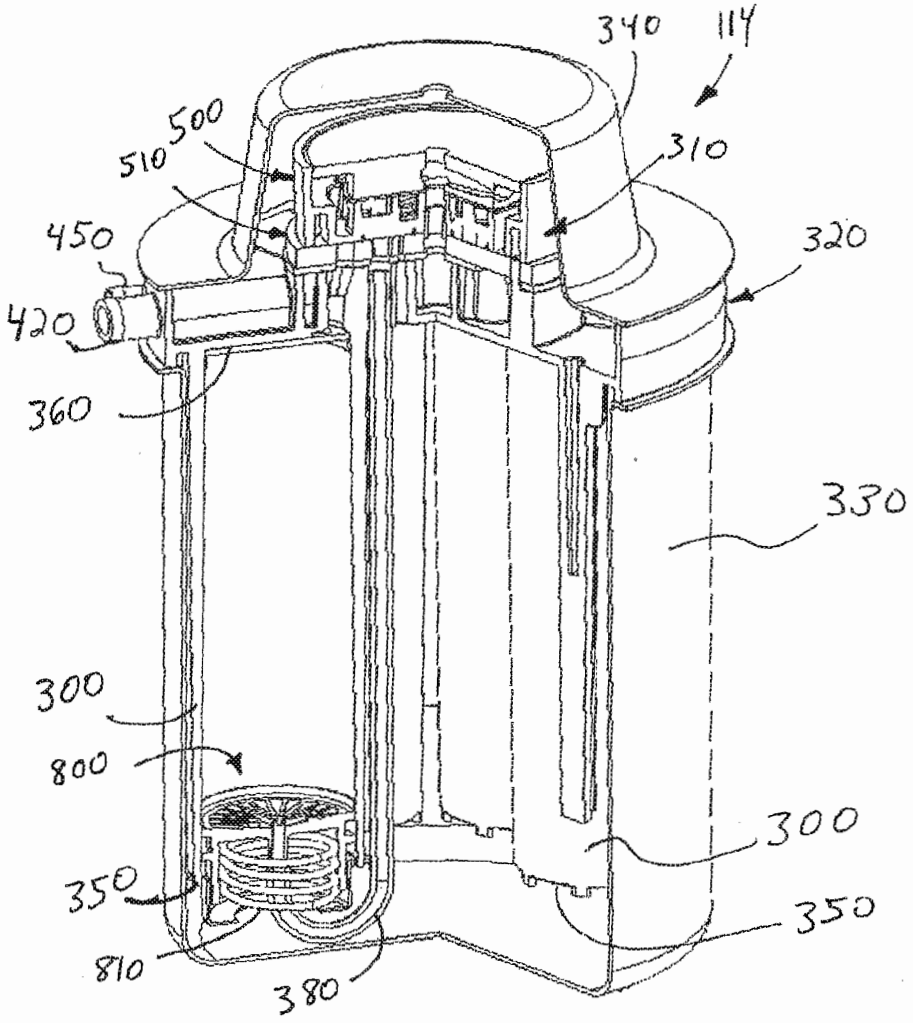

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Figure 3A

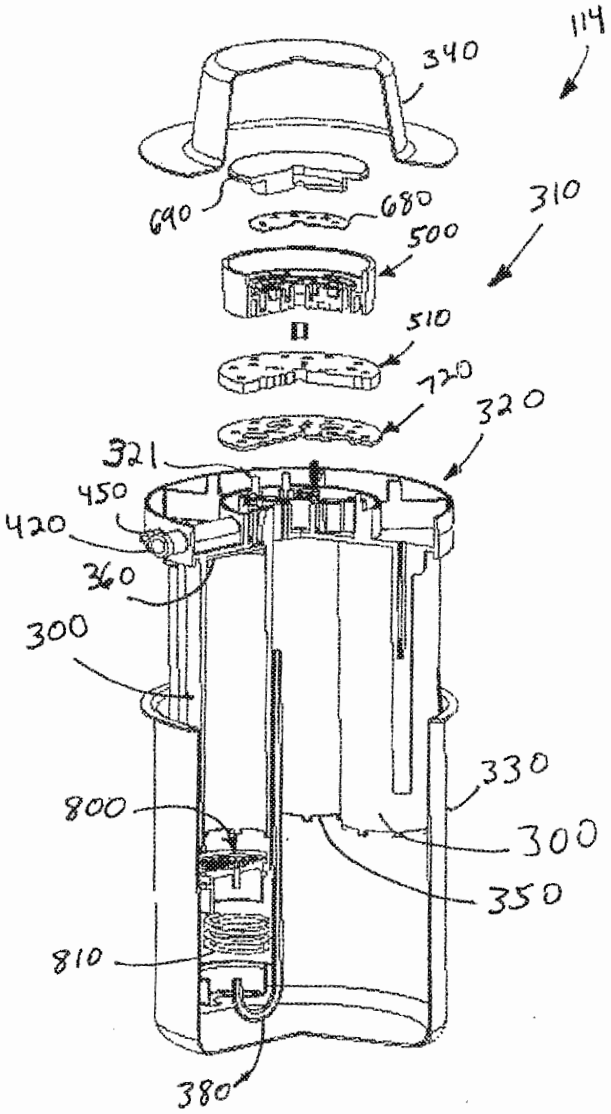

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Figure 3B

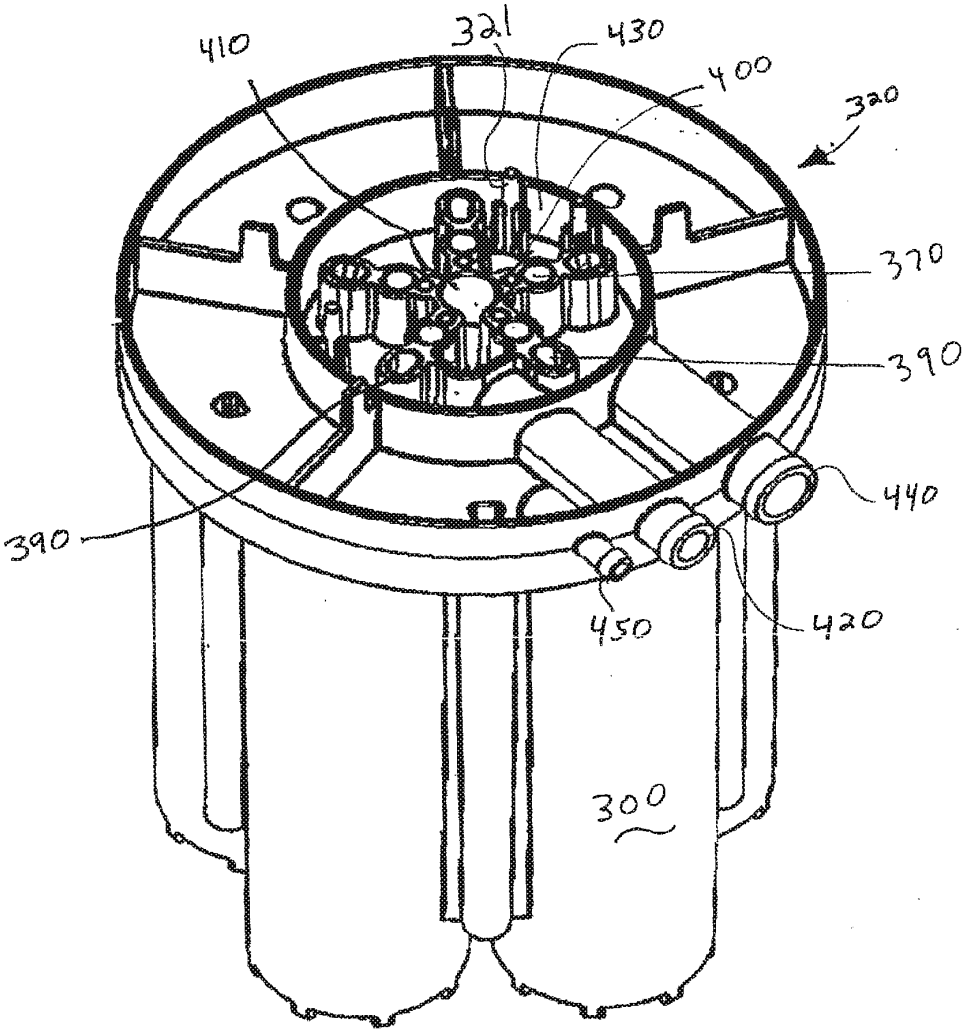



Figure 4

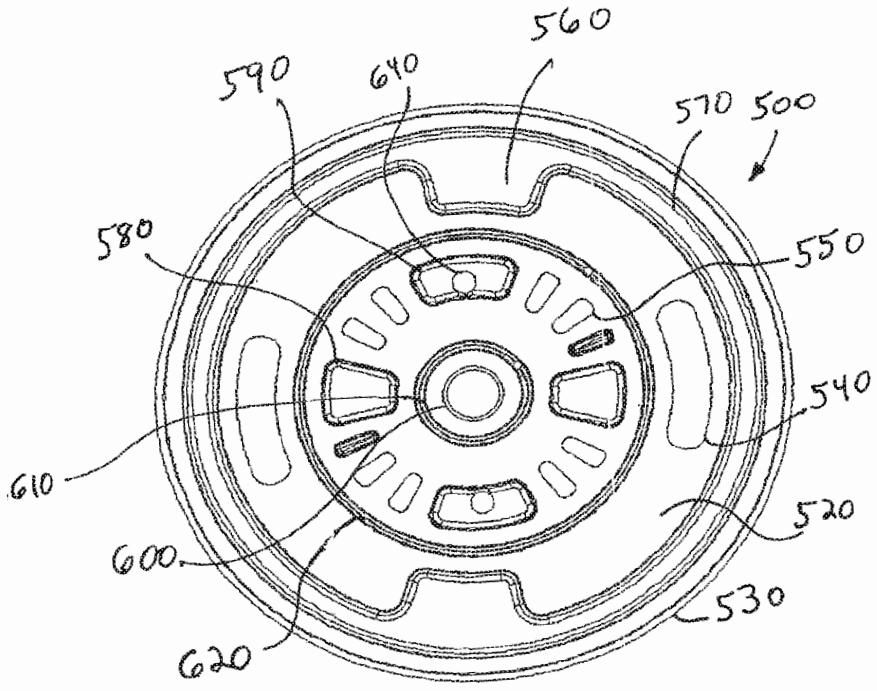



Figure 5A

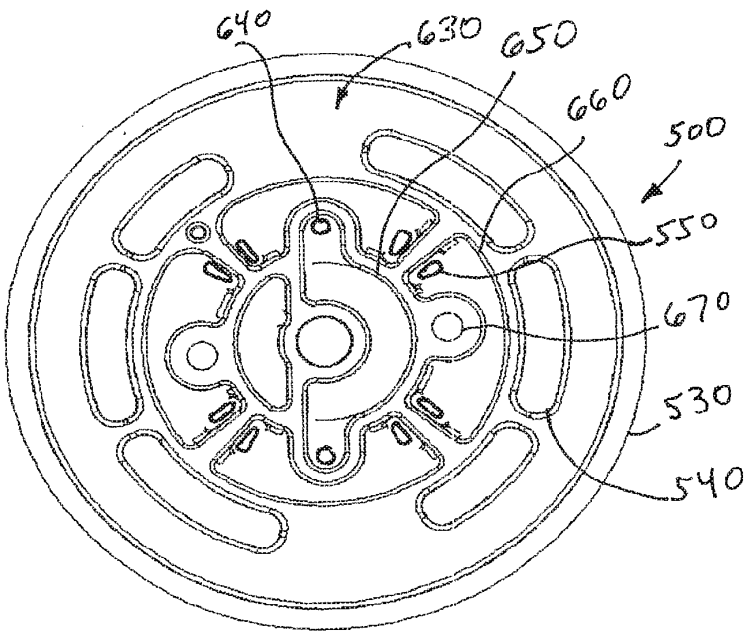



Figure 5B

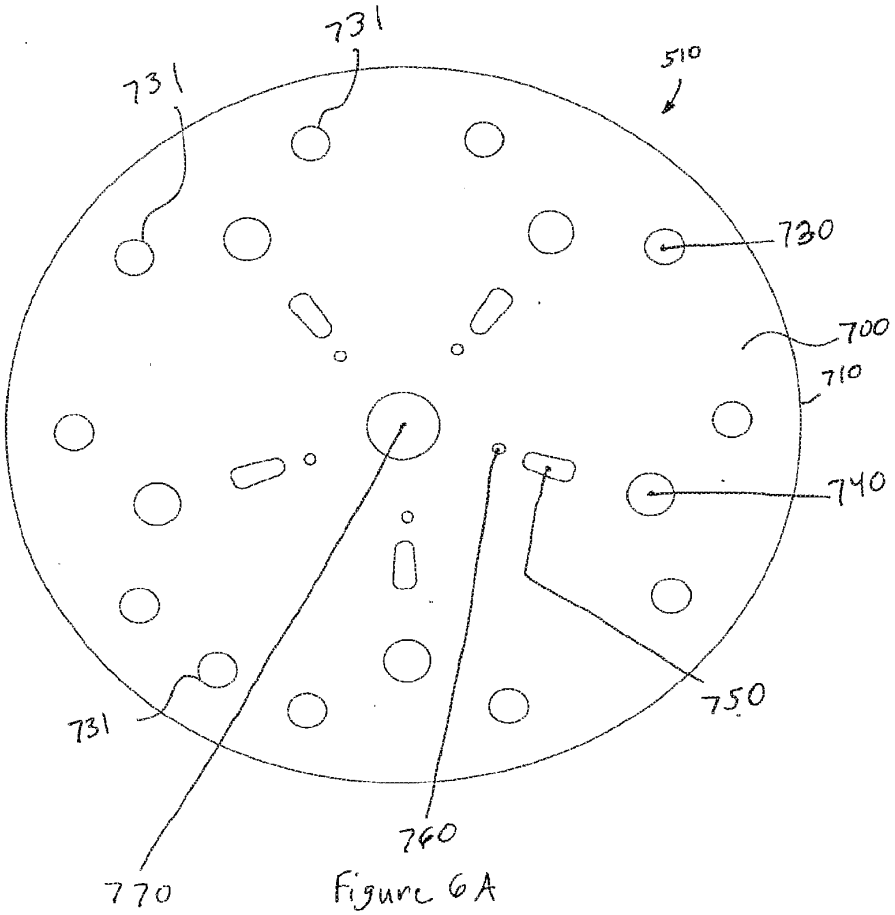



Figure 6A

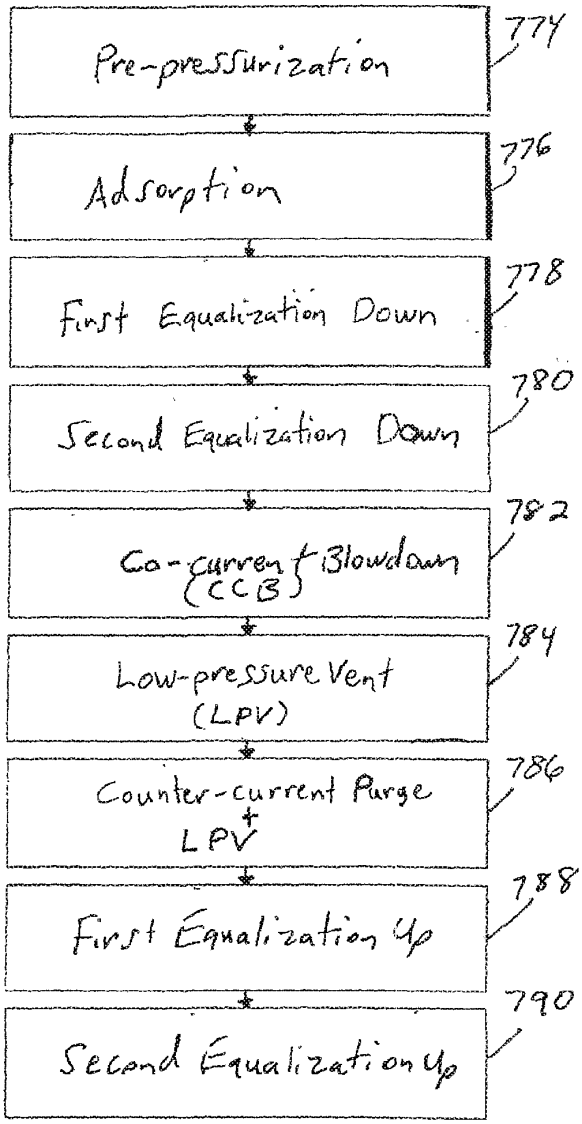

Pre-pressurization
774
Adsorption
776
First Equalization Down
778
Second Equalization Down
780
Co-current Blowdown (CCB)
782
Low-pressure Vent (LPV)
784
Counter-current Purge + LPV
786
First Equalization Up
788
Second Equalization Up
790

Figure 6B

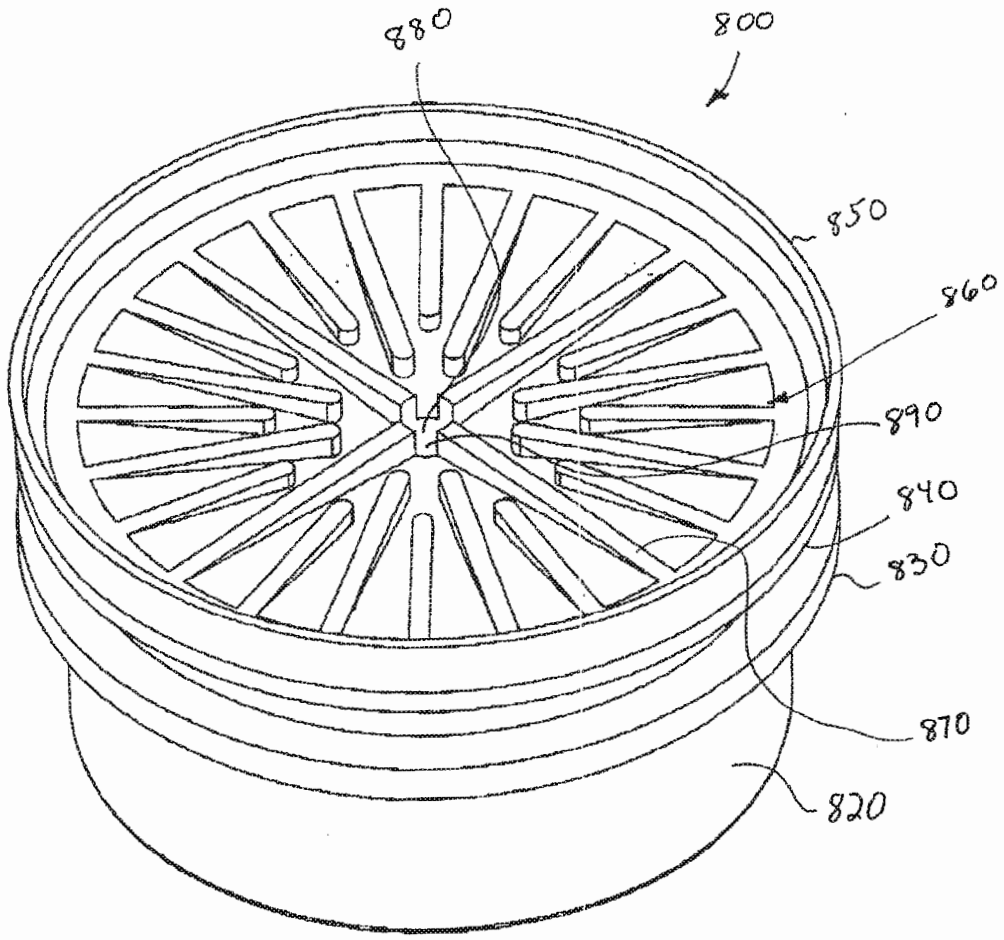



Figure 7A

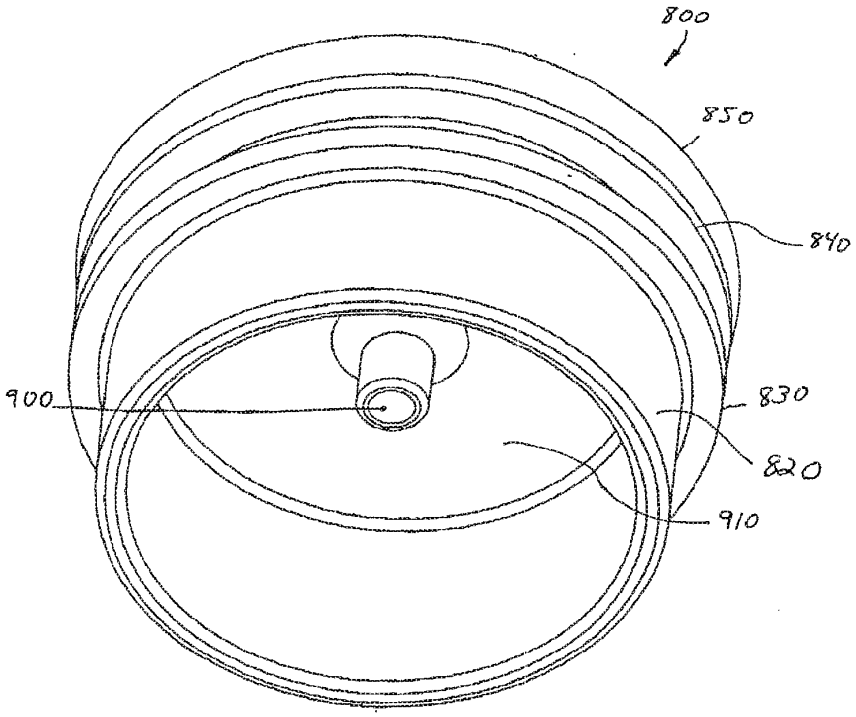



Figure 7B

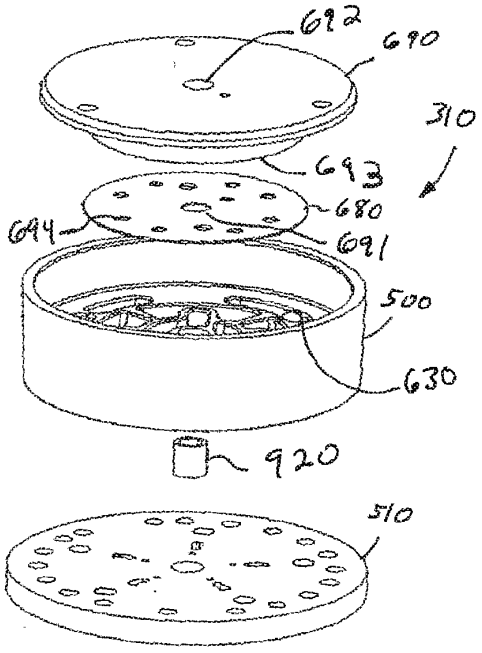



Figure 8A

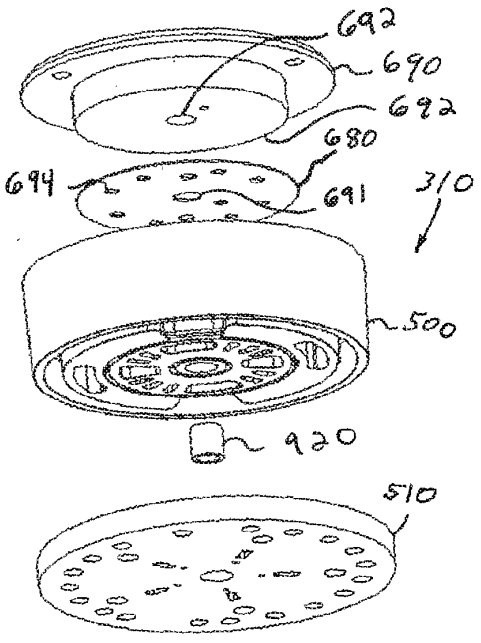



Figure 8B

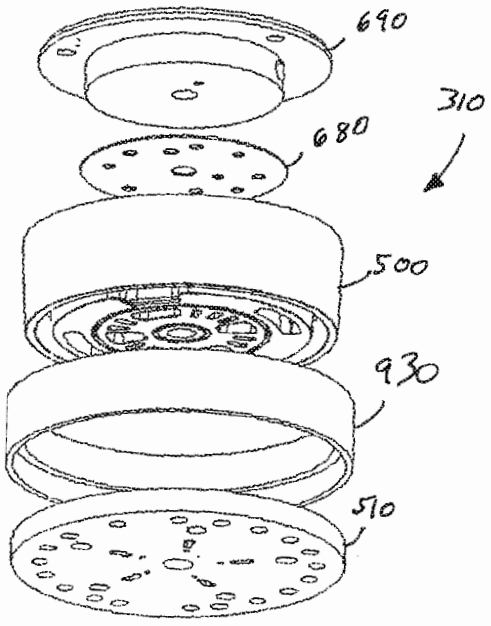



Figure 9A

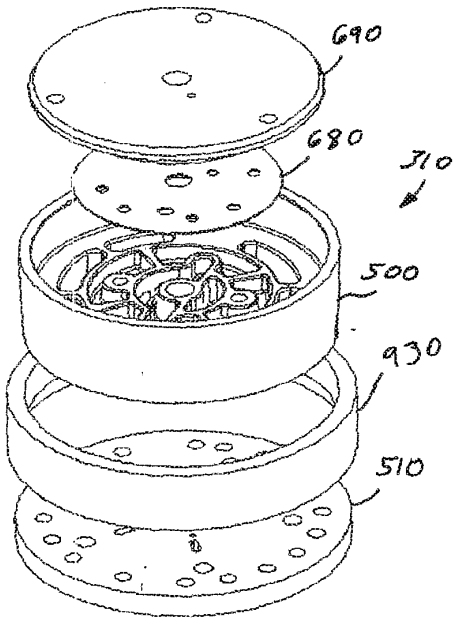



Figure 9B

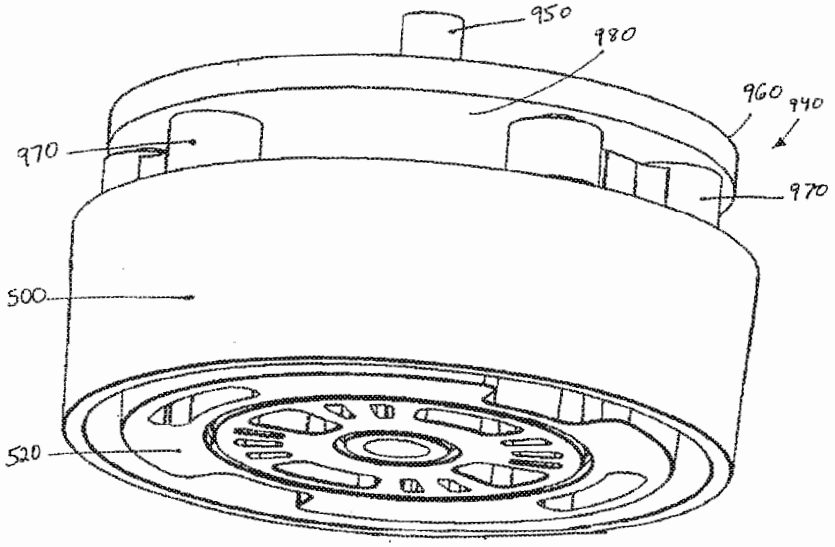



Figure 10A

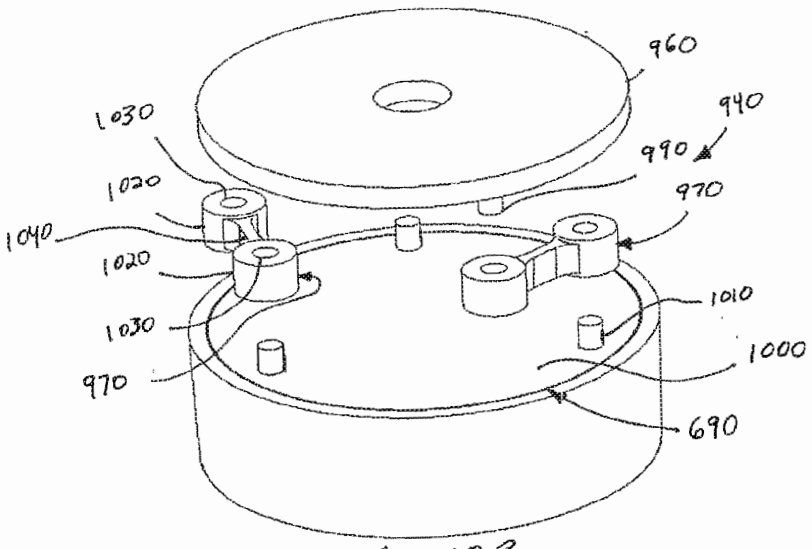



Figure 10 B

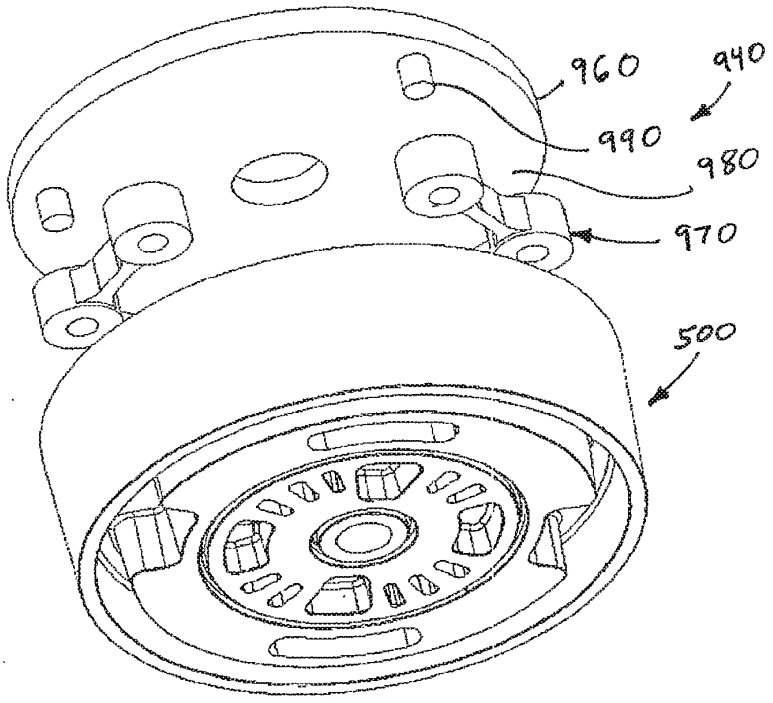



Figure 10C

| Product Flow Rate (slpm) | Recovery (%) | Product Purity (%) | Feed Pressure (psig) | Vacuum Pressure (psig) | Flow Rate (slpm) | | Adiabatic Work (W)* | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | Comp. | Vacuum | Comp | Vacuum | Total |
| 3.0 | 55.1 | 91.7 | 7.7 | -7.0 | 23.8 | 20.8 | 17.9 | 20.6 | 38.5 |
| 3.0 | 53.6 | 90.4 | 7.2 | -7.0 | 24.1 | 21.1 | 17.1 | 21.2 | 38.3 |
| 3.0 | 53.6 | 90.9 | 7.2 | -7.0 | 24.2 | 21.2 | 17.2 | 21.2 | 38.4 |
| 1.0 | 54.6 | 91.6 | 9.0 | -7.0 | 8.0 | 7.0 | 6.9 | 7.0 | 13.9 |
| 2.0 | 57.2 | 92.0 | 10.6 | -7.0 | 15.3 | 13.3 | 15.2 | 13.3 | 28.4 |
| 3.0 | 54.9 | 92.3 | 9.6 | -7.5 | 24.0 | 21.0 | 21.8 | 22.9 | 44.7 |
| 2.8 | 51.7 | 93.6 | 10.1 | -7.4 | 24.1 | 21.3 | 22.9 | 22.8 | 45.8 |
| 2.5 | 46.3 | 94.5 | 10.3 | -7.5 | 24.3 | 21.8 | 23.5 | 23.8 | 47.2 |
| 3.2 | 56.3 | 90.7 | 9.9 | -7.5 | 24.6 | 21.4 | 23.0 | 23.3 | 46.3 |
| 3.4 | 58.4 | 88.0 | 9.9 | -7.0 | 24.4 | 21.0 | 22.8 | 20.9 | 43.7 |
| 2.0 | 46.9 | 94.7 | 12.1 | -7.1 | 19.3 | 17.3 | 21.3 | 17.5 | 38.8 |
| 2.2 | 51.3 | 94.3 | 11.9 | -7.2 | 19.3 | 17.1 | 21.0 | 17.6 | 38.6 |
| 2.4 | 55.6 | 93.6 | 11.7 | -7.3 | 19.3 | 16.9 | 20.8 | 17.7 | 38.5 |
| 2.5 | 57.9 | 92.7 | 11.1 | -7.3 | 19.1 | 16.6 | 19.7 | 17.4 | 37.1 |
| 2.8 | 61.8 | 89.0 | 11.3 | -7.2 | 19.2 | 16.4 | 20.0 | 16.9 | 37.0 |
| 1.5 | 58.1 | 94.3 | 10.8 | -7.2 | 11.2 | 9.8 | 11.3 | 10.1 | 21.4 |
| 1.5 | 59.9 | 94.0 | 10.8 | -7.2 | 11.2 | 9.7 | 11.3 | 10.0 | 21.3 |
| 1.6 | 67.3 | 90.1 | 10.4 | -7.3 | 11.2 | 9.4 | 10.9 | 9.9 | 20.8 |
| 1.0 | 64.0 | 94.1 | 9.0 | -7.2 | 7.0 | 6.0 | 6.1 | 6.2 | 12.3 |
| 1.2 | 70.4 | 86.3 | 8.5 | -7.4 | 7.0 | 5.8 | 5.8 | 6.2 | 12.0 |
| 1.1 | 68.1 | 90.3 | 8.7 | -7.3 | 7.0 | 5.9 | 5.8 | 6.2 | 12.0 |
| 2.5 | 45.5 | 94.4 | 9.5 | -6.1 | 24.7 | 22.2 | 22.3 | 18.8 | 41.1 |
| 2.8 | 50.5 | 93.7 | 9.7 | -6.2 | 24.8 | 22.0 | 22.8 | 18.9 | 41.7 |
| 3.0 | 54.1 | 92.5 | 10.1 | -6.3 | 24.4 | 21.4 | 23.2 | 18.6 | 41.8 |
| 3.2 | 56.3 | 90.7 | 9.9 | -6.4 | 24.6 | 21.4 | 22.9 | 18.8 | 41.7 |
| 3.5 | 59.2 | 87.4 | 9.7 | -6.4 | 24.6 | 21.1 | 22.7 | 18.8 | 41.5 |
| 1.0 | 46.0 | 90.9 | 5.8 | -5.3 | 9.4 | 8.4 | 5.5 | 5.9 | 11.5 |
| 2.0 | 47.8 | 90.5 | 6.7 | -5.4 | 18.0 | 16.0 | 12.0 | 11.6 | 23.6 |
| 3.0 | 48.2 | 90.7 | 10.1 | -5.6 | 26.9 | 23.9 | 25.6 | 17.9 | 43.5 |
| 1.0 | 52.3 | 92.3 | 5.7 | -6.1 | 8.4 | 7.4 | 4.9 | 6.1 | 11.0 |
| 1.0 | 51.3 | 91.1 | 5.3 | -6.0 | 8.5 | 7.5 | 4.6 | 6.2 | 10.8 |
| 2.0 | 51.6 | 92.4 | 7.3 | -5.9 | 17.1 | 15.1 | 12.2 | 12.2 | 24.4 |
| 2.0 | 52.4 | 91.5 | 7.1 | -5.9 | 16.6 | 14.6 | 11.7 | 11.8 | 23.5 |
| 3.0 | 48.6 | 89.9 | 8.5 | -5.8 | 26.4 | 23.4 | 21.8 | 18.3 | 40.1 |
| 3.0 | 48.9 | 90.7 | 8.8 | -5.8 | 26.5 | 23.5 | 22.6 | 18.4 | 40.9 |

Figure 11

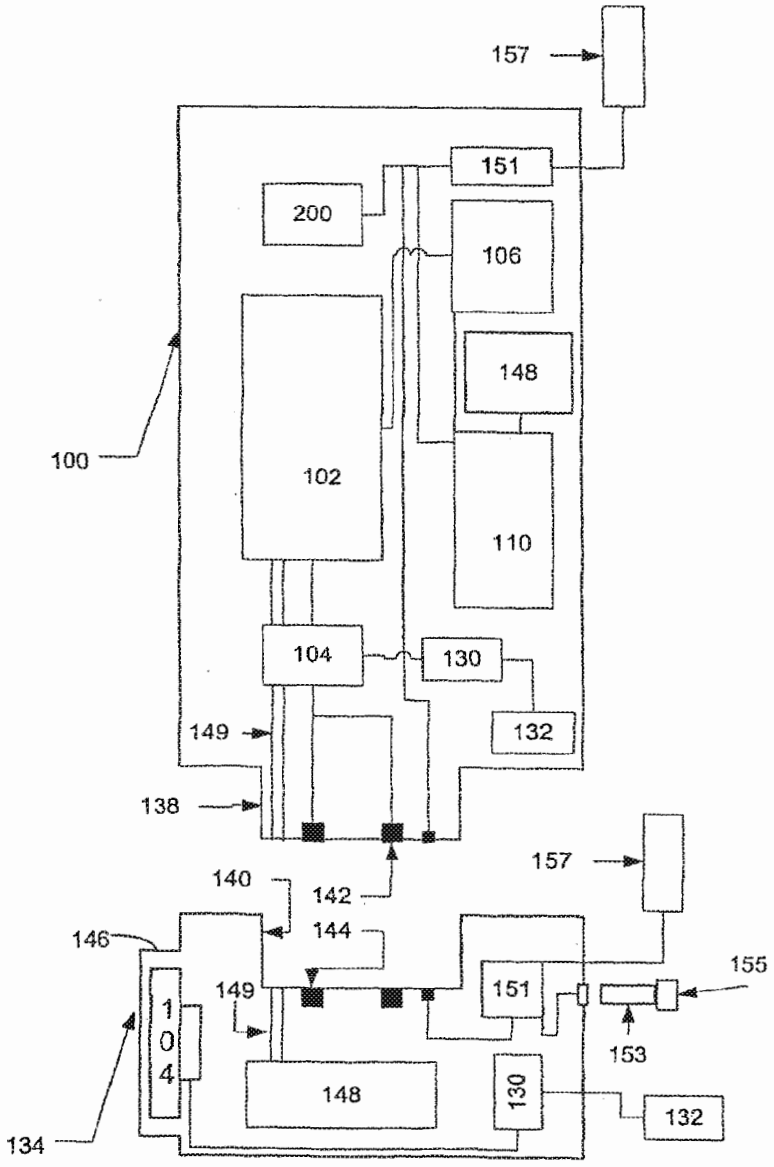

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Figure 12

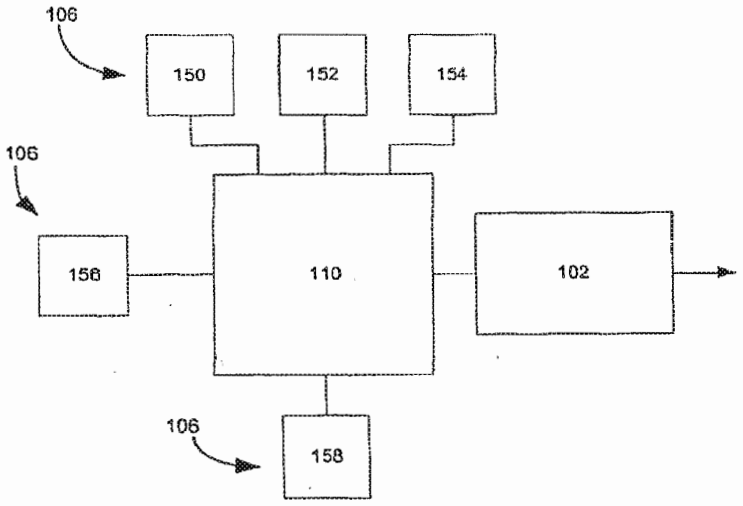



Figure 13

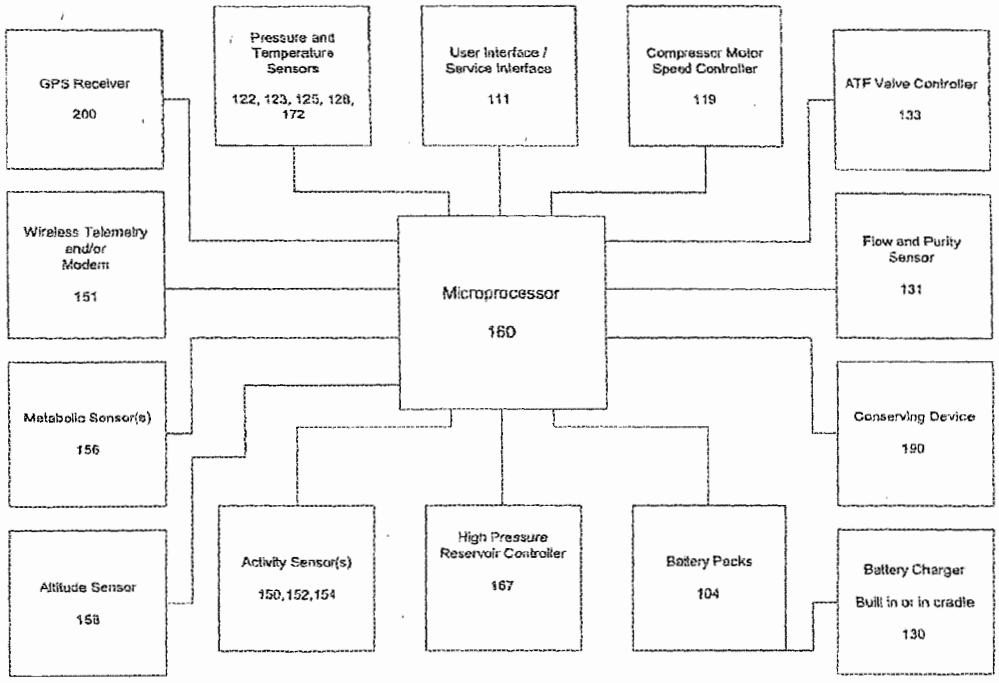



Figure 14

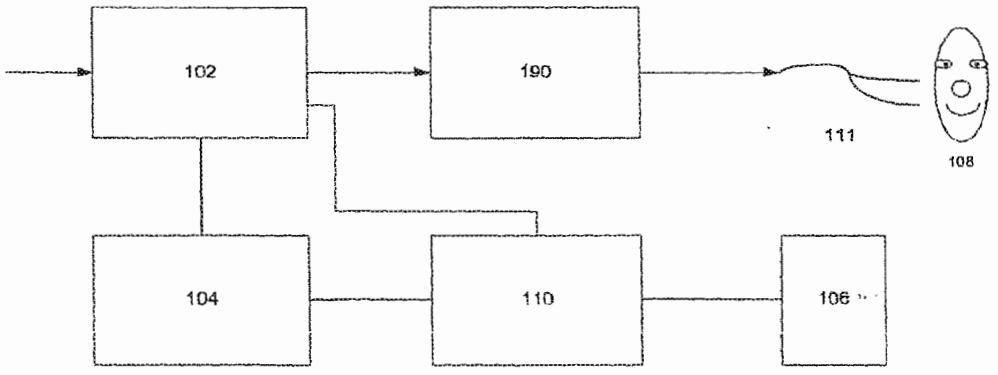



Figure 15

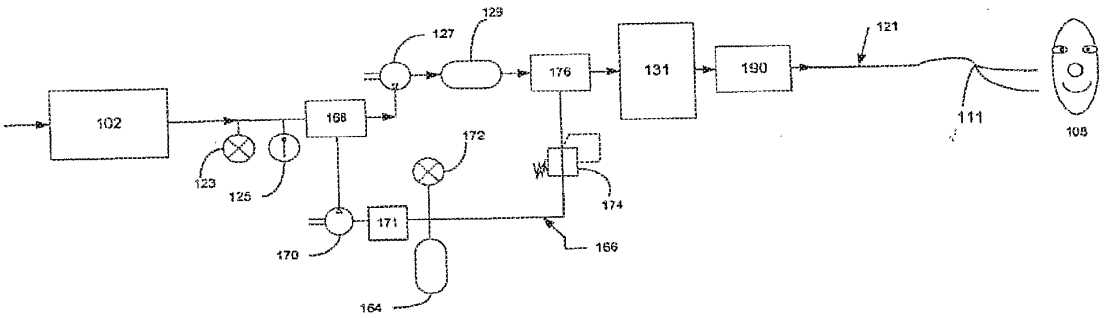

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Figure 16

# INTERNATIONAL SEARCH REPORT

International application No.
PCT/US03/13496

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(7) : A62B 7/10, 9/00, 19/00, 23/02; A61M 16/00; F24J 3/00; G05B 1/00
US CL : 128/201.25, 204.17, 205.11, 205.18

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)
U.S. : 128/201.25, 204.17, 205.11, 205.18

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 practicable, 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. |
|---|---|---|
| X | US 5, 268, 021 A (HILL et al) 7 December 1993, see entire document | 26 |
| X | US 5, 366, 541 (HILL et al) 22 November 1994, see entire document | 26 |
| X | US 5,827, 358 (KULISH et al) 27 October 1998, see entire document | 26 |
| A | US 5, 882, 380 A (SIRCAR) 16 march 1999, see entire document | 1-26 |
| A | US 6, 056, 804 A (KEEFER et al) 02 May 2000, see entire document | 1-26 |
| A | US 4, 576, 616 A (MOTTRAM et al) 18 March 1986, see entire document | 1-26 |

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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Date of the actual completion of the international search
15 September 2003 (15.09.2003)

Date of mailing of the international search report
02 OCT 2003

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Alexandria, Virginia 22313-1450
Facsimile No. (703)305-3230

Authorized officer
Weilun Lo
Telephone No. 308-0858

Form PCT/ISA/210 (second sheet) (July 1998)

PTO/SB/08a (01-10)
Approved for use through 07/31/2012. OMB 0651-0031
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| **INFORMATION DISCLOSURE STATEMENT BY APPLICANT** ( **Not for submission under 37 CFR 1.99)** | | |
|---|---|---|
| | Application Number | |
| | Filing Date | |
| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

| **U.S.PATENTS** | | | | | | Remove |
|---|---|---|---|---|---|---|
| Examiner Initial* | Cite No | Patent Number | Kind Code[1] | Issue Date | Name of Patentee or Applicant of cited Document | Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear |
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| | Application Number | |
| | Filing Date | |
| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

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|---|---|---|
| | Application Number | |
| | Filing Date | |
| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

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| INFORMATION DISCLOSURE STATEMENT BY APPLICANT ( Not for submission under 37 CFR 1.99) | | |
|---|---|---|
| | Application Number | |
| | Filing Date | |
| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

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|---|---|---|---|---|---|---|
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| INFORMATION DISCLOSURE STATEMENT BY APPLICANT ( Not for submission under 37 CFR 1.99) | | |
|---|---|---|
| | Application Number | |
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| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

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|---|---|---|
| | Application Number | |
| | Filing Date | |
| | First Named Inventor | Theodore W. Jagger |
| | Art Unit | |
| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

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| | Attorney Docket Number | 67853-012 PUS4 |

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| INFORMATION DISCLOSURE STATEMENT BY APPLICANT ( Not for submission under 37 CFR 1.99) | | |
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| | Examiner Name | |
| | Attorney Docket Number | 67853-012 PUS4 |

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| | 1 | 20030106430 | A1 | 2003-06-12 | Dee et al. | |
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| INFORMATION DISCLOSURE STATEMENT BY APPLICANT ( Not for submission under 37 CFR 1.99) | | |
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| | First Named Inventor | Theodore W. Jagger |
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| | Attorney Docket Number | 67853-012 PUS4 |

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| | Attorney Docket Number | 67853-012 PUS4 |

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| | Attorney Docket Number | 67853-012 PUS4 |

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| | 1 | Inter Partes Reexamination Certificate (1210th) US 7,841,343 C1, issued December 7, 2015, Title: Systems and Methods for Delivering Therapeutic Gas to Patients, Inventor: Deane et al. Assignee: Inogen, Inc. Reexamination Request No. 95/001,885, Mar. 1, 2012. | |
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A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

| Signature | /Timothy J. Murphy/ | Date (YYYY-MM-DD) | 2020-05-14 |
|---|---|---|---|
| Name/Print | Timothy J. Murphy | Registration Number | 64613 |

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| **Application Number:** | |
| **Filing Date:** | |
| **Title of Invention:** | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR |
| **First Named Inventor/Applicant Name:** | Theodore W. Jagger |
| **Filer:** | Timothy J. Murphy/Carolyn Hannenberg |
| **Attorney Docket Number:** | 67853-012 PUS4 |

Filed as Small Entity

**Filing Fees for Utility under 35 USC 111(a)**

| Description | Fee Code | Quantity | Amount | Sub-Total in USD($) |
|---|---|---|---|---|
| **Basic Filing:** | | | | |
| UTILITY FILING FEE (ELECTRONIC FILING) | 4011 | 1 | 75 | 75 |
| UTILITY SEARCH FEE | 2111 | 1 | 330 | 330 |
| UTILITY EXAMINATION FEE | 2311 | 1 | 380 | 380 |
| **Pages:** | | | | |
| **Claims:** | | | | |
| **Miscellaneous-Filing:** | | | | |
| **Petition:** | | | | |
| **Patent-Appeals-and-Interference:** | | | | |

| Description | Fee Code | Quantity | Amount | Sub-Total in USD($) |
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| **Post-Allowance-and-Post-Issuance:** | | | | |
| **Extension-of-Time:** | | | | |
| **Miscellaneous:** | | | | |
| | | **Total in USD ($)** | | **785** |

| Electronic Acknowledgement Receipt | |
|---|---|
| **EFS ID:** | 39439014 |
| **Application Number:** | 15931716 |
| **International Application Number:** | |
| **Confirmation Number:** | 4948 |
| **Title of Invention:** | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR |
| **First Named Inventor/Applicant Name:** | Theodore W. Jagger |
| **Customer Number:** | 26096 |
| **Filer:** | Timothy J. Murphy/Carolyn Hannenberg |
| **Filer Authorized By:** | Timothy J. Murphy |
| **Attorney Docket Number:** | 67853-012 PUS4 |
| **Receipt Date:** | 14-MAY-2020 |
| **Filing Date:** | |
| **Time Stamp:** | 11:18:17 |
| **Application Type:** | Utility under 35 USC 111(a) |

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| 1 | Application Data Sheet | ADS_012PUS4.PDF | 1793767<br>d8ac28fead6c970547ce4a278a6deece771ac269 | no | 9 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 2 | | Application_012PUS4.pdf | 230917<br>61ff6ea969a91a4a99399e60d53f59a0e3e191a4 | yes | 43 |

| | Multipart Description/PDF files in .zip description | | |
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| | **Document Description** | **Start** | **End** |
| | Specification | 1 | 37 |
| | Claims | 38 | 42 |
| | Abstract | 43 | 43 |

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| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 3 | Drawings-only black and white line drawings | 012PUS4_Drawings.pdf | 1034695<br>04d244f6bf89e5acfebbfb14ee93a204a47b2d03 | no | 19 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 4 | Oath or Declaration filed | Declaration_012PUS4.pdf | 256758<br>6fe05f55de57feb0e31ca154010a025d56b6a396 | no | 4 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 5 | Power of Attorney | Power_of_Attorney_012PUS4.pdf | 337707<br>c4291394aec55f0661f652e6aad14d29a876fd3e | no | 4 |

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| **Information:** | | | | | |
| 6 | Information Disclosure Statement (IDS) Form (SB08) | US_IDS_Form_SB_08a_012PUS4_05-12-20.pdf | 617972<br>3bbe8f52438f484e52e7e54e931ae682f7278781 | no | 23 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 7 | Foreign Reference | GB2174922A.pdf | 777966<br>5655b57e08bc19c0f610e3bd27a0be3f1daa6cd9 | no | 10 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 8 | Foreign Reference | EP0368649A1.pdf | 1075030<br>9ae7e0759d3edb8b6c00bf574066d8402f0ccb87 | no | 20 |
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| **Information:** | | | | | |
| 9 | Foreign Reference | EP0760247A2.pdf | 1089723<br>c2cc2719bd2a1e7355d2f0d25a9c3edb00c0e9c0 | no | 23 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 10 | Foreign Reference | EP0860646A2.pdf | 970248<br>d0495e012cfbb93c12b1d940b706368d64284ade | no | 21 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 11 | Foreign Reference | EP0873776A2.pdf | 945964<br>284d87d7950aa35a96ea4700ad5a5822e1d158fe | no | 16 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 12 | Foreign Reference | WO9857727A1.pdf | 849814<br>ff356a6b751b19f0520965c3bf214491ec8e82ac | no | 19 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |

| | | | | | |
|---|---|---|---|---|---|
| 13 | Foreign Reference | WO9858219A1.pdf | 2528886<br>123b130d81950b899a603446 75b43c66a0fa5de5 | no | 80 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 14 | Foreign Reference | WO9943416A1.pdf | 1979422<br>9c21a68121b103d7cbd3028360e9b9cd2f253dde | no | 56 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 15 | Foreign Reference | WO9943418A1.pdf | 1934468<br>195f666d763d8eae059372dd210acc145cf0f8e4 | no | 50 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 16 | Foreign Reference | EP1125623A1.pdf | 659708<br>610b18ed0a031b193b2d28dfa45765969d1f7f05 | no | 13 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 17 | Foreign Reference | WO03092817A1.pdf | 8223104<br>3abfca331c0d145090454f196fc191728c7f1a41 | no | 69 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 18 | Non Patent Literature | Rec_5th_Oxygen_Conf_1999.pdf | 266717<br>69672667c535ee2619295f2235e160acb6a2d427 | no | 3 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 19 | Non Patent Literature | AirSep_lowers_boom_2004.pdf | 256248<br>eb07be0243a35af4f8441152755c97a0bd79fced | no | 2 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |

| | | | | | |
|---|---|---|---|---|---|
| 20 | Non Patent Literature | Bliss_1999.pdf | 402578<br>9e33fb057ec7524331d2ae8b4e7dadd46743792b | no | 7 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 21 | Information Disclosure Statement (IDS) Form (SB08) | US_IDS_Form_SB_08a_012PUS4_NFOA.pdf | 615754<br>f2d1a04d6888130c10fd448a19857930c8d7a86c | no | 18 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 22 | Non Patent Literature | 95001885_ReexamCert.pdf | 88677<br>fd1555dda835bc00a2b384bccbc5a365240fdda2 | no | 2 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 23 | Non Patent Literature | 95001886_ReExamCert.pdf | 41534<br>e4d7abb25d5cc0073c219a0f86c46b643242 43ef | no | 2 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| 24 | Fee Worksheet (SB06) | fee-info.pdf | 35467<br>9ebfc5b1f50206d1d580ee3dc5444cb923b728c4 | no | 2 |
| **Warnings:** | | | | | |
| **Information:** | | | | | |
| | | **Total Files Size (in bytes):** | 27013124 | | |

**This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.**

**New Applications Under 35 U.S.C. 111**

**If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.**

**National Stage of an International Application under 35 U.S.C. 371**

**If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.**

**New International Application Filed with the USPTO as a Receiving Office**

**If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.**

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| Application Data Sheet 37 CFR 1.76 | Attorney Docket Number | 67853-012 PUS4 |
|---|---|---|
| | Application Number | |
| Title of Invention | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR | |

The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76.
This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.

## Secrecy Order 37 CFR 5.2:

☐ Portions or all of the application associated with this Application Data Sheet may fall under a Secrecy Order pursuant to 37 CFR 5.2 (Paper filers only. Applications that fall under Secrecy Order may not be filed electronically.)

## Inventor Information:

**Inventor 1** Remove

**Legal Name**

| Prefix | Given Name | Middle Name | Family Name | Suffix |
|---|---|---|---|---|
| | Theodore | W. | Jagger | |

**Residence Information (Select One)** ● US Residency Non US Residency Active US Military Service

| City | State/Province | Country of Residence |
|---|---|---|
| White Bear Lake | MN | US |

**Mailing Address of Inventor:**

| | |
|---|---|
| Address 1 | 5743 Birch Road |
| Address 2 | |

| City | State/Province | Postal Code | Country |
|---|---|---|---|
| White Bear Lake | MN | 55110 | US |

**Inventor 2** Remove

**Legal Name**

| Prefix | Given Name | Middle Name | Family Name | Suffix |
|---|---|---|---|---|
| | Nicholas | P. | Van Brunt | |

**Residence Information (Select One)** ◉ US Residency Non US Residency Active US Military Service

| City | State/Province | Country of Residence |
|---|---|---|
| White Bear Lake | MN | US |

**Mailing Address of Inventor:**

| | |
|---|---|
| Address 1 | One Bald Eagle Island |
| Address 2 | |

| City | State/Province | Postal Code | Country |
|---|---|---|---|
| White Bear Lake | MN | 55110 | US |

**Inventor 3** Remove

**Legal Name**

| Prefix | Given Name | Middle Name | Family Name | Suffix |
|---|---|---|---|---|
| | John | A. | Kivisto | |

**Residence Information (Select One)** ◉ US Residency Non US Residency Active US Military Service

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| Application Data Sheet 37 CFR 1.76 | Attorney Docket Number | 67853-012 PUS4 |
|---|---|---|
| | Application Number | |
| Title of Invention | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR | |

| City | Oak Grove | State/Province | MN | Country of Residence | US |
|---|---|---|---|---|---|

**Mailing Address of Inventor:**

| Address 1 | 3367 221st Avenue NW | | |
|---|---|---|---|
| Address 2 | | | |
| City | Oak Grove | State/Province | MN |
| Postal Code | 55303 | Country | US |

Inventor 4 Remove

**Legal Name**

| Prefix | Given Name | Middle Name | Family Name | Suffix |
|---|---|---|---|---|
| | Perry | B. | Lonnes | |

**Residence Information (Select One)** ◉ US Residency Non US Residency Active US Military Service

| City | White Bear Lake | State/Province | MN | Country of Residence | US |
|---|---|---|---|---|---|

**Mailing Address of Inventor:**

| Address 1 | 5300 East Bald Eagle Boulevard | | |
|---|---|---|---|
| Address 2 | | | |
| City | White Bear Lake | State/Province | MN |
| Postal Code | 55110 | Country | US |

All Inventors Must Be Listed - Additional Inventor Information blocks may be generated within this form by selecting the **Add** button. Add

## Correspondence Information:

**Enter either Customer Number or complete the Correspondence Information section below.**
**For further information see 37 CFR 1.33(a).**

☐ **An Address is being provided for the correspondence Information of this application.**

| Customer Number | 26096 |
|---|---|
| Email Address | Add Email Remove Email |

## Application Information:

| Title of the Invention | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR | | |
|---|---|---|---|
| Attorney Docket Number | 67853-012 PUS4 | Small Entity Status Claimed | ☒ |
| Application Type | Nonprovisional | | |
| Subject Matter | Utility | | |
| Total Number of Drawing Sheets (if any) | 19 | Suggested Figure for Publication (if any) | |

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| Application Data Sheet 37 CFR 1.76 | Attorney Docket Number | 67853-012 PUS4 |
|---|---|---|
| | Application Number | |
| Title of Invention | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR | |

## Filing By Reference:

Only complete this section when filing an application by reference under 35 U.S.C. 111(c) and 37 CFR 1.57(a). Do not complete this section if application papers including a specification and any drawings are being filed. Any domestic benefit or foreign priority information must be provided in the appropriate section(s) below (i.e., "Domestic Benefit/National Stage Information" and "Foreign Priority Information").

For the purposes of a filing date under 37 CFR 1.53(b), the description and any drawings of the present application are replaced by this reference to the previously filed application, subject to conditions and requirements of 37 CFR 1.57(a).

| Application number of the previously filed application | Filing date (YYYY-MM-DD) | Intellectual Property Authority or Country |
|---|---|---|
| | | |

## Publication Information:

☐ Request Early Publication (Fee required at time of Request 37 CFR 1.219)

☐ **Request Not to Publish.** I hereby request that the attached application not be published under 35 U.S.C. 122(b) and certify that the invention disclosed in the attached application **has not and will not** be the subject of an application filed in another country, or under a multilateral international agreement, that requires publication at eighteen months after filing.

## Representative Information:

Representative information should be provided for all practitioners having a power of attorney in the application. Providing this information in the Application Data Sheet does not constitute a power of attorney in the application (see 37 CFR 1.32).
Either enter Customer Number or complete the Representative Name section below. If both sections are completed the customer Number will be used for the Representative Information during processing.

| Please Select One: | ● Customer Number | US Patent Practitioner | ○ Limited Recognition (37 CFR 11.9) |
|---|---|---|---|
| Customer Number | | | |

## Domestic Benefit/National Stage Information:

This section allows for the applicant to either claim benefit under 35 U.S.C. 119(e), 120, 121, 365(c), or 386(c) or indicate National Stage entry from a PCT application. Providing benefit claim information in the Application Data Sheet constitutes the specific reference required by 35 U.S.C. 119(e) or 120, and 37 CFR 1.78.
When referring to the current application, please leave the "Application Number" field blank.

| Prior Application Status | Pending | | Remove |
|---|---|---|---|
| Application Number | Continuity Type | Prior Application Number | Filing or 371(c) Date (YYYY-MM-DD) |
| | Continuation of | 14193504 | 2014-02-28 |

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| **Application Data Sheet 37 CFR 1.76** | Attorney Docket Number | 67853-012 PUS4 |
|---|---|---|
| | Application Number | |
| Title of Invention | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR | |

| Prior Application Status | Abandoned | | Remove |
|---|---|---|---|
| Application Number | Continuity Type | Prior Application Number | Filing or 371(c) Date (YYYY-MM-DD) |
| 14193504 | Continuation of | 13795977 | 2013-03-12 |
| Prior Application Status | Abandoned | | Remove |
| Application Number | Continuity Type | Prior Application Number | Filing or 371(c) Date (YYYY-MM-DD) |
| 13795977 | Continuation of | 13096362 | 2011-04-28 |
| Prior Application Status | Patented | | Remove |

| Application Number | Continuity Type | Prior Application Number | Filing Date (YYYY-MM-DD) | Patent Number | Issue Date (YYYY-MM-DD) |
|---|---|---|---|---|---|
| 13096362 | Continuation of | 11054341 | 2005-02-09 | 7954490 | 2011-06-07 |

Additional Domestic Benefit/National Stage Data may be generated within this form by selecting the **Add** button. Add

## Foreign Priority Information:

This section allows for the applicant to claim priority to a foreign application. Providing this information in the application data sheet constitutes the claim for priority as required by 35 U.S.C. 119(b) and 37 CFR 1.55. When priority is claimed to a foreign application that is eligible for retrieval under the priority document exchange program (PDX)[i] the information will be used by the Office to automatically attempt retrieval pursuant to 37 CFR 1.55(i)(1) and (2). Under the PDX program, applicant bears the ultimate responsibility for ensuring that a copy of the foreign application is received by the Office from the participating foreign intellectual property office, or a certified copy of the foreign priority application is filed, within the time period specified in 37 CFR 1.55(g)(1).

Remove

| Application Number | Country[i] | Filing Date (YYYY-MM-DD) | Access Code[i] (if applicable) |
|---|---|---|---|
| | | | |

Additional Foreign Priority Data may be generated within this form by selecting the **Add** button. Add

## Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications

☐ This application (1) claims priority to or the benefit of an application filed before March 16, 2013 and (2) also contains, or contained at any time, a claim to a claimed invention that has an effective filing date on or after March 16, 2013.
NOTE: By providing this statement under 37 CFR 1.55 or 1.78, this application, with a filing date on or after March 16, 2013, will be examined under the first inventor to file provisions of the AIA.

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| Application Data Sheet 37 CFR 1.76 | Attorney Docket Number | 67853-012 PUS4 |
|---|---|---|
| | Application Number | |
| Title of Invention | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR | |

## Authorization or Opt-Out of Authorization to Permit Access:

When this Application Data Sheet is properly signed and filed with the application, applicant has provided written authority to permit a participating foreign intellectual property (IP) office access to the instant application-as-filed (see paragraph A in subsection 1 below) and the European Patent Office (EPO) access to any search results from the instant application (see paragraph B in subsection 1 below).

Should applicant choose not to provide an authorization identified in subsection 1 below, applicant **must opt-out** of the authorization by checking the corresponding box A or B or both in subsection 2 below.

**NOTE**: This section of the Application Data Sheet is **ONLY** reviewed and processed with the **INITIAL** filing of an application. After the initial filing of an application, an Application Data Sheet cannot be used to provide or rescind authorization for access by a foreign IP office(s). Instead, Form PTO/SB/39 or PTO/SB/69 must be used as appropriate.

**1. Authorization to Permit Access by a Foreign Intellectual Property Office(s)**

**A. Priority Document Exchange (PDX)** - Unless box A in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the European Patent Office (EPO), the Japan Patent Office (JPO), the Korean Intellectual Property Office (KIPO), the State Intellectual Property Office of the People's Republic of China (SIPO), the World Intellectual Property Organization (WIPO), and any other foreign intellectual property office participating with the USPTO in a bilateral or multilateral priority document exchange agreement in which a foreign application claiming priority to the instant patent application is filed, access to: (1) the instant patent application-as-filed and its related bibliographic data, (2) any foreign or domestic application to which priority or benefit is claimed by the instant application and its related bibliographic data, and (3) the date of filing of this Authorization. See 37 CFR 1.14(h)(1).

**B. Search Results from U.S. Application to EPO** - Unless box B in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the EPO access to the bibliographic data and search results from the instant patent application when a European patent application claiming priority to the instant patent application is filed. See 37 CFR 1.14(h)(2).

The applicant is reminded that the EPO's Rule 141(1) EPC (European Patent Convention) requires applicants to submit a copy of search results from the instant application without delay in a European patent application that claims priority to the instant application.

**2. Opt-Out of Authorizations to Permit Access by a Foreign Intellectual Property Office(s)**

☐ A. Applicant **DOES NOT** authorize the USPTO to permit a participating foreign IP office access to the instant application-as-filed. If this box is checked, the USPTO will not be providing a participating foreign IP office with any documents and information identified in subsection 1A above.

☐ B. Applicant **DOES NOT** authorize the USPTO to transmit to the EPO any search results from the instant patent application. If this box is checked, the USPTO will not be providing the EPO with search results from the instant application.

**NOTE:** Once the application has published or is otherwise publicly available, the USPTO may provide access to the application in accordance with 37 CFR 1.14.

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| Application Data Sheet 37 CFR 1.76 | Attorney Docket Number | 67853-012 PUS4 |
|---|---|---|
| | Application Number | |
| Title of Invention | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR | |

## Applicant Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

**Applicant 1** Remove

If the applicant is the inventor (or the remaining joint inventor or inventors under 37 CFR 1.45), this section should not be completed. The information to be provided in this section is the name and address of the legal representative who is the applicant under 37 CFR 1.43; or the name and address of the assignee, person to whom the inventor is under an obligation to assign the invention, or person who otherwise shows sufficient proprietary interest in the matter who is the applicant under 37 CFR 1.46. If the applicant is an applicant under 37 CFR 1.46 (assignee, person to whom the inventor is obligated to assign, or person who otherwise shows sufficient proprietary interest) together with one or more joint inventors, then the joint inventor or inventors who are also the applicant should be identified in this section.

Clear

| ● Assignee | Legal Representative under 35 U.S.C. 117 | Joint Inventor |
|---|---|---|
| Person to whom the inventor is obligated to assign. | Person who shows sufficient proprietary interest | |

If applicant is the legal representative, indicate the authority to file the patent application, the inventor is:

Name of the Deceased or Legally Incapacitated Inventor:

If the Applicant is an Organization check here. ☒

| Organization Name | Vbox, Incorporated | | |
|---|---|---|---|
| **Mailing Address Information For Applicant:** | | | |
| **Address 1** | 2342 East County Road J | | |
| Address 2 | | | |
| **City** | White Bear Lake | **State/Province** | MN |
| **Country** | US | Postal Code | 55110 |
| Phone Number | | Fax Number | |
| Email Address | | | |

Additional Applicant Data may be generated within this form by selecting the Add button. Add

## Assignee Information including Non-Applicant Assignee Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

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| **Application Data Sheet 37 CFR 1.76** | Attorney Docket Number | 67853-012 PUS4 |
|---|---|---|
| | Application Number | |
| Title of Invention | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR | |

**Assignee 1**

Complete this section if assignee information, including non-applicant assignee information, is desired to be included on the patent application publication. An assignee-applicant identified in the "Applicant Information" section will appear on the patent application publication as an applicant. For an assignee-applicant, complete this section only if identification as an assignee is also desired on the patent application publication.

Remove

If the Assignee or Non-Applicant Assignee is an Organization check here. ☐

| Prefix | **Given Name** | Middle Name | **Family Name** | Suffix |
|---|---|---|---|---|
| | | | | |

**Mailing Address Information For Assignee including Non-Applicant Assignee:**

| | | | |
|---|---|---|---|
| **Address 1** | | | |
| Address 2 | | | |
| **City** | | **State/Province** | |
| **Country** i | | Postal Code | |
| Phone Number | | Fax Number | |
| Email Address | | | |

Additional Assignee or Non-Applicant Assignee Data may be generated within this form by selecting the Add button. Add

## Signature:

Remove

**NOTE:** This Application Data Sheet must be signed in accordance with 37 CFR 1.33(b). **However, if this Application Data Sheet is submitted with the INITIAL filing of the application and either box A or B is not checked in subsection 2 of the "Authorization or Opt-Out of Authorization to Permit Access" section, then this form must also be signed in accordance with 37 CFR 1.14(c).**

This Application Data Sheet **must** be signed by a patent practitioner if one or more of the applicants is a **juristic entity** (e.g., corporation or association). If the applicant is two or more joint inventors, this form must be signed by a patent practitioner, **all** joint inventors who are the applicant, or one or more joint inventor-applicants who have been given power of attorney (e.g., see USPTO Form PTO/AIA/81) on behalf of **all** joint inventor-applicants.

See 37 CFR 1.4(d) for the manner of making signatures and certifications.

| **Signature** | /Timothy J. Murphy/ | | | Date (YYYY-MM-DD) | 2020-05-14 |
|---|---|---|---|---|---|
| First Name | Timothy J. | Last Name | Murphy | Registration Number | 64613 |

Additional Signature may be generated within this form by selecting the Add button. Add

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| **Application Data Sheet 37 CFR 1.76** | Attorney Docket Number | 67853-012 PUS4 |
|---|---|---|
| | Application Number | |
| Title of Invention | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR | |

This collection of information is required by 37 CFR 1.76. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 23 minutes to complete, including gathering, preparing, and submitting the completed application data sheet form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

# Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1 The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these records.

2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.

3 A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.

4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).

5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent CooperationTreaty.

6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).

7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.

8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.

9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

# REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR

## RELATED APPLICATIONS

**[0001]** This application is a continuation of earlier filed Application No. 14/193,504, filed February 28, 2014, which is a continuation of Application No. 13/795,977, filed March 12, 2013, which is a continuation of Application No. 13/096,362 filed April 28, 2011, which is a continuation of Application No. 11/054,341, filed February 9, 2005. The '504, '977, '362, and '341 Applications are incorporated by reference herein in their entirety.

## BACKGROUND

**[0002]** The field of this invention relates to oxygen concentrators. In particular, the invention relates to wearable oxygen concentration systems utilizing vacuum swing adsorption for creating an oxygen stream for ambulatory respiratory patients.

**[0003]** There is a need for home and ambulatory oxygen systems for use by patients. Supplemental oxygen is required for patients exhibiting symptoms from certain diseases and lung disorders; for example, pulmonary fibrosis, sarcoidosis, or occupational lung disease. For such patients, oxygen therapy is an increasingly beneficial prescription to help the patients live normal and productive lives. While not a cure for lung disease, prescriptive supplemental oxygen increases blood oxygenation, which reverses hypoxemia. Oxygen prescriptions prevent long-term effects of oxygen deficiency on organ systems, the heart, brain and kidneys. Oxygen treatment is also prescribed for Chronic Obstructive Pulmonary Disease (COPD), heart disease, AIDS, asthma, and emphysema.

**[0004]** Currently, supplemental medical oxygen for therapy is provided to a patient from high pressure gas cylinders; cryogenic liquid in vacuum-insulated containers or thermos bottles commonly called "dewars", and oxygen concentrators. Some patients require in-home oxygen only, while others require in-home as well as ambulatory oxygen depending on the prescription. The three systems are all used for in-home use. However, oxygen concentrators provide a special beneficial advantage because they do not require refilling of dewars or exchange of empty cylinders with full ones. Home oxygen concentrators, however, do have drawbacks. They consume relatively large amounts of electricity; are relatively large and heavy; emit excessive heat and are relatively noisy.

**[0005]** There has been a need for an improved portable device for supplying oxygen to a patient. Only small high pressure gas bottles and small liquid dewars are truly portable enough to be used for ambulatory needs. Either system may be used for both in-home and ambulatory use. A patient using a stationary oxygen system at home (or even a portable system which cannot be readily transported), who travels must opt for small cylinders towed in a wheeled stroller or for portable containers that they carry, typically on a shoulder sling. Both of these options have significant drawbacks.

**[0006]** A major drawback of the cylinder option is that small cylinders only provide oxygen for a short duration. Moreover, these cylinders are maintained at a high pressure, and thus their use is restricted due to safety considerations. Another drawback of the cylinders is the refill requirement after depletion of the contents of the cylinder. Empty cylinders must be refilled at specialized facilities, or in the patient's home using a commercial oxygen concentrator which extracts oxygen from the air. The latter option requires an on-site compressor to boost the output pressure of the concentrator to meet cylinder refill pressure requirements. Filling of cylinders with oxygen in the home is potentially dangerous due to the physics involved with compressing gas. Another detriment to cylinder usage is fire hazards associated with storage of large volumes of oxygen in the home environment.

**[0007]** Convenience and safety issues are not the only drawbacks associated with the use of cylinders. Another drawback is the cost associated with cylinders. Cylinders require special care, and specialized materials are required for high pressure oxygen compatibility, which in turn drives up the cost of cylinder-based systems.

**[0008]** The liquid oxygen storage system also has drawbacks. The primary drawback is the requirement of a base reservoir which necessitates refilling once a week or more from an outside source. Liquid oxygen is transferred from the base unit to a portable dewar, which is used by an ambulatory patient. However, there is substantial waste, as a certain amount of oxygen is lost during the transfer to the portable containers and from evaporation. Up to twenty percent of the contents of the base cylinder is lost in the course of two weeks because of losses in transfers and normal evaporation. Even without withdrawal by the patient, the base reservoir will typically boil dry over a period of one to two months.

**[0009]** The aforementioned systems all require a refilling station. When the patient is out in public, such stations are not readily available. Upon running low (or out) of oxygen, the



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patient must return home to a specified place that can refill the system. Such a requirement detracts from the ambulatory usefulness of the systems.

**[0010]** The industry has developed a set of recommendations for systems targeted to provide portable oxygen for ambulatory patients. The Fifth Oxygen Consensus Conference set forth the following standards for long-term oxygen therapy ambulatory equipment: 1) equipment must weigh less than 10 lbs., 2) equipment must provide the equivalent of 2 liter/min of continuous flow $O_2$, and 3) the flow rate must be maintained for four hours or more. Thus, ambulatory equipment, or personal oxygen systems (POS), are to be inconspicuous to the public as well as unrestricting to the patient. Cylinders and other liquid oxygen systems tend to be bulky, which interferes with normal daily activities. Similarly, cylinders and liquid oxygen systems are difficult to conceal from public view. Ideally, a POS is small, lightweight, quiet, and flexible which allows the device to be concealed from the public. The present invention, whereby oxygen rich gas is provided to a patient from a wearable oxygen concentrator, meets and exceeds these standards.

## BRIEF SUMMARY OF THE INVENTION

**[0011]** A removable gas separation cartridge includes a housing having an inlet port, an outlet port, and a bed of adsorbent material. The cartridge is removable from an oxygen concentrator which separates oxygen from ambient air by using an absorption process.

## BRIEF DESCRIPTION OF THE DRAWINGS

**[0012]** FIG. 1 is a front view of a patient carrying the oxygen concentrator of the present invention.

**[0013]** FIG. 2 is a front perspective view of the oxygen concentrator.

**[0014]** FIG. 3 is a rear perspective view of the oxygen concentrator.

**[0015]** FIG. 4 is a front perspective view of the oxygen concentrator and a docking station.

**[0016]** FIG. 5 is a block diagram showing the components and connections of the oxygen concentrator.

**[0017]** FIG. 6 is a diagram showing rotary valve timing.



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**[0018]** FIG. 7 is an exploded view of the oxygen concentrator in which the power pack and the adsorbent cartridge have been removed.

**[0019]** FIG. 8 is a perspective view of the oxygen concentrator with a portion of the belt removed.

**[0020]** FIG. 9 is a perspective view of the components of the oxygen concentrator without the belt.

**[0021]** FIG. 10 is a perspective view of the components contained within the case portions of the modules and their associated pneumatic and electrical connections of the oxygen concentrator.

**[0022]** FIG. 11 is a perspective view of the components contained within a battery module.

**[0023]** FIG. 12a is a perspective view of an accumulator.

**[0024]** FIG. 12b is a top view of the accumulator.

**[0025]** FIG. 12c is a sectional view of the accumulator.

**[0026]** FIG. 12d is another sectional view of the accumulator.

**[0027]** FIG.13 is a perspective view of a control module.

**[0028]** FIG. 14 is a front view of the interior components of the control module.

**[0029]** FIG. 15 is a perspective view of the cartridge contained within a cartridge module.

**[0030]** FIG. 16 is an exploded view of columns and filters within the cartridge.

**[0031]** FIG. 17 is a rear perspective view of the oxygen concentrator and the docking station.

**[0032]** FIG. 18 is a front perspective view of the docking station.

**[0033]** FIG. 19 is a chart showing the adsorption isotherms for two different adsorbent materials.

## DETAILED DESCRIPTION

**[0034]** The current invention relates to separation of gases using vacuum swing adsorption. Specifically, disclosed is an oxygen concentrator for a patient who requires a source of oxygen. The present invention is further explained with reference to the drawn figures in which like structures are referred to by like numerals throughout the several views.



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Overview - Oxygen Concentrator 100 (FIGS. 1-4)

**[0035]** FIG. 1 is a front view showing patient P with oxygen concentrator 100 and oxygen delivery tube 102. Oxygen concentrator 100 is a small unit which utilizes vacuum swing adsorption to separate oxygen from the ambient air around patient P. Oxygen concentrator 100 is compact and light so as not to interfere with the ambulatory movement of patient P, and can produce a product stream of gas containing a range of eighty-five to ninety-five percent oxygen.

**[0036]** Oxygen delivery tube 102 is a polymer tube or similar oxidation resistant structure, which extends from oxygen concentrator 100 to the nose, mouth, or port into the upper airway of patient P. Tube 102 allows delivery of oxygen to patient P for inhalation. In FIG. 1, patient P is about six foot tall to illustrate an approximation of the relative size of oxygen concentrator 100.

**[0037]** FIG. 2 is a perspective view of oxygen concentrator 100. Oxygen concentrator 100 is comprised of belt 104, power module 106 containing power pack 108, reservoir module 110, control module 112 containing user interface 114, and separation cartridge module 116. Oxygen concentrator 100 is a portable oxygen separator used to provide an oxygen rich gas stream to patient P. Belt 104 connects and carries the modules 106, 110, 112, and 116 of oxygen concentrator 100. Belt 104 may contain belt loops (not illustrated), clips, or a pair of straps that contain a buckle and holes or like fastening device for securing oxygen concentrator 100 to patient P. Alternatively, oxygen concentrator 100 may be placed in a purse, fanny pack, or similar personal carrying device for transport with patient P.

**[0038]** Power module 106 provides the necessary power to operate the systems of oxygen concentrator 100. In the embodiment illustrated, power module 106 contains replaceable power pack 108. Reservoir module 110 stores oxygen rich gas that has been separated from ambient air by cartridge module 116. Control module 112 pilots and regulates the interaction of the power module 106, reservoir module 110, and separation cartridge module 116 of oxygen concentrator 100. User interface 114 on control module 112 is a console which allows patient P to adjust and monitor oxygen concentrator 100.

**[0039]** FIG. 3 is a perspective view of the opposite side of oxygen concentrator 100 as shown in FIG. 2. Illustrated in FIG. 3 are belt 104, power module 106, reservoir module 110, control module 112, and separation cartridge module 116. Belt 104 is constructed to contain belt segments 120 formed by serrations 118. This allows the belt 104 to be flexible and conform to



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patient P=s body while wearing oxygen concentrator 100. Belt 104 is fabricated from a flexible material, such as textile or plastic and contains an inner padding such as foam. Belt 104 also houses the electrical and pneumatic connections of oxygen concentrator 100.

**[0040]** FIG. 4 is a perspective view of the front side of the oxygen concentrator 100 on docking station 122. Illustrated are oxygen concentrator 100 comprising belt 104, power module 106, reservoir module 110, control module 112, and separation cartridge module 116, along with docking station 122 containing power pack chargers 124a and 124b. Belt 104 is flexible and thus rests on the arc shaped docking station 122. Docking station 122 contains power pack chargers 124a (with power pack 108a inserted therein) and 124b, as well as concentrator dock 126 which supports the oxygen concentrator 100 while on the docking station 122. Docking station 122 converts AC power to recharge power packs 108.

Oxygen Concentrator 100 System Components and Connections (FIG. 5)

**[0041]** FIG. 5 is a block diagram of oxygen concentrator 100 illustrating power module 106, reservoir module 110, control module 112, and separation cartridge module 116, along with docking station 122 and showing the components and connections among the modules and docking station 122. Oxygen concentrator 100 components includes product gas outlet port 103, adsorbent columns 130a-130c (each containing a respective inlet port 132a-132c and a respective outlet port 134a-134c), air inlet port 135, air inlet filter 136, product gas final filter 138, main valve 140, drive reducer 142, vacuum pump 144, drive 146, electric control module (ECM) 148, breakthrough flow sensor 150, valve position sensor 152, product control pump 154, check valve 156, main storage reservoir 158 containing pressure sensor 159, dispensing valve 160, as well as previously identified components tubing 102, power pack 108, user interface 114, and docking station 122.

**[0042]** As shown in FIG. 5, adsorbent cartridge module 116 includes adsorbent columns 130a-130c each containing respective inlet ports 132a-132c and outlet ports 134a-134c, air inlet filter 136, and product gas final filter 138. Ambient air enters air inlet port 135, passes through air inlet filter 136, and enters valve 140 for distribution to adsorbent columns 130a-130c. Product gas passes through final filter 138 and product gas outlet port 103 into tubing 102 for delivery to patient P. Inlet ports 132a-132c of adsorbent columns 130a-130c connect to valve 140 through inlet lines 164a-164c. Similarly, outlet ports 134a-134c connect to valve 140 through outlet lines 166a-166c.



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**[0043]** Control module 112 houses main valve 140, drive reducer 142, vacuum pump 144, drive 146, electric control module (ECM) 148, breakthrough flow sensor 150, valve position sensor 152, and contains user interface 114. Main valve 140 is pneumatically connected to adsorbent columns 130a-130c, as well as inlet filter 136 via inlet line 168, vacuum pump 144 via vacuum inlet line 170, product control pump 154 via vacuum line 172 and product gas line 174, and main reservoir 158 via product gas line 176. Valve 140 is actuated by drive 146 through a motor speed reducer 142. Also, valve 140 connects to breakthrough flow sensor 150 and valve position sensor 152 which send electrical inputs 178 and 180 to ECM 148.

**[0044]** ECM 148 is a logic control, such as a PLC (programmable logic controller), microprocessor, or similar structure which controls operation of oxygen concentrator 100. ECM 148 contains the set of inputs and outputs associated with the modules for regulating oxygen concentrator 100. ECM 148 also receives control setting inputs182 and 184 from user interface 114, and docking station 122, respectively, power pack management input 186 from power pack 108, reservoir pressure input 188 from pressure sensor 159 in main reservoir 158, and nasal pressure input 190 from dispensing valve 160. ECM 148 provides interface output 192 to the user interface114, interface output 194 to docking station 122, power management output 196 to power pack 108, dispensing valve time open output 198 to dispensing valve 160, and motor drive output 200 to drive 146.

**[0045]** User interface 114 contains physical controls such as dials, toggle switches, push button switches, and similar controls, for operating oxygen concentrator 100. The physical controls provide electrical control settings to ECM 148. ECM 148 reads these settings as inputs 182 and provides output 192 to the user interface 114. The status is converted from electric signals to physical output by indicator lights, status display, and similar structures of user interface 114.

**[0046]** Power pack management input 186 and output 196 control the charge and discharge of voltage from power pack 108 to drive 146 via ECM 148. Drive 146 will activate vacuum pump 144, valve 140 through drive speed reducer 142, and any other systems requiring power. Power pack management output 196 will also supply power to indicator lights, status display, audible alarm (if included), and other passive electrical system requirements on user interface 114 through ECM 148.



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**[0047]** ECM 148 controls and coordinates the steps of the vacuum swing adsorption cycle through its inputs and outputs. In one embodiment, breakthrough flow sensor 150 provides an input 178 into ECM 148 by measuring air flow rates. The position of valve 140 is detected by valve position sensor 152 to produce input 180. Reservoir 158 contains a sensor to produce reservoir pressure input 188. Dispensing valve 160 also contains a pressure sensor which provides nasal pressure input 190 in response to differential pressure. ECM 148 reads these inputs to control the cycle by changing outputs, such as motor drive output 200 for drive 146. Drive 146 propels vacuum pump 144. Vacuum pump 144 creates a vacuum that is communicated to valve 140 through vacuum input line 170, while dispelling nitrogen rich gas as exhaust 177. Another output 198 controls the time that dispensing valve 160 is open. In this embodiment, the inputs and outputs are connected to a PLC within ECM 148 which is programmed to control the cycle of oxygen concentrator 100.

**[0048]** Contained within reservoir module 110 is an oxygen-rich gas accumulator comprising reservoir 158, check valve 156, product control pump 154, and check valve 153. Reservoir 158 receives oxygen-rich gas produced by oxygen concentrator 100 and stores it at a low pressure above ambient until it is required for use. A portion of the stored oxygen-rich gas is delivered back to valve 140 by product gas line 176 for use in ordering the nitrogen content in adsorbent columns 130a-130c by moving much of the residual nitrogen held after evacuation near the outlets134a-134c toward inlets 132a-132c of the columns 130a-130c. Reservoir 158 is in communication with dispensing valve 160 through product gas line 202. Check valve 156 opens to allow oxygen into reservoir 158 and closes to prevent backflow of oxygen upon reaching the desired pressure in reservoir 158.

**[0049]** Product control pump 154 is driven by vacuum provided by the vacuum pump 144 through valve 140 via vacuum line 172. Product line 174 is in communication from separation cartridge module 116 to check valve 153, which opens to allow product control pump 154 to transport separated oxygen-rich gas to reservoir 158. Product control pump 154 delivers the product gas to main reservoir 158 through check valve 156.

**[0050]** Dispensing valve 160 and power pack 108 are contained within power module 106. Dispensing valve 160 is used to feed the flow of oxygen-rich gas to the patient P by delivery of the product gas through final product gas line 205 to product final filter 138. The product gas is obtained from the main reservoir 158 through product gas line 202. Power pack



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108 provides the power supply for oxygen concentrator 100 as previously described. Power pack 108 is rechargeable through docking station 122 as represented by power connection 204.

Vacuum Swing Adsorption (VSA) Process- Overview

**[0051]** Oxygen concentrator 100 operates using a vacuum swing adsorption process, which involves a series of cycles that include a feed step or phase, an evacuation step or phase, and a repressurization step or phase. Each of these three phases takes place in one of the three columns 130a-130c at any given time. Each column 130a-130c is in a different phase. For purposes of explanation, the VSA process will be described in reference to Acolumn 130", which is representative of each of the three columns 130a-130c.

**[0052]** In the feed phase, a gas stream of ambient air 162 enters inlet end 132 of column 130 while product gas containing concentrated oxygen is delivered from outlet end 134 of column 130. The slight vacuum in column 130 draws air 162 into column 130 and through an adsorbent material (typically a zeolite) which preferentially retains specific components of air (nitrogen), allowing the desired product (oxygen) to pass through. A mass transfer zone (MTZ), which is a small region in which nitrogen is being adsorbed, is passing through the adsorbent material. The MTZ divides the column 130 into two segments: a nitrogen-rich segment where the MTZ has passed through, and an oxygen-rich segment ahead of the moving MTZ. The MTZ forms at the inlet 132 at the start of the process and gradually moves through the column to the outlet 134 as the process proceeds. Outlet end 134 of column 130 is connected to main reservoir 158 through main valve 140, check valve 153, and product control pump 154, so that oxygen-rich product gas from column 130 is pumped into reservoir 158.

**[0053]** In the evacuation phase, column 130 is brought to a stronger vacuum by vacuum pump 144, causing the adsorbed component, i.e. nitrogen, to be desorbed. The nitrogen is evacuated from column 130 through main valve 140, and is discharged by vacuum pump 144 as waste exhaust 177.

**[0054]** In the repressurization phase, the previously evacuated column 130 is returned to near 1 atm. Ambient air 162 enters column 130 through inlet end 132, and recycled product gas from product line 176 enters column 130 through outlet end 134. The gases replace the vacuum that was previously drawn in column 130 during the evacuation phase. Just prior to column 130 reaching about 1 atm, the repressurization phase ends and the feed phase of the cycle begins again.



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**[0055]** This constitutes the general principles of vacuum swing adsorption (VSA) for gas separation. All phases can be accomplished with a single column, or with a plurality of columns. If a plurality of columns are used, it is preferable to have a multiple of three (illustrated as 130a-130c in FIG. 5) that are sequenced out of phase for the different cycle phases in order to maintain constant product flow.

The Feed Phase- Breakthrough Detection

**[0056]** During the feed phase of the separation cycle, the position of the MTZ within adsorbent column 130 is monitored, determined, and beneficially used to control the termination of the feed phase. The control results in improvements in product purity and recovery with concomitant decrease in energy consumed, as well as system size and system weight for a given volume of product produced.

**[0057]** Breakthrough is defined as the point when the MTZ reaches outlet 134 of adsorbent column 130. At this point, feed gas begins to flow into the separated product gas stream. This is undesirable because the purity of the product stream is reduced by the feed stream gas if the feed is allowed to continue past this point. Conversely, if the feed phase is terminated before the MTZ nears outlet 134 of column 130, product recovery will be reduced because product gas contained in column 130 between the MTZ and outlet 134 of column 130 will be subjected to the evacuation phase that follows the feed phase in the separation cycle, and much of this remaining product gas will be lost with the desorbed gas in the waste stream.

**[0058]** For a particular column geometry, temperature, adsorbent type and condition, and cycle vacuum levels, there is an optimal time during the feed phase of the cycle to terminate the feed -- before purity requirements are compromised, but after the maximum possible product has been recovered from column 130. This optimal time is determined by the detection of the passage of the mass transfer zone through a specific position relative to outlet end 134 of column 130.

**[0059]** For some combinations of system variables, the optimum feed termination time corresponds to the beginning of breakthrough when the leading edge of the MTZ has just reached outlet end 134 of column 130. This event can be detected by monitoring either or both of the gas flow rates at inlet 132 or outlet 134 to column 130. Before breakthrough, the outlet flow rate is less than the inlet flow rate by an amount equal to the rate of nitrogen gas adsorption of column 130 from the feed gas flow. After breakthrough, column 130 is no longer adsorbing



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nitrogen from the feed gas, so the inflow and outflow rates of column 130 become equal. Any method of measuring gas flow rates to determine the point in time when these flow rates begin the transition toward equality can be used to detect this beginning of breakthrough.

**[0060]** It has been determined that if the inflow rate of air to column 130 is maintained constant, a simple detection of a significant rise in slope of the outflow rate marks breakthrough. Conversely, if the outflow rate is held fairly steady, then a falling slope of the inflow rate marks the breakthrough. Monitoring the ratio of flow for the inlet and outlet and detecting a significant change in the ratio of flows toward a ratio of 1:1 can mark breakthrough in systems where inflow or outflow may not be steady enough to detect breakthrough by monitoring just one of the flow rates.

**[0061]** For other combinations of system variables, the optimum feed termination time may correspond to the MTZ position prior to breakthrough. In these cases, it is beneficial for a specific amount of product to be intentionally left in column 130 at the end of a feed phase. Detecting the position of the MTZ before breakthrough can be accomplished by additional methods.

**[0062]** One method used determines the volume of gas passed into or out of adsorbent column 130 up to the point of breakthrough by integrating the flow rate during the time interval between an initial feed and breakthrough while using some breakthrough detection method as previously described. Volume of flow may also be directly measured by physical equivalent methods using displacements of known volumes. Once the volume of gas that passes the column up to the point of breakthrough is determined, the volume of gas flow can be monitored during subsequent feed phases and the feed terminated when the volume reaches a specific value less than that for breakthrough. At any time during the feed phase, the volume of gas passed through column 130 since the beginning of feed divided by the volume of gas at breakthrough will be the same ratio as the position of the mass transfer zone divided by the length of column 130 (assuming a constant cross sectional area along the length). Using this relationship, the position of the MTZ within column 130 can be adequately determined during the feed phase.

**[0063]** The components of oxygen concentrator 100 as previously described are used to complete the cyclical phases of VSA to separate gases. The feed phase operates at a slight vacuum just below ambient (in the range of 0.9 to 1 atm). This provides just enough driving force to pull ambient air 162 into adsorbent column 130 through inlet filter 136. The vacuum is



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caused by product control pump 154, which is driven by the vacuum drawn by vacuum pump 144. Product control pump 154 is a piston pump or similar structure that meters a volume of gas. Product control pump 154 connects with a volume much greater than the piston displacement volume, such as main reservoir 158.

**[0064]** The feed phase is allowed to proceed until breakthrough is detected. Up to this point, the outflow gas from adsorbent column 130 has been a high purity oxygen/argon, low percent nitrogen mixture. The MTZ position is controlled to minimize nitrogen into the product gas mixture. The MTZ position is monitored by breakthrough flow sensor 150, which detects a large increase in flow rate associated with breakthrough when the nitrogen no longer is preferentially adsorbed by adsorbent column 130. Breakthrough flow sensor is located near the column inlet 132, column outlet 134, or similar place where the flow rate being measured is accessible. When the increase MTZ flow is detected, a signal is sent to ECM 148, which also receives valve position signal 180 from the valve position sensor 152. The ECM 148 compares the timing of the MTZ breakthrough signal and the valve position signal and makes a minor adjustment to motor speed 200 based on lead, lag, or on-time status to keep the breakthrough time near the end of each feed phase. Alternately, ECM 148 receives a signal from breakthrough flow sensor 150 and immediately terminates the current feed phase in column 130 by signaling valve 140 to rotate to start the next phase. In yet another embodiment, the separation system contains a shut off valve that is signaled to close the feed of ambient air 162 into column 130, or the delivery of product gas from the column upon breakthrough detection.

**[0065]** In another embodiment, the method for determining the position of the mass transfer zone prior to breakthrough is accomplished by placing a small amount of non-adsorbing material within adsorbent column 130 at a particular position. When the mass transfer zone passes through this position, a flow change is detectable as the adsorption of gas is briefly interrupted by the non-adsorbing segment of column 130. The resulting flow change is detectable using the same methods for breakthrough detection previously described.

**[0066]** With larger columns and slower feed phases, the position of the mass transfer zone has been established by measuring temperature rise at positions of interest within column 130. Significant temperature increases result from the heat of adsorption at the MTZ and can be detected by thermistors or similar devices placed within column 130.

The Evacuation Phase



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**[0067]** The evacuation phase brings the gas in adsorbent column 130 that was just in the feed phase to a vacuum state. At the end of the feed phase, the adsorbent column 130 is in equilibrium with the air infeed mixture near 1 atm from column inlet 132 up to the MTZ. Hence, if the ending position of the MTZ is established, and the nitrogen, oxygen, and argon isotherms for the chosen adsorbent mass are known, then the quantity of these gases present in adsorbent column 130 at the end of each feed phase is known. Vacuum pump 144 draws a vacuum on adsorbent column 130. This vacuum level is determined and set to a state that will remove a large portion of the gas left in column 130. In one embodiment, this is 0.2 to 0.3 atm. By percentage, the vast majority of gas discharged is nitrogen. The evacuated gas is discharged as waste from exhaust 177 of vacuum pump 144. The preferred embodiment uses a fixed displacement type of vacuum pump 144. During each evacuation phase, the adsorbed gas in column 130 is expanded into a much larger volume made up of the column volume plus the fixed displacement volume of pump 144.

**[0068]** The evacuate phase creates a self regulating effect that compensates for reductions in the amount of nitrogen adsorbed by adsorbent column 130 as the adsorbent degrades (ages). If the adsorbent loses efficiency, less nitrogen will be present in column 130 at the end of the phase, but the volume of the pump that the nitrogen expands into remains the same. A stronger vacuum will result that will remove more nitrogen and therefore allow more air to be fed during the next feed phase. A more constant breakthrough time results and provides a more robust product cycle.

**[0069]** The evacuation is provided by vacuum pump 144, which is controlled and activated by drive 146. The volume removed for each cycle of the vacuum pump 144 will remain constant, but the motor drive output 200 will be controlled by the rate of product gas used by patient P. The amount of oxygen used by patient P depends on the patient P=s on-demand respiratory rate, which is sensed by the device and from a variable position switch which sends an input 192 from user interface 114 to ECM 148, which in turn provides motor drive output 200 to drive 146. This determines the speed of each successive phase and, therefore, the oxygen production rate.

**[0070]** In one embodiment, a purge is applied at the very end of an evacuation phase. While still in the evacuation phase, a purge of product gas (mostly oxygen) introduced through outlet 134 effectively drives out a portion of nitrogen in column 130 through inlet 132. Adding



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the purge gas of high purity oxygen/argon through outlet 134 desorbs more nitrogen from outlet 134 of column 130, and pushes the nitrogen toward inlet 132 of adsorbent column 130 and creates an ordering of the gases. The purge volume is a function of vacuum level and adsorbent characteristics. A purge portion of the evacuation phase is not a necessary phase for a functioning device, but allows high oxygen purity to be maintained with weaker vacuum levels.

The Repressurization Phase

**[0071]** The repressurization phase brings adsorbent column 130 (just previously evacuated and purged) up to the feed pressure. In one embodiment, the gas used for repressurization is from both the infeed ambient air 162 and a counter stream from the (oxygen-rich) product gas line 176 from the main reservoir 158. Alternately, the repressurization of product gas can be accomplished through valve design negating the need for a separate line. The product gas is dispensed from a stream of product gas from the adsorbent column that is in the feed phase through a vacuum break valve used during repressurization. Repressurization with product gas can be done before, simultaneously, or after partial pressurization with ambient air. Repressurization with product gas is done at the opposite end of column 130 as repressurization with ambient air 162.

**[0072]** The effect of adding the repressurization gas of high purity oxygen/argon through outlet 134 creates a cleaning zone at outlet 134 of adsorbent column 130 where, during the feed phase that follows next, any stray nitrogen can be preferentially adsorbed and not discharged as product gas. This improves the ordering of gases in the adsorbent column 130. By repeating this phase during successive cycles, the purity will continue to increase in the product output. Weaker vacuums require more oxygen volume returned to column 130 during repressurization if high purity is desired. That is, a stronger vacuum must be drawn on the column 130 to effectuate the same purity of oxygen absent the use of oxygen-rich gas as a back flush for repressurization at the outlet 134 of column 130. At the end of repressurization, the feed phase will proceed.

Valve 140 Timing (FIG. 6)

**[0073]** The VSA cycle comprises three phases: evacuation, repressurization, and feed, which occur sequentially in each column 130a-130c. For clarity, only column 130a will be discussed, although each phase is performed (at different times during a complete cycle) in each of columns 130a-130c.



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**[0074]** Starting with the evacuation phase of the cycle, a small amount of oxygen (not illustrated) may flow into outlet 134a of adsorption column 130a to purge adsorption column 130a, while vacuum pump 144 withdraws gas present at inlet 134a of the column, i.e. nitrogen-rich gas.

**[0075]** During the repressurization phase, an amount of previously separated oxygen flows into outlet 134a of adsorption column 130a for a short time, and then air is allowed to enter inlet 132a of column 130a that has been previously evacuated. There may be a slight overlap of the oxygen flow into outlet 134a of adsorption column 130a and the air flow in the opposite direction into inlet 132a. Air freely flows into inlet 132a of adsorption column 130a upon opening of valve 140 as adsorption column 130a has been previously evacuated during the evacuation phase.

**[0076]** During the feed phase, air continues to flow into inlet 132a of adsorption column 130a while oxygen is removed from outlet 134a of column 130a by a pressure differential created by product control pump 154. As the MTZ passes through the adsorption column and reaches a position at or near outlet 134a, vacuum pump 144 will again begin to evacuate adsorbent column 130a and restart with the evacuation phase. In the embodiment illustrated in FIG. 6, these phases are controlled by main valve 140.

**[0077]** FIG. 6 is a diagram showing timing for main valve 140, which is a rotary valve that moves 360E(one full revolution about a central axis) for each complete cycle of the VSA process. In the embodiment with three columns 130a-130c, the timing for each phase of the cycle is 120E. Each column 130a-130c is present in a different phase for each 120E of rotation of valve 140 that is different from the other two columns to obtain a sequence that creates a steady flow of oxygen as valve 140 keeps rotating. As shown in the timing diagram, adsorption column 130a is in the feed phase of the cycle at a start point of zero degrees. Air is being let in through inlet filter 136 and column inlet 132a while separated gas consisting of highly concentrated oxygen is being removed through column outlet 134a. A portion of the oxygen-rich product gas is used in the repressurization of column 130b. Adsorption column 130b is in the repressurization phase at a point of rotary valve 140 being in initial position zero degrees. As valve 140 is turned, column outlet 134b is fed with the oxygen-rich gas for a portion of the valve=s rotation, preferably less than 120E. After the flow of oxygen-rich gas enters column 130b through the column outlet 134b, air repressurization through the opening of column inlet



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132b begins. In the embodiment shown, this takes place at a point after valve 140 has begun its rotation and ends before it reaches a third of its rotation, or a 120E rotation.

**[0078]** While column 130a is in feed phase and column 130b is in the repressurization phase, column 130c is in the evacuation phase. During the evacuation phase, a vacuum is drawn to remove adsorbed gas through inlet 132c, thereby regenerating it for the following feed phase.

**[0079]** In the embodiment shown, each column 130a, 130b, and 130c, is in a different phase of the cycle as one moves vertically down the diagram in FIG. 6. During the first one hundred twenty degrees of rotation of the rotary valve, column 130a is in the feed phase. Simultaneously, from zero to one hundred twenty degrees of rotation, column 130b is being repressurized, while column 130c is being evacuated.

**[0080]** For the next one hundred twenty degrees of rotation of valve 140 (i.e., from 120E to 240E), adsorption column 130a is in the evacuation phase. At this same time, column 130b is in the feed phase, and column 130c is in the repressurization phase. Moving horizontally across the diagram for column 130a, during the final one hundred twenty degrees of rotation of valve 140 (i.e., from 240E to 360E), column 130a is repressurized first using separated gas, and then ambient air. Separated gas and ambient air are introduced to the column 130a through column inlet 132a and column outlet 174a at opposite ends of column 130a. During the final one hundred twenty degrees of rotation (i.e. from 2401 to 3601) of main valve 140, column 130b is in the evacuation phase, and column 130c is in the feed phase. Upon reaching three hundred sixty degrees, valve 140 is back at its starting position (zero degrees), and the cycles for each column 130a-130c restart from the zero degree position.

Oxygen Concentrator 100 Physical Components (FIGS. 7-16 )

**[0081]** FIG. 7 shows an exploded view of the oxygen concentrator 100, which includes belt 104, power module 106 containing removable power pack 108, reservoir module 110, control module 112, and separation cartridge module 116 containing adsorbent cartridge 206. Power pack 108 has been removed from receptacle 210 of power module 106. Adsorbent cartridge 206 has been removed from receptacle 208 of cartridge module 116. Adsorbent cartridge 206 and power pack 108 are easily removable to facilitate replacement.

**[0082]** In this embodiment, power pack 108 is a rechargeable battery. Receptacle 210 contains electrical contacts (not illustrated) for connection to power pack 108. Cartridge 206 contains a quick-connect attachment (not illustrated) for inlet lines 164a-164c, outlet lines 166a-



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166c, inlet air line 168, and final product gas line 205 (not illustrated) within receptacle 208. Also present on cartridge 206 are air inlet ports 135 which receive ambient air 162 for separation. Adsorbent cartridge 206 contains adsorbent material that deteriorates in efficiency as it is used and ages.

**[0083]** FIG. 8 is a perspective view of oxygen concentrator 100. A portion of belt 104 has been removed revealing back interior surface 211 and inner connections amongst the modules, including utility tubes 212, tube pathways 214, module apertures 216a-216d, and module sockets 218a-218d.

**[0084]** Utility tubes 212 run between the adjacent modules and contain either electrical wiring or pneumatic lines, or comprise pneumatic lines and electrical wiring and associated connections. Tubes 212 are constructed to be flexible and bend as belt 104 is manipulated. If the tubes 212 contain electrical lines, the tubes are constructed from a dielectric material to insulate electrical wires, or similar material commonly used in electrical connections. If the tubes 212 comprise pneumatic lines, they may be air tight, small diameter polyvinyl or PVC tubes to connect the various gas input, gas separation, and gas removal systems of the oxygen concentrator 100. Tubes 212 contain openings or connections as required for electrical and pneumatic communication with each module. The back interior surface 211 of belt 104 contains tube pathways 214. Pathways 214 fabricated on the interior surface 211 of belt 104 allow the utility tubes 212 to extend between the modules 106, 110, 112, and 116. Tube pathways 214 create a semi-partitioned area on back interior surface 211 of belt 104 which support tubes 212.

**[0085]** Belt 104 is fabricated to contain apertures 216a-216d which allow modules 106, 110, 112, and 116 to connect utility tubes 212 of belt 104 through sockets 218a-218d. Apertures 216a-216d and sockets 218a-218d are fabricated as part of modules 106, 110, 112, and 116. Serrations 118 can be seen between the upper and lower edges 220 and 222 of belt 104. When fabrication of belt 104 is completed, padding will be inserted between edges 220 and 222, and material will be wrapped around creating serrations 118 and belt segments 120 to complete belt 104 as illustrated in FIG. 3. The padding is fabricated over the top of tubes 212 and tube pathways 214, or separately fabricated and fastened to back interior surface 211 during assembly of belt 104. Individual modules allow the device to flex when mounted about a curved surface, such as a belt around patient P=s waist. The construction of belt 104 with tubes 212 allows patient P to manipulate the oxygen concentrator 100, such as by bending belt 104 to wear around



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the waist, place on docking station 122, or folding concentrator 100 in half for transport in a carryall.

**[0086]** FIG. 9 is a perspective view of modules 106, 110, 112, and 116 of oxygen concentrator 100. In this view, belt 104 has been removed to illustrate the positions of sockets 218a-218d containing apertures 216a-216d on each respective module 106, 110, 112, and 116. Each respective module 106, 110, 112, and 116 is constructed from a thermoplastic material such as acrylanitrile butadine styrene (ABS) or high density polyethylene (HDPE), or a lightweight metal or a similar rigid material that is oxidation resistant.

**[0087]** Each module 106, 110, 112, and 116, comprises a case portion 224, 226, 228, and 230, defining the outer volume of each respectively. Bottom padding 232, 234, 236, and 238, covers the lower base portion of each module 106, 110, 112, and 116, respectively. Similarly, top padding 240 extends around the top perimeter of power module 106, while top padding 242 and 244 covers the top portions of modules 110 and 112. Power pack top padding 246 covers the top portion of power pack 108 and cartridge top padding 248 covers the top of separation cartridge 206. Padding 222-248 is a foam or similar lightweight material that adds protection to the modules as well as acts to reduce vibration of oxygen concentrator 100 felt by patient P. Alternately, oxygen concentrator 100 is enclosed in soft, flexible material to further increase comfort and maintain flexibility. In one embodiment, padding 232-248 is fabricated separately from the modules 106, 110, 112, and 116, power pack 108 and cartridge 206. In assembling the oxygen concentrator 100, padding 232-248 and case portions 224, 226, 228, and 230, are merged and secured either using fasteners, adhesives, or a manufacturing process such as ultrasonic welding.

**[0088]** Case portions 224, 226, 228, and 230, of each of the modules 106, 110, 112, and 116, contain sockets 218a-218d fabricated on the surface that contacts belt 104. Socket 218a-218d for each module is constructed to have support paths 250 for electrical wiring and pneumatic tubing similar to those contained within belt 104 represented by tube pathways 214. Sockets 218a-218d are constructed so that support paths 250 on sockets 218a-218d align with tube pathways 214 in belt 104 when each individual module 106, 110 ,112, and 116, is connected to belt 104. In one embodiment, sockets 218a-218d are constructed to allow each individual module 106, 110, 112, and 116, to snap onto belt 104 or attach in a similar quick connect fashion. Utility tubes 212 comprise quick connects at module apertures 216a-216d. Apertures



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216a-216d are openings in the case portions 224, 226, 228, and 230, provided for connection of utility tubes 212 to components contained within each module 106, 110, 112, and 116. This allows for removal of a single module 106, 110, 112, or 116 should a specific component require maintenance or replacement. Sockets 218a-218d are constructed from the same material as the case portions 224, 226, 228, and 230.

**[0089]** FIG. 10 is a perspective view of the components contained within modules 106, 110, 112, and 116, and the associated pneumatic and electrical connections of oxygen concentrator 100. Illustrated are power pack 108, valve 140, drive reducer 142, vacuum pump 144, drive 146, oxygen accumulator 252 (comprising product control pump 154, check valves 153 and 156, and reservoir 158), dispensing valve 160 connected to nasal pressure sensor line 190 and dispensing valve open line 198, column inlet lines 164a-164c, column outlet lines 166a-166c, product control pump vacuum line 172, product control pump inlet line 174, product gas line 202, final product gas line 205, adsorbent cartridge 206, and main electrical cable 254.

**[0090]** Main electrical cable 254 contains a set of electrical wires that carry inputs 178, 180, 182, 188, and 190, outputs 192 and 198, and power lines 186, 198, and 200 shown in FIG. 5. Main electrical cable 254 extends from power pack 108 to ECM 148 (not visible in FIG. 10). Dispensing valve time open output 198 and nasal pressure sensor input 190 are wires that extend between ECM 148 and dispensing valve 160. Similarly, the other inputs and outputs are wired to the appropriate system components as illustrated in FIG 5 (although not specifically illustrated in FIG 10.)

**[0091]** Product gas line 202 connects dispensing valve 160 with reservoir 158 of accumulator 252. Final product gas line 205 connects dispensing valve 160 to product gas outlet port 103 for connection to delivery tube 102 after passing through final filter 136 located in adsorbent cartridge 206 to provide patient P with oxygen rich product gas. Product control pump inlet line 174 extends from main valve 140 to product control pump154, which pumps separated oxygen rich gas into reservoir 158. Vacuum line 172 connects product control pump 154 through valve 140 to a vacuum drawn by vacuum pump 144, and provides the actuation for product control pump 154. Column inlet lines 164a-164c and column outlet lines 166a-166c connect main valve 140 with column inlet ports 132a-132c and column outlet ports 134a-134c of columns 130a-130c, respectively (not illustrated). Inlet air line 168 transports ambient air 162 from separation cartridge 206 to main valve 140, while vacuum inlet line 170 connects vacuum



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pump 144 to main valve 140. All lines 164a-164c, 166a-166c, 168, 170, 172, 174, 176, 202, and 205, are pneumatic lines or similar structures that allow for the isolated flow of gases between system components.

**[0092]** FIG. 11 is a perspective view of the components contained within power module 106: power pack 108 (comprising cells 256, outer wall 258, and power pack life indicator 260) and dispensing valve 160. In the embodiment illustrated, power pack 108 is a lithium-ion battery pack comprised of five cells 256. Individual cells 256 are contained within outer wall 258, part of which has been removed to show cell 256. Power pack 108 is a battery that is rechargeable and removable from power module 106. Although illustrated as a trapezoid containing five cylindrical cells, the shape and number of cells will vary depending on the shape of power module 106 and power requirements of oxygen concentrator 100.

**[0093]** Power pack 108 is a lithium based battery pack capable of being recharged in a recharging socket or station that connects to an external power supply. Alternatively, power pack 108 comprises a battery or fuel cell. In one embodiment, power pack 108 is a lithium ion battery pack that is constructed from several interconnected lithium-ion batteries. Oxygen concentrator 100 uses a maximum of fifteen watts of power. This results in a battery weight of less than 0.7 pounds (0.3 kg). In this embodiment, patient P taking twenty breaths per minute at a setting of 2 liters per minute equivalent can use oxygen concentrator 100 for a minimum of four hours on a fully charged battery. Power pack 108 is easily exchanged with another similar battery pack, and can be removed with a simple pulling or tugging motion. In another embodiment (not illustrated), oxygen concentrator 100 contains a jack for receiving a power cord which can then be plugged into either a 110 volt wall outlet or a 12 volt power supply system (such as a car utility plug) so that power pack 108 can be charged in place in oxygen concentrator 100.

**[0094]** Power pack life indicator 260 displays the amount of time left that the power pack will operate the oxygen concentrator 100. As illustrated, power pack life indicator 260 is a display, such as a liquid-crystal display (LCD) or light emitting diode (LED) screen, with numeric output of expected life in hours. The LCD or LED screen may also contain a series of bars that act as indicators. Alternately, power pack life indicator 260 is a light or series of lights.

**[0095]** Dispensing valve 160 is contained within power module 106 and is used to feed the flow of oxygen to patient P. Dispensing valve 160 is a valve activated by a change in



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pressure, such as that caused when a person is inhaling. A sensor in the dispensing valve circuit monitors pressure, and opens dispensing valve 160 when a drop in pressure is sensed. ECM 148 communicates with dispensing valve 160 through input 190 and output 198 (see FIG. 5). Dispensing valve 160 is in communication with reservoir 158 through product gas line 202. Reservoir 158 is kept at a slight pressure above ambient. Thus, when dispensing valve 160 is opened, oxygen rich gas will flow from reservoir 158 through final product gas line 205, final product filter 138, and product gas outlet port 103 for delivery to patient P through tubing 102. The flow of gas is further assisted by the pressure drop created by patient P=s inhaling. Dispensing valve 160 can be set to deliver oxygen rich gas to patient P for the beginning portion of a breath when patient P first inhales rather than the whole breath.

**[0096]** Dispensing valve 160 provides for operating oxygen concentrator 100 in one of two possible modes: pulse flow or continuous flow. When patient P is using the oxygen concentrator 100 in the pulse flow mode, dispensing valve 160 will open intermittently in response to inhalation and will stay open for a pulse time according to the setting of the controls as set by patient P. If continuous flow is desired, dispensing valve 160 is maintained at an open or partially opened state. The product is dispensed to patient P though oxygen delivery tube 102 at a continuous rate, typically in the range of 1-1.5 lpm (liters per minute). The pressure difference corresponding to a dispensing orifice in delivery tube 102 will accommodate the flow rate from the reservoir 158.

**[0097]** FIGS. 12a-12d are various views of components contained inside of reservoir module 110. FIG. 12a is a perspective view of accumulator 252. FIG. 12b is a top view of accumulator 12b. FIGS. 12c and 12d are sectional views corresponding to the section lines in 12b. Contained within reservoir module 110 is oxygen accumulator 252 (comprising reservoir 158, check valves 153 and 156, and product control pump 154), inlet port 261, and outlet port 263. Accumulator 252 receives separated product gas through inlet port 261. Product inlet line 174 (shown in FIG. 10) is connected to inlet port 261 and links product control pump 154 to separation cartridge 206 through main valve 140 to transport oxygen rich gas separated by cartridge 206. Inlet port 261 connects to check valve 153 which allows product gas into product control pump 154.

**[0098]** Product control pump 154 includes piston 262, actuated by spring 264 within pump chamber 265, which pushes separated oxygen-rich product gas into reservoir 158. Check



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valve 156 opens to allow oxygen into the reservoir 158 and closes to prevent back flow of oxygen-rich gas when the desired pressure in reservoir 158 is attained. The low pressure of reservoir 158 exerts a force on check valve 156 to keep valve 156 closed. Reservoir 158 takes oxygen-rich gas produced by oxygen concentrator 100 and stores it at a low pressure above ambient until the product is required for use by patient P.

**[0099]** Product control pump 154 is driven by vacuum. Product control pump vacuum line 174 is connected to the vacuum drawn by vacuum pump 144 through main valve 140. When a vacuum is drawn, the force draws piston 262 down to compress spring 264 which expands pump chamber 265, and causes check valve 153 to open. Oxygen-rich gas from separation cartridge 206 flows through check valve 153 and enters pump chamber 265 in the volume created by the displacement of piston 262. At the appropriate time in the cycle, valve 140 will interrupt the vacuum to product control pump 154, and spring 264 will force piston 262 upwards. The movement of piston 262 will force oxygen rich gas in pump chamber 265 through check valve 156 and into reservoir 158. At the same time, check valve 153 closes to prevent more gas from entering pump 154. With this embodiment, no additional drive (other than the vacuum to pull piston 262 down and the spring force to move it up) is required for product control pump 154 which adds to the overall efficiency of the system.

**[00100]** In one embodiment, reservoir 158 has a capacity that is about four times larger than the size of the largest pulse provided by oxygen concentrator 100. In one embodiment, extra volume is included to account for separated oxygen used as a back flow in adsorbent columns 130a-130c. Specifically, main storage reservoir 158 for oxygen concentrator 100 can be designed according to the flow rates listed in Table 1. Storage reservoir 158 is 100cc (cubic centimeters) to 400cc in volume. Main storage reservoir 158 is maintained at a low pressure to provide delivery of the product gas to patient P through outlet 263 which is connected to final product dispensing line 202. In one embodiment, the pressure is between 1 atm (ambient) and 1.5 atm. Also acceptable are pressures less than eight psi (55,158 Pa), with a pressure of two and one half to five psi (17,236 to 34,473 Pa) preferred. The low pressure of reservoir 158 allows oxygen concentrator 100 to be used in most areas where high pressure oxygen is banned. Also, low pressure requires less energy to fill reservoir 158, which adds to the efficiency of the system by requiring a simpler pressurizing mechanism compared to high pressure systems.



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**[00101]** Reservoir 158 contains pressure sensor 159 (such as a piezoresistive or capacitive sensor) that sends reservoir pressure signal 188 to ECM 148 (see FIG. 5). ECM 148 adjusts the speed of the motor of drive 146 based on reservoir pressure signal 188 in combination with the current settings on user interface 114. As patient P's respiratory rate increases for the current setting, more oxygen-rich gas from reservoir 158 is dispensed thus lowering the pressure of reservoir 158. The drop in pressure is sensed and the system will react by increasing the production of product gas. Similarly, a decrease in the respiratory rate of patient P at the current setting of oxygen concentrator 100 raises the pressure in reservoir 158. The rise in pressure is sensed and ECM 148 adjusts drive 146 accordingly to maintain a preset pressure range in reservoir 158. Thus, only the amount of oxygen used by patient P is produced by oxygen concentrator 100.

**[00102]** FIG. 13 is a perspective view of the control module 112. Illustrated are the case 228 containing aperture 216c and socket 218c, and padding 236 and 234. Control module 112 contains user interface 114 comprising power switch 266, flow level indicator lights 268, flow setting switches 270a and 270b, boost switch 272, and indicator lights 274 and 276. Power switch 266 is an ordinary toggle or push button switch capable of turning oxygen concentrator 100 on and off.

**[00103]** Flow settings are dually controlled by patient P utilizing flow setting switches 270. First, continuous or pulse mode is selected by patient P. In a continuous flow mode, oxygen is dispensed at a continuous flow rate such as one to one and a half liters per minute. If oxygen concentrator 100 is set in a pulse mode for controlling flow, oxygen concentrator 100 utilizes dispensing valve 160 to provide pulse dispensing of product gas. The pulse mode is set to meet patient P=s needs for the equivalent of one to five liters per minute of continuous oxygen flow. In one embodiment, a dial containing settings of one to five is utilized. In the embodiment illustrated in FIG. 13, flow setting switches are used to adjust the flow rate between various stepped levels. Each setting corresponds to the specific value for continuous flow, or a corresponding pulse volume. For example, settings for a pulse mode are contained in the table below.



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TABLE 1

| Setting | Total Volume Pulse Range (cc/pulse) | Trigger Time (sec) | Pulse Flow Ramp Rate (sec) | Pulse Duration Max (sec) | Peak Pulse Flow (LPM) |
|---|---|---|---|---|---|
| 1 | 10 to 12 | .001 to .02 | .03 to .07 | .15 | 14 |
| 2 | 20 to 24 | .001to .02 | .03 to .07 | .20 | 14 |
| 3 | 30 to 36 | .001 to .02 | .03 to .07 | .25 | 15 |
| 4 | 40 to 48 | .001 to .02 | .03 to .07 | .30 | 16 |
| 5 | 50 to 60 | .001 to .02 | .03 to .07 | .35 | 17 |

**[00104]** When the unit is set in pulse mode, product gas is dispensed only at the beginning of inhalation. In one embodiment, product dispensing valve 160 is only opened between zero and 0.4 seconds of the beginning of a breath of patient P. This controls the amount of oxygen removed from reservoir 158. In another embodiment, oxygen concentrator 100 is shut off if no pressure drop is sensed by nasal pressure sensor 190 for a set amount of time, such as two minutes, which in turn closes dispensing valve 160.

**[00105]** Patient P can temporarily increase (or Aboost@) the flow rate of oxygen by actuating boost control switch 272 on user interface 114. When boost switch 272 is activated, oxygen concentrator 100 increases the flow rate of oxygen for a set period of time, such as 10 minutes. After timing out, oxygen concentrator 100 returns to the previous setting. The boost function will not work if oxygen concentrator 100 is already operating at the maximum flow rate.

**[00106]** Indicator light 274 indicates power pack 108 is running low. Indicator light 276 indicates that there is a problem with separation cartridge 206, such as a bad connection with receptacle 208. In one embodiment, oxygen concentrator 100 contains three different colored lights: red, yellow, and green. The green light indicates that there are no problems detected with oxygen concentrator 100. A yellow flashing light or a yellow non-flashing light indicates a condition has been sensed that should be addressed. An example of such a condition is a low battery. A red flashing light indicates that a condition has been detected that requires an immediate response. A red non-flashing light indicates that oxygen concentrator 100 has failed, and has shut down. For example, if oxygen concentrator 100 fails to produce a stream of separated gas of eighty-five percent oxygen, oxygen concentrator 100 will detect this problem via breakthrough flow sensor 150. ECM 148 shuts off main valve 140 so no ambient air 162 is being submitted to the gas separation cartridge 206. Product gas is no longer being supplied to



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reservoir 158. After a few breaths, reservoir 158 will empty, triggering reservoir pressure sensor output 188 (shown in FIG. 5), which communicates to ECM 148 to shut down oxygen concentrator 100, and display the red warning light. This signals patient P that maintenance is needed. In addition to the aforementioned indicator lights, the unit may also contain a boost indicator light to indicate when a boost function is in operation. Similarly, an audible alarm may be included in oxygen concentrator 100 to indicate failure.

**[00107]** FIG. 14 is a front view of the interior components of control module 112. Illustrated are drive 146, vacuum pump 144, drive speed reducer 142, and valve 140. Drive 146 includes a DC motor, driven by battery power pack 108, which supplies the necessary power to operate vacuum pump 144. The motor draws a maximum of 15 watts of power. Vacuum pump 144 is a positive displacement pump. In this embodiment, drive 146 runs both vacuum pump 144 and valve 140. Vacuum pump 144 is run by the motor at one speed, while valve 140 is run off the same motor but at a reduced speed. The reduction in speed is accomplished with gears that comprise drive speed reducer 142 between the motor of drive 146 and valve 140.

**[00108]** Valve 140 is a valve containing a minimum number of ports equal to two times the number of adsorbent beds (columns) in separation cartridge 206. Additionally, main valve 140 contains other ports for the inlet of ambient air 162, vacuum provided by vacuum pump 144, and recycling of product gas used to purge columns 130a-130c during repressurization. In the preferred embodiment, valve 140 is a rotary valve, but may also be a solenoid valve, directional control valve, or series of individual valves in communication with each other and each connected to an adsorbent column 130a-130c.

**[00109]** In an alternate embodiment, drive 146 may contain an independent motor for operating valve 140. If valve 140 is run by an independent motor, that motor is powered by power pack 108 and synchronized with the other motor(s) of drive 146 by ECM 148. As illustrated, drive 146 contains a single motor and valve 140 is connected to a system of gears that comprise drive speed reducer 142. Alternately, drive speed reducer 142 can be any common power transmission components such as pulley and belts, or gears and sprockets.

**[00110]** FIG. 15 is a perspective view of separation cartridge 206 contained within separation cartridge module 116. Illustrated are inlet ports 132a-132c, outlet ports 134a-134c, and casing 230. In the embodiment illustrated, three adsorption columns are contained within casing 230 with one inlet port 132a-132c, and one outlet port 134a-134c, for each adsorption



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column 130a-130c. Each adsorption column 130a-130c is a hermetically sealed container containing a bed of adsorption material, preferably a zeolite capable of adsorbing nitrogen gas, such as lithium low silica 13X zeolite. Each bed contains between five and twenty-five cubic centimeters of material, and in one embodiment contains fifteen (plus or minus one) cubic centimeters of material. The adsorbent bead size is a thirty by sixty mesh, wherein thirty mesh is equal to 0.0234 inches (0.0594 cm) and sixty mesh is equal to 0.01 inches (0.0254 cm).

**[00111]** Column inlet ports 132a-134c are connected to receive either ambient air 162 or vacuum, while outlet ports 134a-134c expel product gas or receive purge gas. This arrangement promotes ordering of gases within the columns 130a-130c by having oxygen rich gas always present at one end of the column. This results in improved efficiency as air flow through the columns 130a-130c creates an oxygen rich zone continuously at one end, which allows the vacuum to evacuate and desorb the previously adsorbed nitrogen where it is contained in the greatest concentration.

**[00112]** FIG. 16 is a perspective view of the columns 130a-130c and the filters 136 and 138 within casing 230 of separation cartridge 206. Illustrated are final product filter 138 connected to product gas outlet port 103 which connects to tubing 102, inlet air filter 136, and adsorbent columns 130a-130c each comprising spin inducers 280a-280f, adsorbent material 282a-282c, porous filters 284a-284f, and springs 286a-286c. Springs 286a-286c are coil springs that hold each adsorbent column in compression 130a-130c in place within casing 230 of separation cartridge 206 to prevent movement of adsorbent beads. Spin inducers 280a-280f help force even distribution of gases through columns 130a-130c, which helps to keep the MTZ well defined for more accurate detection.

**[00113]** Adsorbent material 282a-282c is the same as that previously described. Filters 136 and 138 are constructed of common filtering materials and are used to remove dust and other large particulate matter from the air streams to assure that the flow of oxygen out to patient P is free of such materials. Porous filters 284a-284f are a small section of material commonly used as a particle filter provided at each end of columns 130a-130c. Porous filters 284a-284f act to prevent adsorbent particles from contacting the mechanisms and valving of concentrator 100.

Docking station 122 (FIGS. 17-18)

**[00114]** FIG. 17 is a perspective view of the back side of oxygen concentrator 100 on docking station 122. Illustrated are oxygen concentrator 100 comprising power module 106,



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reservoir module 110, control module 112, and separation cartridge module 116, belt 104, and docking station 122 containing status display 288. Status display 288 is an LED, LCD, or similar digital display used to provide information to patient P such as time of day, time the concentrator has been used, time of recharging for power pack 108, or similar information. Additionally, docking station 122 may contain other controls (not illustrated) including a boost setting while the concentrator is docked, a mode switch for switching between pulse and continuous flow of oxygen, and indicator lights to show expected battery life, adsorbent column life, gas input, gas output, or gas separation system malfunctions, or other similar items that were previously described as part of user interface 114.

**[00115]** FIG. 18 is a perspective view of the docking station 122 with oxygen concentrator 100 removed. Concentrator dock 126 is visible on docking station 122 with oxygen concentrator 100 removed. Concentrator dock 126 may optionally contain electrical connections (not illustrated) to charge power pack 108 contained within power module 106 while oxygen concentrator 100 is docked. Additionally, docking station 122 contains a power cord (not illustrated) available to connect to a wall socket or other power source such as a car utility plug. Docking station 122 uses power provided through the power cord to operate oxygen concentrator 100 and/or recharge power packs 108. Docking station 122 contains a flat bottom 290 to rest on a level surface and allow oxygen concentrator 100 to be in the docking station without moving. Alternately, docking station 122 is mountable to a wall in one embodiment, and is a free standing device that is set on a generally flat surface in another embodiment.

**[00116]** In one embodiment, docking station 122 comprises indicator lights and control power switch (not illustrated) in addition to status display 288, power pack chargers 124a and 124b, and concentrator dock 126. Indicator lights provide information to patient P utilizing oxygen concentrator 100. Indicator lights will indicate if oxygen concentrator 100 is functioning properly, requires maintenance, or has failed. Control switch is a master switch for supplying or terminating power or controlling the setting of flow for oxygen concentrator 100. Status display 128 is an LED, LCD or similar digital display that can be used to indicate various information to patient P such as time of day, time oxygen concentrator 100 has been docked, time of recharging for the power pack, or similar information.

**[00117]** Docking station 122 also contains power pack chargers 124a and 124b and concentrator dock 126. Docking station 122 contains a power cord (not illustrated) available to



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plug into a wall socket or a similar power pack. Docking station 122 converts AC power to recharge power pack 108 in power pack chargers 124a and 124b. Power pack chargers 124a and 124b contain contacts that are used to transfer power to power pack 108 while recharging. Alternatively, a power pack 108 placed in charger 124a or 124b is inductively coupled to recharge the power pack 108. Similarly, power is provided to oxygen concentrator 100 itself while on docking station 122, and to recharge of the power pack 108 (see FIG. 4) still attached to the oxygen concentrator 100. Concentrator dock 126 is shaped to provide a place to set oxygen concentrator 100 while docked, as well as facilitate easy removal of oxygen concentrator 100 for ambulatory use.

**[00118]** In one embodiment, docking station 122 performs several functions with oxygen concentrator 100 docked. First, oxygen concentrator 100 is allowed to run without utilizing power pack 108 while it is docked. Second, a boost setting is available to increase the delivery rate of oxygen while oxygen concentrator 100 is docked. Boost switch 272 is located on user interface 114 (See FIG. 13). In an alternate embodiment, a boost switch is located on docking station 122. Upon removal of oxygen concentrator 100 from docking station 122, the boost setting is removed and oxygen concentrator 100 operates at a set delivery rate in either a continuous or pulse mode.

**[00119]** Oxygen concentrator 100 contains flow setting switch 270 (FIG. 13), and a mode switch (not illustrated). The mode switch allows patient P to select continuous or pulse flow. In one embodiment, patient P is allowed to adjust the setting of oxygen concentrator 100 only while docked. That is, patient P can reprogram by changing a pulse setting (e.g., from 2 to 3), or continuous flow mode (e.g., from 1.0 to 1.5 liters per minute), only while oxygen concentrator 100 is on docking station 122. Patient P wishing to adjust settings will be required to hold the control switch while adjusting the flow setting dial or mode switch. In another embodiment, docking station 122 contains a switch automatically activated by placing oxygen concentrator 100 in docking station 122 which allows patient P to adjust flow setting. The requirement that settings can only be changed during docking prevents accidental switching of the flow mode of oxygen concentrator 100 during ambulatory use. For example, there is no change in flow if flow setting switch 130 is bumped, which would normally increase oxygen flow. If oxygen concentrator 100 can only be reprogrammed in docking station 122, oxygen concentrator 100 will remain in the preset mode set at docking station 122 and will not increase or decrease flow



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by a change of the setting. In one embodiment, the flow setting switch and mode switch are located on a user interface located directly on docking station 122.

**[00120]** Another function of docking station 122 is to provide diagnostic features of the system. Docking station 122 may indicate expected battery life, adsorbent column life, or pump malfunctions through the use of indicator lights, or status display 128, or a combination of both. Alternatively, these items are located on user interface 114, or at a combination of locations of user interface 114 and docking station 122. For example, battery life indicator 142 is located directly on power pack 108 that comprises the battery itself, and battery problem warning light 274 is on user interface 114 also. Similarly, adsorbent cartridge warning light 276 is located on user interface 114, but may also be on either the cartridge module 116 or docking station 122 as well.

Concentrator Efficiency

**[00121]** Oxygen concentrator 100 can produce a stream of product gas containing a range of 85-95 percent oxygen which provides up to 5 liters per minute pulsed equivalent of product gas. By utilizing vacuum swing adsorption, the separation process phases are all performed at less than 1 atm.

**[00122]** Utilizing a vacuum to exhaust unwanted gas from adsorbent columns 130a-130c improves efficiency of the oxygen concentrator 100. Less power is required than pressure swing adsorption (PSA) or vacuum-pressure swing adsorption (VPSA), which results in a smaller battery and thus a lighter weight product. Oxygen concentrator 100 as disclosed weighs less than 3 pounds (1.4kg) and occupies less than 1 liter of volume. Also, the efficiency of the system allows for oxygen concentrator 100 to operate for at least three hours while producing up to 5 liters per minute pulsed equivalent of product gas without requiring patient P to attend to the unit, e.g. changing the battery. Further, the low energy consumption causes less heat transfer. The product gas is discharged from the separation system at a temperature of $\pm$ six degrees Celsius from that of the ambient air. This eliminates the need for heat exchangers which add to the overall weight and reduces system efficiency. The amount of heat generated causes no discomfort to patient P wearing and utilizing the oxygen concentrator 100. Also, upon starting oxygen concentrator 100, the flow of product gas will increase from 21 percent oxygen (ambient) to 85 percent or more oxygen in under two minutes.



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**[00123]** Improvements over the prior art are attained by regulating the device to only separate the amount of oxygen needed by patient P at any given time. The prior art separates a flow of oxygen and delivers that rate to patient P as a steady flow. Patient P is only inhaling this oxygen during about 1/3 of the normal breathing cycle. Within the inhalation portion of the breathing cycle, the volume of gas inhaled last stays in the dead space of the airways and is not presented to the alveoli. Therefore if oxygen is dispensed to patient P only during the early part of inhalation, less than 1/3 the steady flow is actually required. Moreover, prior art devices do not adjust the flow based on a patient P=s needs, but operate at the same steady flow. The present concentrator slows down its entire cycle rate producing only the amount of oxygen needed. Thus, oxygen concentrator 100 retains a high oxygen recovery percentage at all product flow rates while minimizing energy consumption and maximizing adsorbent life. Patient P=s actual needs vary with real time changes in activity. This causes a corresponding variation in breathing rate. Oxygen concentrator 100 tracks patient P=s breathing rate and adjusts oxygen separation and delivery rates proportionally. In combination, these two features allow oxygen concentrator 100 to separate oxygen only at the rate it is being consumed, resulting in a reduction in the amount of oxygen needing to be separated for patient P.

**[00124]** Another improvement over the prior art involves reducing the waste of separated oxygen in the various adsorb and desorb cycle phases. This is typically referred to as maximizing product recovery. The primary system components become larger or smaller as the amount of oxygen separated increases or decreases. Therefore, a dramatic reduction in size and weight of the concentrator requires use of as much separated oxygen as possible by delivering it to patient P rather than losing it to the waste stream. The prior art works by using the Skarstrom cycle well known to those skilled in the art.

**[00125]** During one phase of the Skarstrom cycle in PSA or VPSA, air is pumped into one end of a column of adsorbent pressurizing it above atmospheric pressure while oxygen is flowing out of the opposing end. Nitrogen is being adsorbed as the MTZ propagates toward the oxygen outlet end of the column. This phase is terminated before the MTZ breaks through into the oxygen stream so that oxygen purity is not diluted by the nitrogen rich air trailing the MTZ. If it is terminated earlier than necessary to maintain purity there will be substantial separated oxygen left in the column in front of the MTZ that is not passed to the patient. During the next cycle phase the column pressure is reduced to a lower cycle pressure desorbing the nitrogen that was



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adsorbed during the separation phase and it is passed to the waste stream. Some of the oxygen left in the column at higher pressure will also be passed to the waste stream as gas flows from the column when pressure is reduced. Recovery of separated oxygen can therefore be maximized by stopping the previous separation phase just short of breakthrough, leaving minimal oxygen in the column to be lost to the waste stream during the reduced pressure evacuation phase. The position of the MTZ needs to be accurately known to terminate the separation phase for optimal recovery without compromising purity. This position cannot be accurately estimated because its propagation rate is a function of many variables including product oxygen flow rate, high and low cycle pressures, temperature, adsorbent water content and the amount of other contaminants accumulated in the adsorbent. Prior art systems stop the separation phase well short of breakthrough to encompass worst case operating conditions without sacrificing purity and thereby waste separated oxygen in the evacuation phases during most typical non-worst case operating conditions.

**[00126]** Oxygen concentrator 100 determines the position of the MTZ just prior to breakthrough and terminates the flow from outlet 134 for the remainder of the feed phase or adjusts the motor speed, as previously described. Additional oxygen is left in the column at the end of a feed phase and is wasted during the evacuation phases. This is oxygen adsorbed by the adsorbent combined with oxygen present in the interstitial and dead spaces of the adsorbent and column. All adsorbents used in oxygen separators adsorb nitrogen, and also oxygen to some extent. The adsorbent used in oxygen concentrator 100 presents a very high ratio of adsorbed nitrogen to adsorbed oxygen. As the amount of oxygen adsorbed is minimized through the choice of an adsorbent with a low affinity for oxygen, the amount of adsorbent needed to separate a given amount of nitrogen during a separation phase will decrease as its affinity for nitrogen increases. The less adsorbent needed to adsorb a given amount of nitrogen, the less adsorbent there is to adsorb oxygen and the smaller the column can be with less interstitial and dead space.

**[00127]** For example, a LiLSX adsorbent referred to as Oxysiv MDX from UOP Corporation has a very high ratio of adsorbed nitrogen to adsorbed oxygen in the operating pressure range of oxygen concentrator 100. The Skarstrom cycle of the prior art uses a purge phase in which separated oxygen is fed back into the product end of the column while nitrogen rich gas is passing out of the opposing end of the column into the waste stream as the pressure



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transitions to the lower cycle pressure. While this purge can enhance product purity, some of the purge oxygen passes all the way through the column and is lost to the waste stream. Oxygen concentrator 100 using VSA achieves a measured 60% oxygen recovery rate, compared to a typical recovery rate of 30% for the prior art utilizing PSA.

**[00128]** Another improvement over the prior art concerns the choice of adsorbent and operating pressure range. The energy required by the separation process directly defines the weight and size of major components such as the battery, motor and gas pump of a concentrator. Minimizing the amount of adsorbent minimizes the amount of energy needed to separate a given amount of oxygen. Each adsorbent has a characteristic pair of isotherms that show the amount of oxygen and nitrogen a given mass of adsorbent will hold at equilibrium over a range of pressures and vacuums for these gasses at a constant temperature. The cycle phases of the system necessarily include the pumping of gas contained in volumes of adsorbent to produce a change in nitrogen partial pressure between a chosen higher pressure and a chosen lower pressure. The pneumatic energy a pump must deliver in the process of cycling a given volume of gas between a higher and a lower level is in direct proportion to the volume of gas pumped multiplied by the difference between the high and low vacuum levels. The isotherms for various adsorbent candidates specify the amount of nitrogen contained in a fixed mass of adsorbent at a fixed temperature as a function of nitrogen partial pressure.

**[00129]** An example of the isotherm for the LiLSX adsorbent Oxysiv MDX along with the isotherm for a typical 13X type adsorbent used in the prior art is shown in FIG 19. Having minimized the amount of oxygen needed to be separated from air and having maximized the recovery percent of oxygen as previously disclosed, along with knowing the percentage of oxygen present in air prescribes a specific minimum amount of air that must be moved into the system to produce the needed oxygen. This minimum amount of air minus the maximized separated oxygen must pass out of the system as a minimized volume of waste gas. Oxygen concentrator 100 acts to minimize the flow rates of the air feed stream and the waste stream. This flow must be pumped across a pressure difference defined by the choice of high and low operating pressures requiring a pumping energy that is proportional to both the flow rate and the pressure difference. The gas streams are pumped into or out of the adsorbent during each complete cycle to produce the needed swing in pressure between high and low cycle pressure levels allowing the separation of nitrogen from oxygen. Minimizing the amount of gas being



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pumped through the system reduces the pumping energy in proportion to reductions in the difference between chosen high and low pressure points that the gas must be pumped across. The isotherm for nitrogen shows that nitrogen is transferred in or out of the adsorbent with the smallest change in pressure where the slope of the isotherm is the steepest. Using typical PSA, a ratio of high to low pressure levels in these systems needs to be 3:1 or greater to maintain the desired oxygen purity. Lower pressure ranges, i.e. sub-atmospheric or vacuum ranges used in VSA, allow this ratio to be maintained with less total difference between the high and low pressure levels.

**[00130]** For example, prior art operates between 1 and 3 atmospheres for a 3:1 ratio and a pressure difference between high and low levels of 2 atmospheres. Oxygen concentrator 100 using VSA operates between 0.3 atmospheres and 1 atmosphere. A ratio of about 3.3:1 is achieved with a pressure difference of only 0.7 atmospheres. Operating on this range of the isotherm as seen in FIG. 19 allows just as much nitrogen to be passed in and out of the LiLSX adsorbent with a 0.7 atmosphere pressure range as a PSA system does with 13X adsorbent and a 2.0 atmosphere pressure range. The LiLSX adsorbent allows a cycle pressure range that is nearly 1/3 that of a PSA system with a proportional reduction in pumping energy.

**[00131]** Oxygen concentrator 100 is a quiet device. When oxygen concentrator 100 is running, it produces a noise level in the range of ten to thirty decibels. Further, with the compact size of the parts, vacuum pump 144 is running continuously and there is very little vibration to affect a person using it docked or wearing it as an ambulatory device. The device of the present invention with the described components weighs less than three pounds (1.36 kg). The compact size (less than about61 cu. in. (1000 cc)) allows for easy portability. Similarly, the small size does not disrupt counter space or storage when used at home. The device does give off some heat, however the outer case is less than 6 degrees Celsius higher than ambient when oxygen concentrator 100 is running on battery power. The device may emit more heat while it is docked and operating on AC power to charge the power pack 108, but is still less than 15 degrees Celsius above ambient.

**[00132]** Based on the foregoing embodiments, the efficiency of the concentrator can be determined. One measure of efficiency is the ratio of oxygen produced to the amount of adsorbent material used to obtain the oxygen, represented by the following:



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Qp = Liter/min $O_2$ produced

Madsorbent = Kg of Adsorbent Material

for example, the disclosed embodiments include adsorbent columns 130a-130c, with each column containing 15 cubic centimeters (cc) of adsorbent material with a density of 0.66gm/cc. That is:

$$(3columns)\left(\frac{15cc}{column}\right)\left(\frac{0.66gm}{cc}\right) = 30gms$$ for the system.

The following flow rates (Qp) were obtained by the above disclosed concentrator:

Qp max = 1.5 L/min

Qp min = 0.14 L/min

**[00133]** This results in a range for kilograms of adsorbent material to oxygen flow rate of:

$$0.020 < \frac{Madsorbent}{Qp} < 0.214$$

**[00134]** Similarly, flow rates (Qp) were determined for a system that contains three adsorption columns, each column containing 15cc of adsorbent material. The separation completed in a range of .3 atm to .95 atm. Values were calculated for breakthrough time, work, battery life, and flow rate. The volume of gas contained in a column at the end of a feed phase was 150cc. These constants were used to determine the following measures of efficiency:

Work per evacuation cycle or pneumatic power requirements were determined based on the following calculations:

W (work) = (volume moved) * (vacuum differences);

The vacuum differences are calculated as the vacuum pump is continuously changing gas out, and as vacuum progresses to end point. From this:

$V_H$ = Vacuum upper level

$V_L$ = Vacuum lower level

Vol = Volume of gas in the column at end of feed phase

$W = Vol*(V_H-V_L)*(1+(V_H/(V_H-V_L))*\ln(V_L/V_H)+\ln(V_H/V_L)$



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Inserting the above constants and converting to joules (multiply by 100.32 to get L*atm to joules) yields:

**[00135]** W = 4.81 joules

**[00136]** Thus, 4.81 joules is required to evacuate the gas which desorbs during the evacuation phase. From experimentation, the following flow rates (LPM is liters per minute) and cycle times were recorded:

| | |
|---|---|
| Qp (flow rate) | Low = .14 LPM |
| | Med = .720 LPM |
| | High = 1.5 LPM |
| Cycle time | Low = 5.6 sec. |
| | Med = 1.12 sec. |
| | High = .54 sec. |

Power consumption can be determined by calculating work divided by the time of the cycle.

Low flow power = 4.81 joule/5.6 sec = .85 watts

Medium Flow power = 4.81 joule/1.12 sec = 4.29 watts

High flow power = 4.81 joule/.54 sec = 8.9 watts

**[00137]** From the above, a measure of energy consumed to the flow rate can be made and used as an indicator of the system efficiency:

$$Low: \frac{.85w}{.14LPM} = 6.07w / LPM$$

$$Medium: \frac{4.29w}{.72LPM} = 5.95w / LPM$$

$$High: \frac{8.9w}{1.5LPM} = 5.93w / LPM$$

**[00138]** Another measure of efficiency is the ratio of mass of the power pack (Mpowerpack) compared to the amount of oxygen produced (Qp) over time:



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$$\frac{Mpowerpack}{QpT(time)}$$

The following constants are used in the calculation: the battery cell is a type 18650 lithium ion battery with 7.4 watts-hrs, measuring 42 g; motor efficiency is 90 percent; and vacuum pump efficiency is 80 percent.

Pneumatic work = 6W/L/min. Thus,

Electric power = $\frac{6W/LPM}{(.9)(.8)} = 8.3W/LPM$

Battery mass compared to energy consumption is:

$$\frac{42g}{7.4(watt)(hr)}\left(\frac{1Kg}{1000g}\right)\frac{8.3watt}{LPM} = .047\frac{Kg}{LPM(HR)}$$

Total battery mass for the power pack can be determined from this equation. For example, if a patient requires the concentrator to run for four hours at setting of A3" and takes 20 breaths per minute (the medium flow rate):

$$(4hr)*\frac{.047kg}{LPM(hr)}.72LPM = .135kg$$

The mass of the batteries needed is 0.135 Kg. Assuming each battery cell is 42g as previously stated, the number of batteries for the power pack can be calculated:

$$.135kg\left(\frac{1000g}{1kg}\right)\frac{\text{battery cell}}{42g} = 3.2battery\ cells$$

**[00139]** Although the present invention has been described with reference to preferred embodiments, workers skilled in the art will recognize that changes may be made in form and



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detail without departing from the spirit and scope of the invention. For example, larger flow rates may be achieved by scaling the concentrator components to achieve desired flow rates at the disclosed efficiencies.



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## CLAIMS

1. A portable oxygen concentrator, comprising:

a housing;

a separation cartridge selectively removable from the housing, the separation cartridge comprising a case at least partially defining an outer volume of the separation cartridge, an inlet, an outlet oriented in a common direction with the inlet, and a bed of adsorbent material configured to preferentially adsorb nitrogen, the case at least partially surrounding the bed of adsorbent material;

a power pack removable from the portable oxygen concentrator;

a pump electrically coupled to the power pack, the pump operable to cause a flow of fluid to move relative to the adsorbent material;

a user interface configured to allow a patient to adjust and monitor operation of the portable oxygen concentrator;

a carrying device for transporting the portable oxygen concentrator;

wherein the housing is configured to at least partially surround the case of the separation cartridge.

2. The portable oxygen concentrator as recited in claim 1, wherein the bed of adsorbent material is disposed along an axis, one of the inlet and the outlet is disposed along the axis, and the other of the inlet and the outlet is spaced-apart from the axis.

3. The portable oxygen concentrator as recited in claim 1, wherein the portable oxygen concentrator weighs less than 3 pounds.



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4. The portable oxygen concentrator as recited in claim 1, wherein the power pack is a battery having a weight of less than 0.7 pounds.

5. The portable oxygen concentrator as recited in claim 1, wherein the bed of adsorbent material comprises a bed of zeolite which provides preferential adsorption of nitrogen.

6. The portable oxygen concentrator as recited in claim 5, wherein the zeolite is an LiLSX adsorbent.

7. The portable oxygen concentrator as recited in claim 1, wherein the carrying device is a belt.



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8. A portable oxygen concentrator, comprising:

a housing;

a separation cartridge selectively removable from the housing, the separation cartridge comprising an inlet, an outlet, and a bed of adsorbent material configured to preferentially adsorb nitrogen; and

a power pack removable from the portable oxygen concentrator.

9. The portable oxygen concentrator as recited in claim 8, wherein:

the separation cartridge further comprises a case at least partially defining an outer volume of the separation cartridge, and

the case at least partially surrounds the bed of adsorbent material.

10. The portable oxygen concentrator as recited in claim 9, wherein:

the housing is configured to at least partially surround the case of the separation cartridge.

11. The portable oxygen concentrator as recited in claim 8, wherein the inlet and the outlet are directed in a common direction.

12. The portable oxygen concentrator as recited in claim 8, wherein:

the bed of adsorbent material is disposed along an axis,

one of the inlet and the outlet is disposed along the axis, and

the other of the inlet and the outlet is spaced-apart from the axis.



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13. The portable oxygen concentrator as recited in claim 8, wherein the portable oxygen concentrator weighs less than 3 pounds.

14. The portable oxygen concentrator as recited in claim 8, wherein the power pack is a battery having a weight of less than 0.7 pounds.

15. The portable oxygen concentrator as recited in claim 14, further comprising a pump electrically coupled to the power pack, the pump operable to cause a flow of fluid to move relative to the adsorbent material.

16. The portable oxygen concentrator as recited in claim 8, wherein the bed of adsorbent material comprises a bed of zeolite which provides preferential adsorption of nitrogen.

17. The portable oxygen concentrator as recited in claim 16, wherein the zeolite is an LiLSX adsorbent.

18. The portable oxygen concentrator as recited in claim 8, further comprising a carrying device for transporting the portable oxygen concentrator.

19. The portable oxygen concentrator as recited in claim 18, wherein the carrying device is a belt.



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20. The portable oxygen concentrator as recited in claim 8, wherein the bed of adsorbent material is one of a plurality of beds of adsorbent material within the separation cartridge.



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# REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR


## ABSTRACT

A removable gas separation cartridge includes a housing having an inlet port, an outlet port, and a bed of adsorbent material. The cartridge is removable from an oxygen concentrator which separates oxygen from ambient air by using an absorption process.




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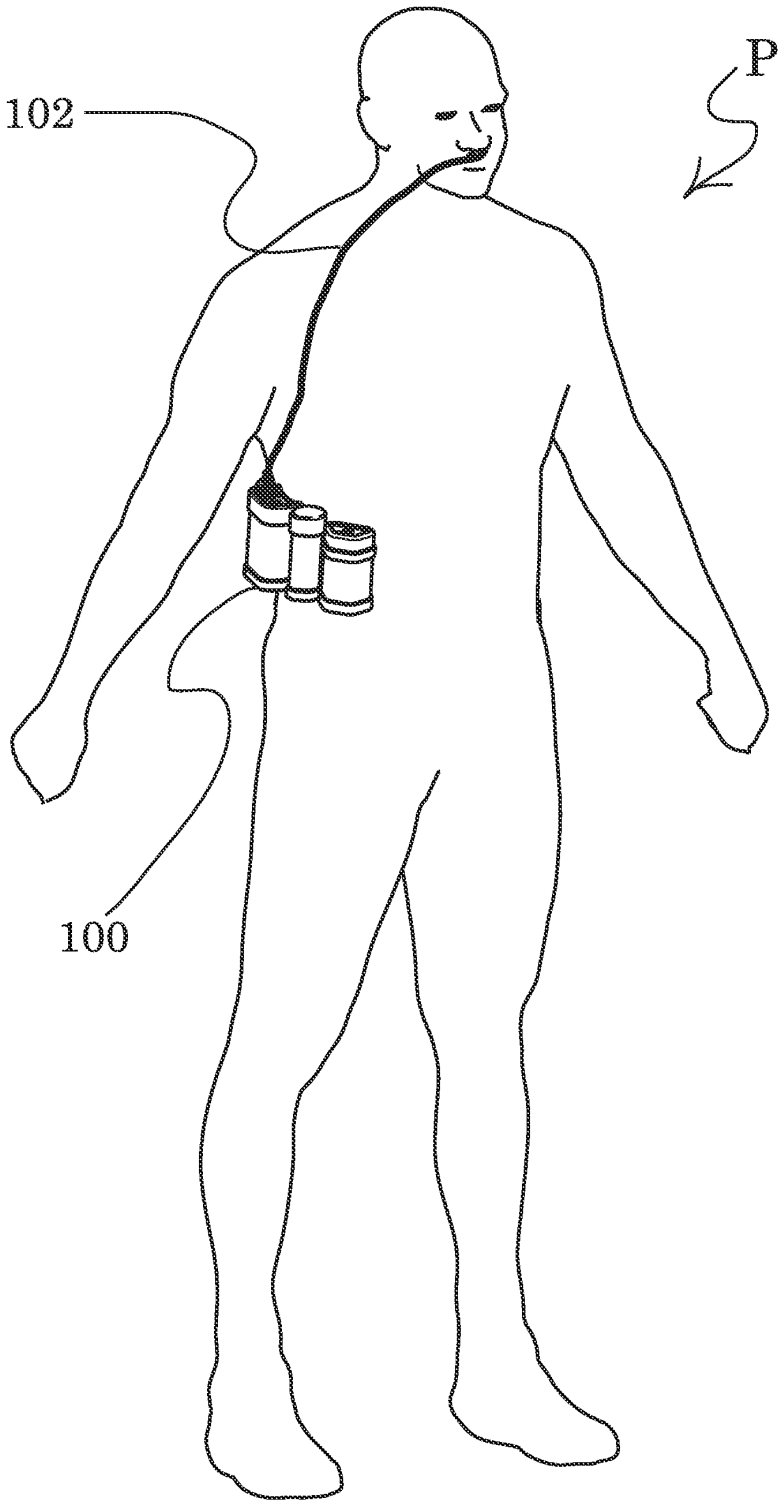



Fig 1

Fig 2

Fig 3

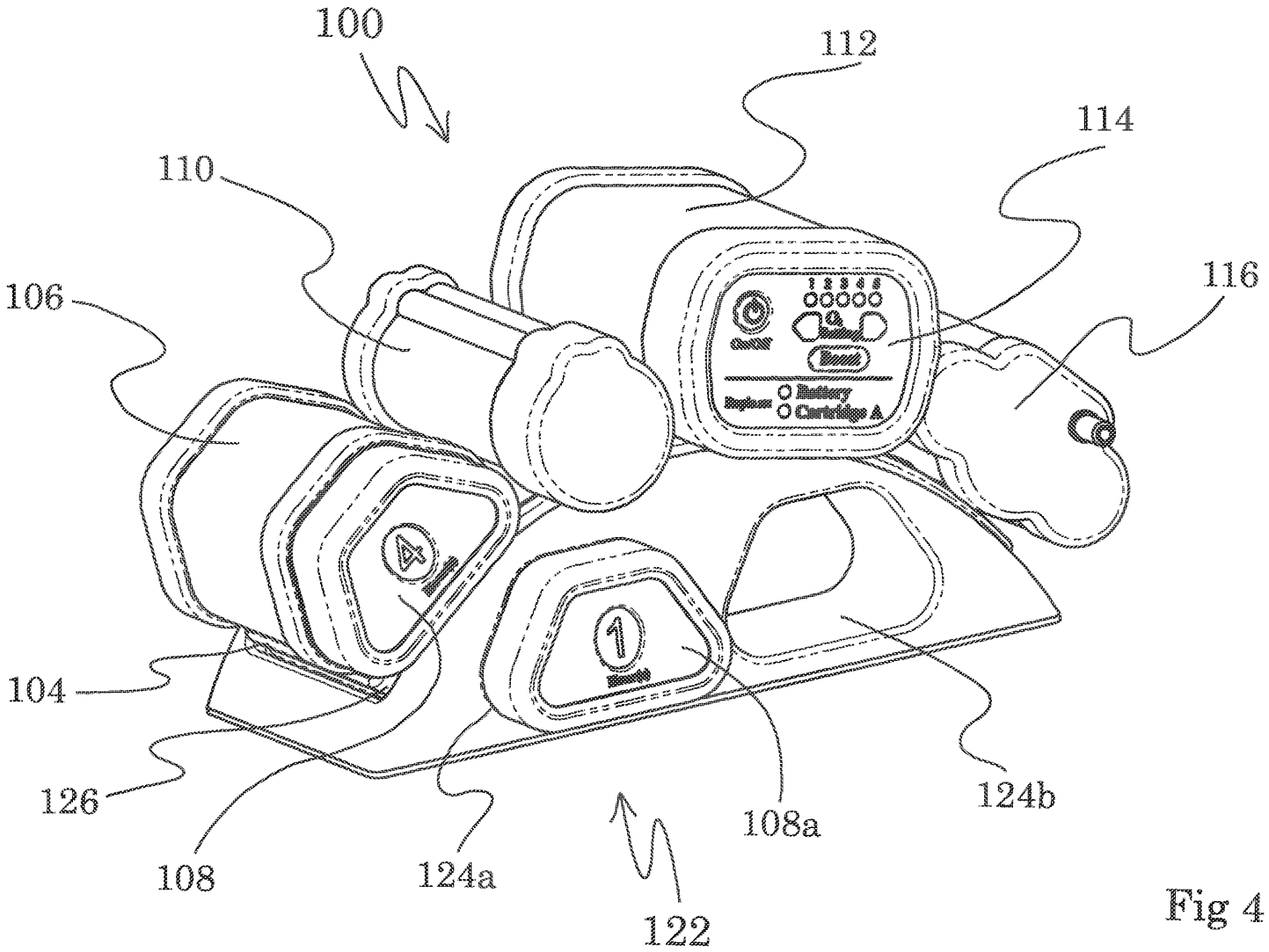



Fig 4

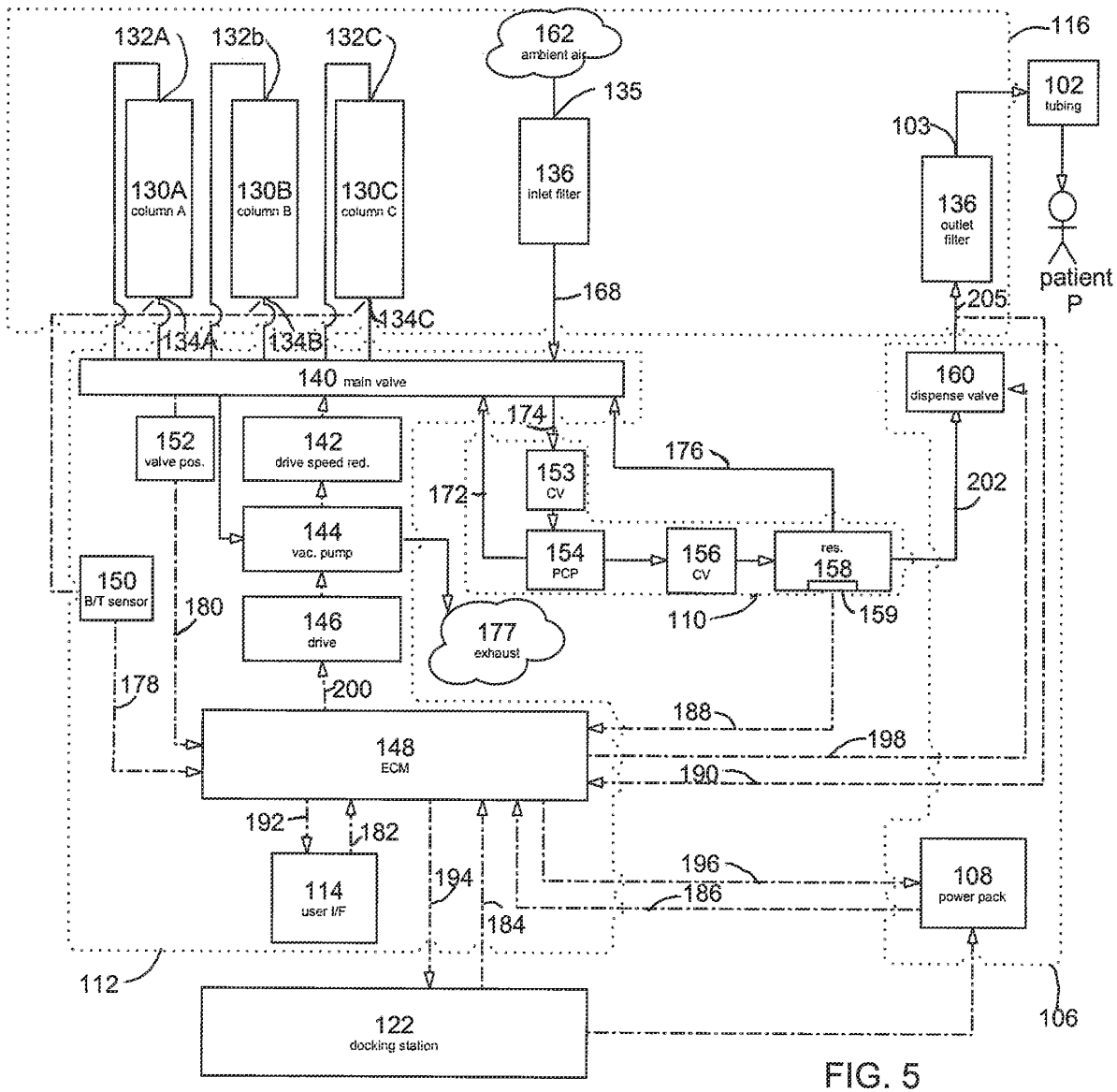

132A
132b
132C
162
ambient air
135
116
102
tubing
103
136
outlet
filter
patient
P
130A
column A
130B
column B
130C
column C
136
inlet filter
168
205
134A
134B
134C
140 main valve
160
dispense valve
152
valve pos.
142
drive speed red.
174
176
153
CV
172
202
144
vac. pump
154
PCP
156
CV
res.
158
150
B/T sensor
180
146
drive
177
exhaust
110
159
178
200
188
148
ECM
198
190
192
182
196
108
power pack
114
user I/F
194
186
184
112
122
docking station
106

FIG. 5

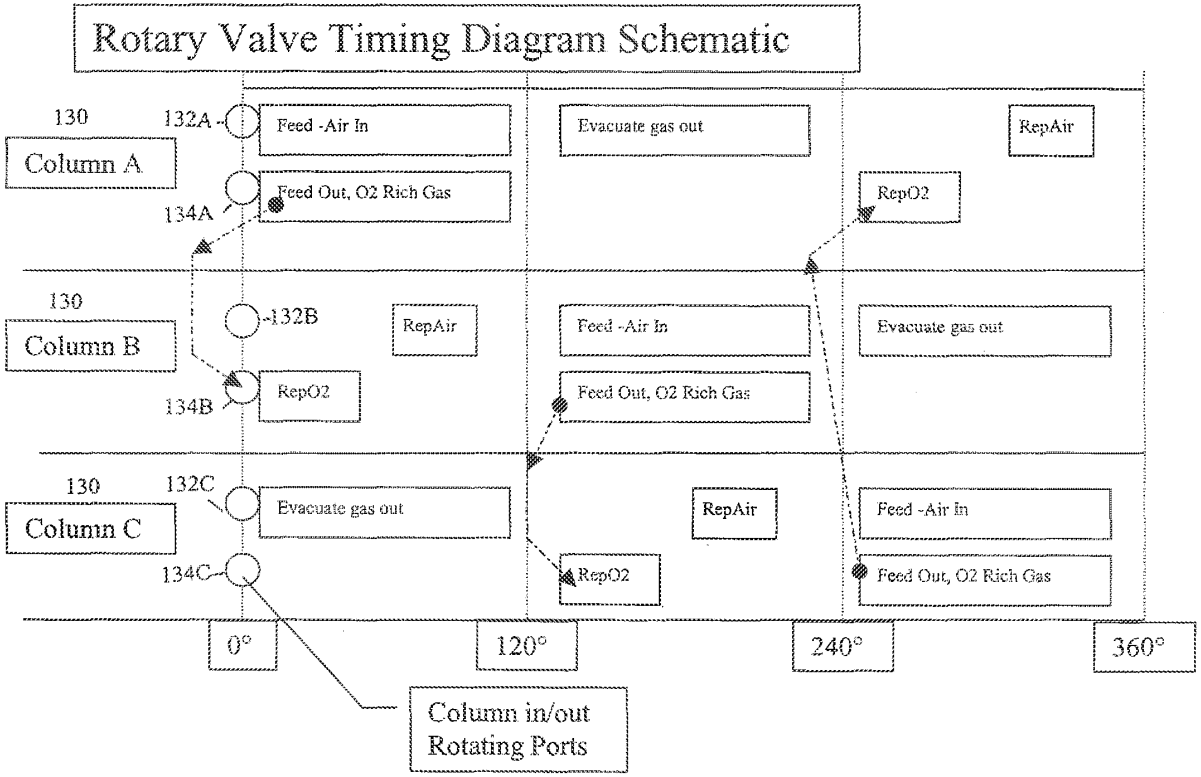

Rotary Valve Timing Diagram Schematic
130
Column A
132A
Feed -Air In
Evacuate gas out
RepAir
Feed Out, O2 Rich Gas
RepO2
134A
130
Column B
132B
RepAir
Feed -Air In
Evacuate gas out
RepO2
Feed Out, O2 Rich Gas
134B
130
Column C
132C
Evacuate gas out
RepAir
Feed -Air In
RepO2
Feed Out, O2 Rich Gas
134C
0°
120°
240°
360°
Column in/out
Rotating Ports

FIG. 6

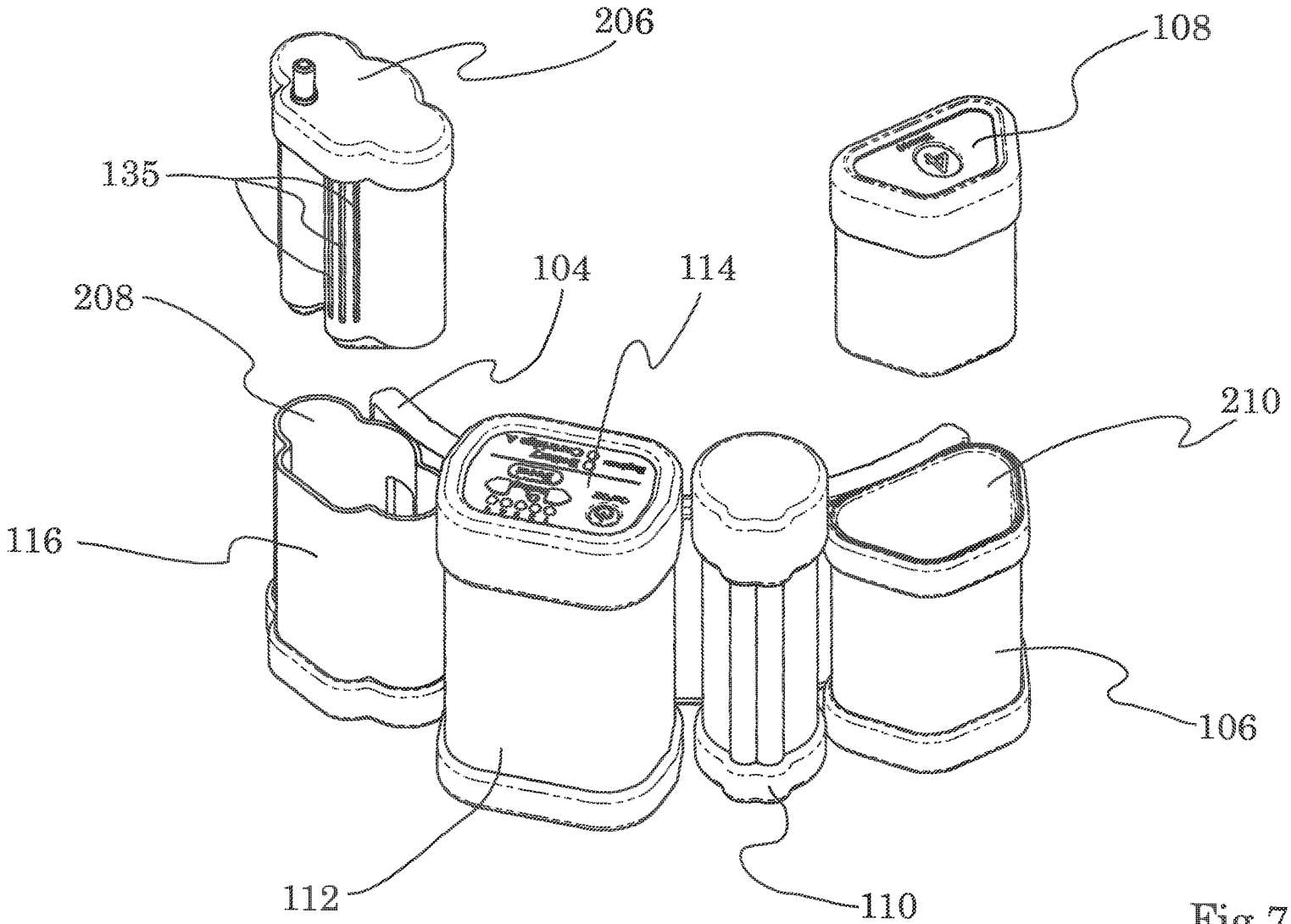



Fig 7

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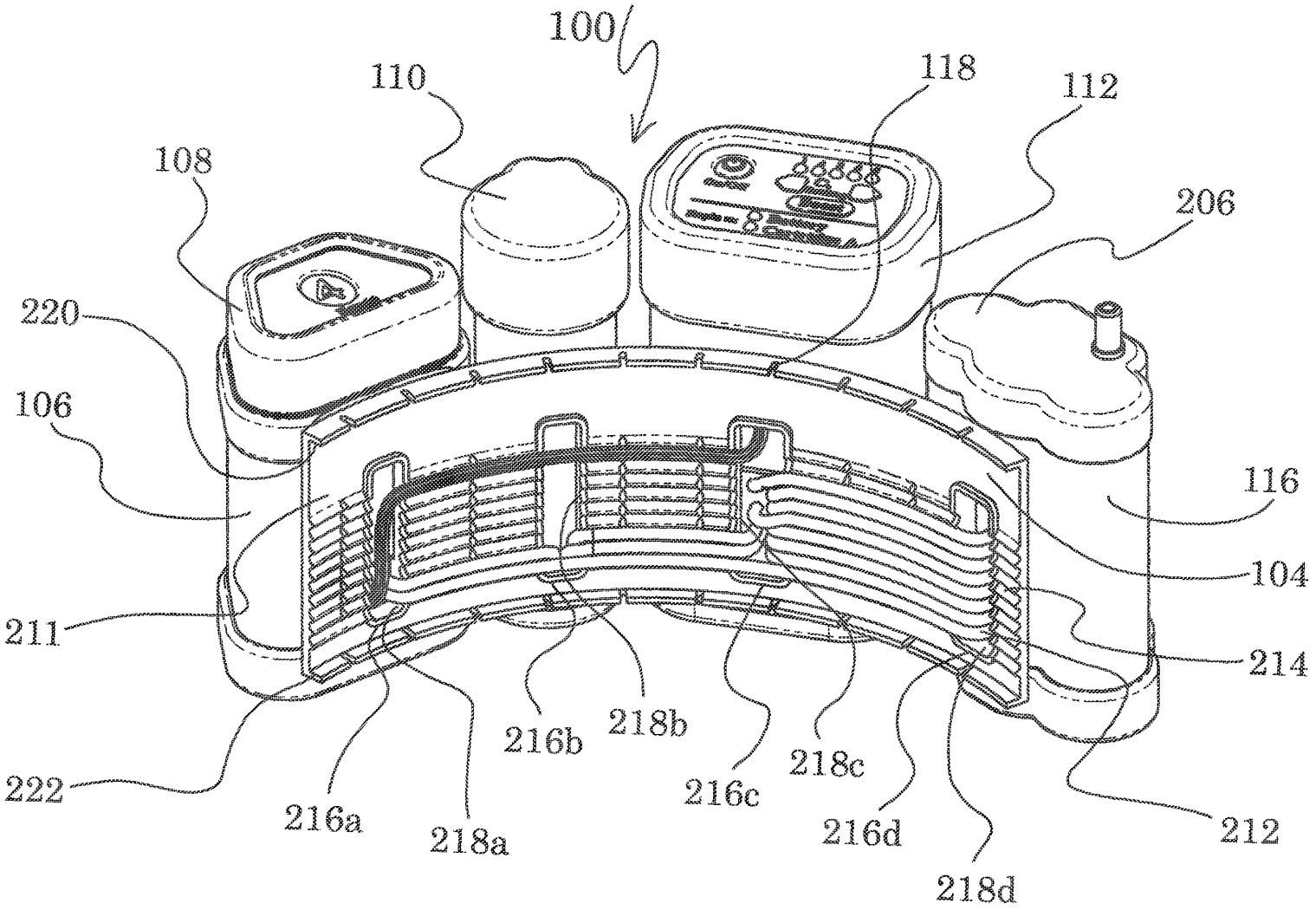

100
110
118
112
108
206
220
106
116
104
211
214
216b
218b
222
216a
218a
216c
218c
216d
212
218d

Fig 8

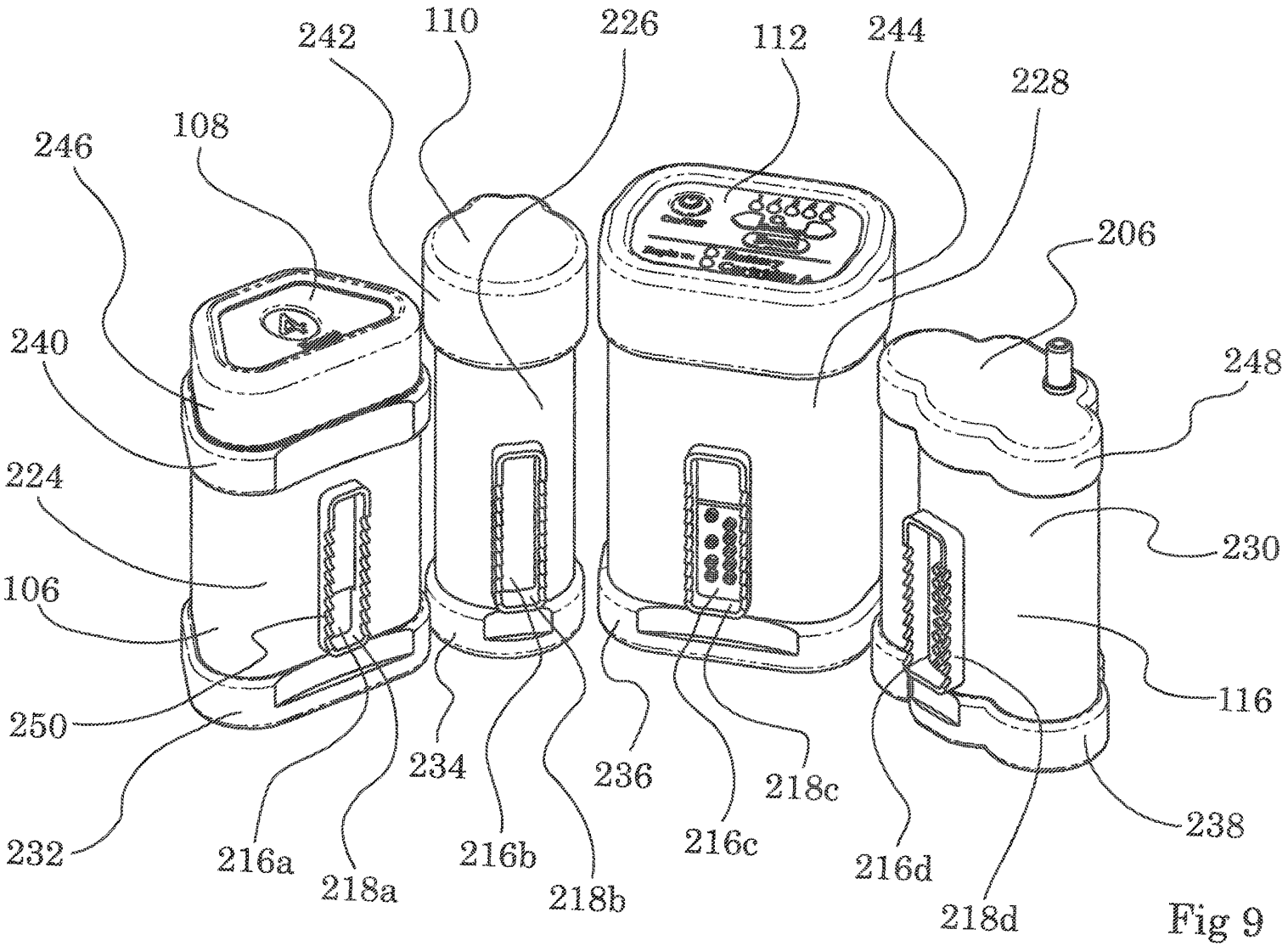

242
110
226
112
244
228
108
246
206
240
248
224
230
106
116
250
234
236
218c
238
232
216a
218a
216b
218b
216c
216d
218d

Fig 9

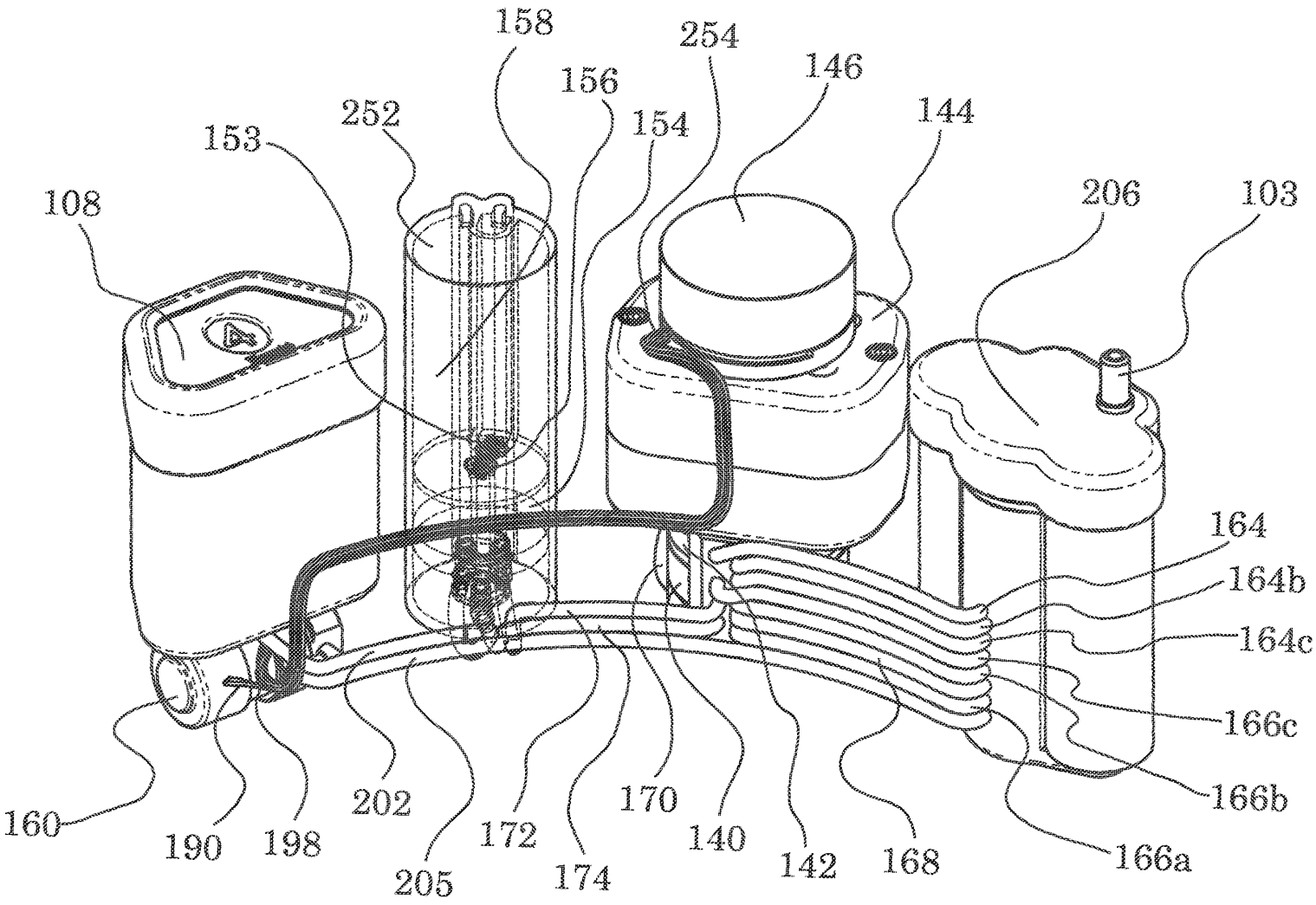

158
254
146
156
252
154
144
153
108
206
103
164
164b
164c
166c
166b
166a
160
190
198
202
205
172
174
170
140
142
168

Fig 10

Fig 11

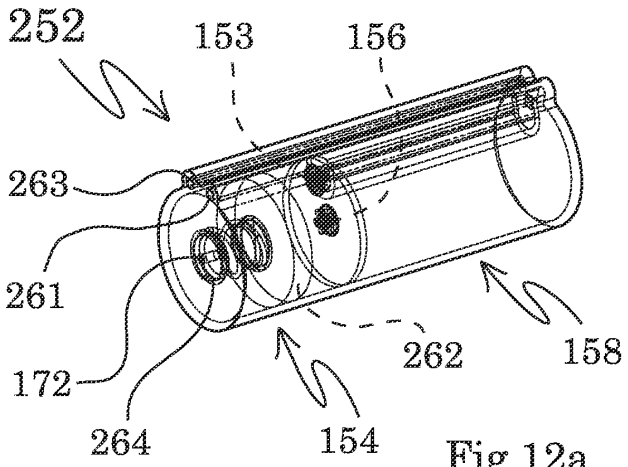
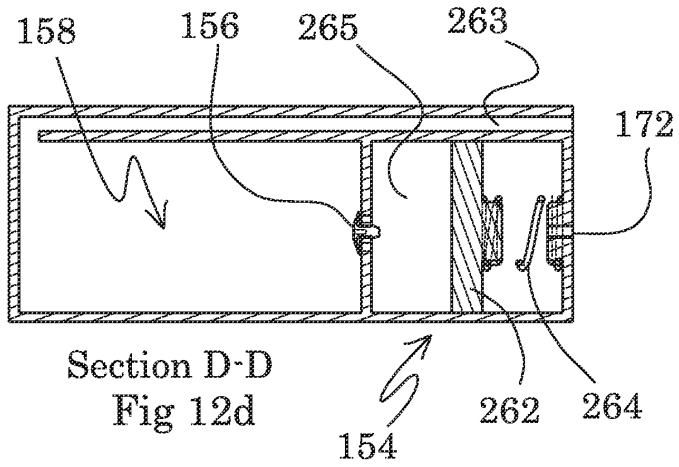



Fig 12a




Section D-D
Fig 12d

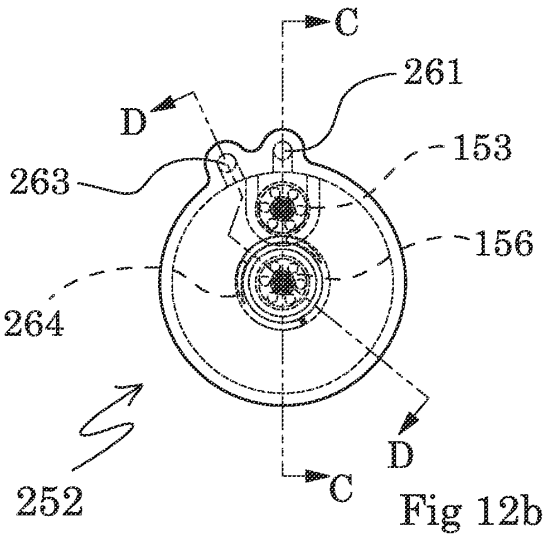
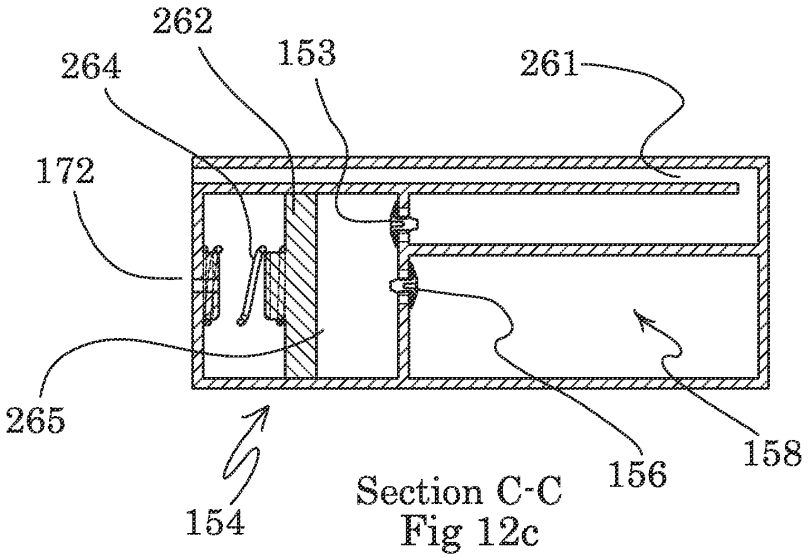



Fig 12b




Section C-C
Fig 12c

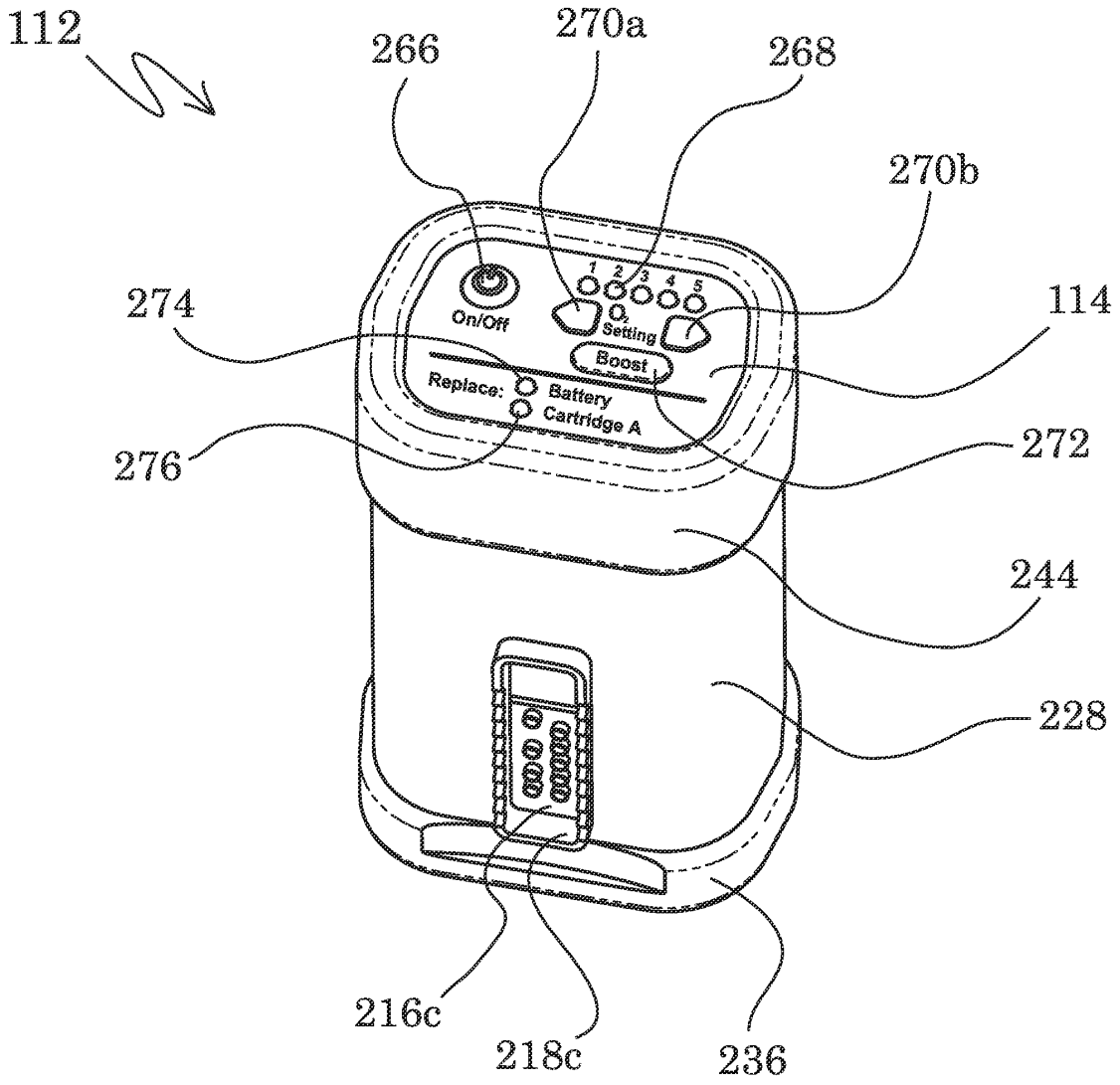

112
266
270a
268
270b
274
114
On/Off
1
2
3
4
5
Setting
Boost
Replace:
Battery
Cartridge A
276
272
244
228
216c
218c
236

Fig 13

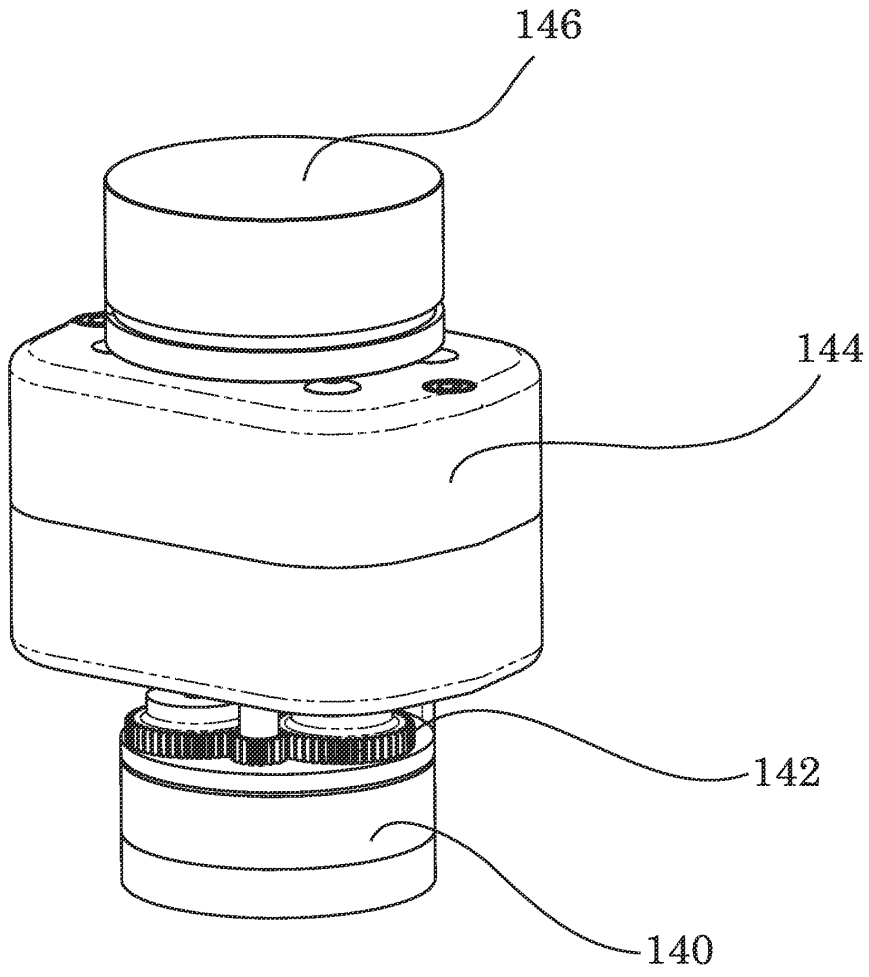



Fig 14

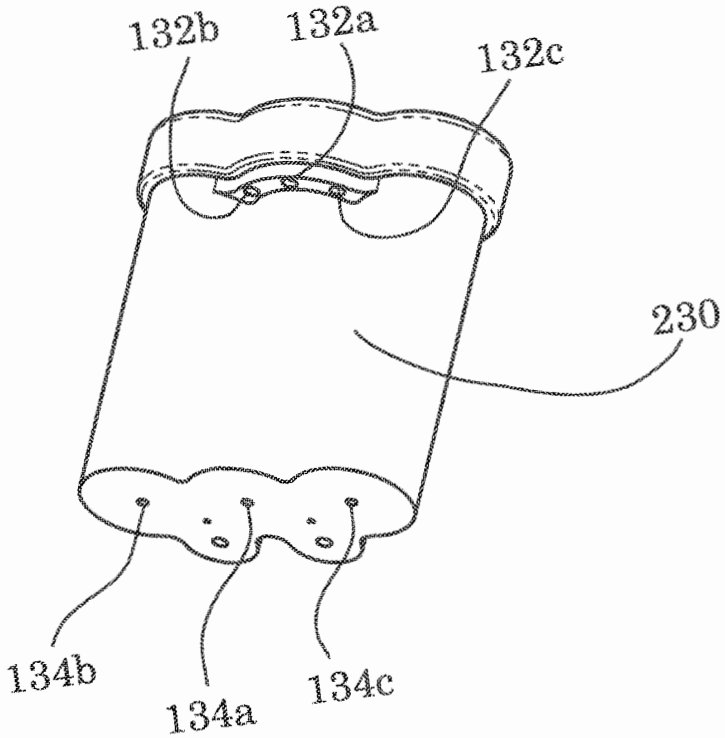



Fig 15

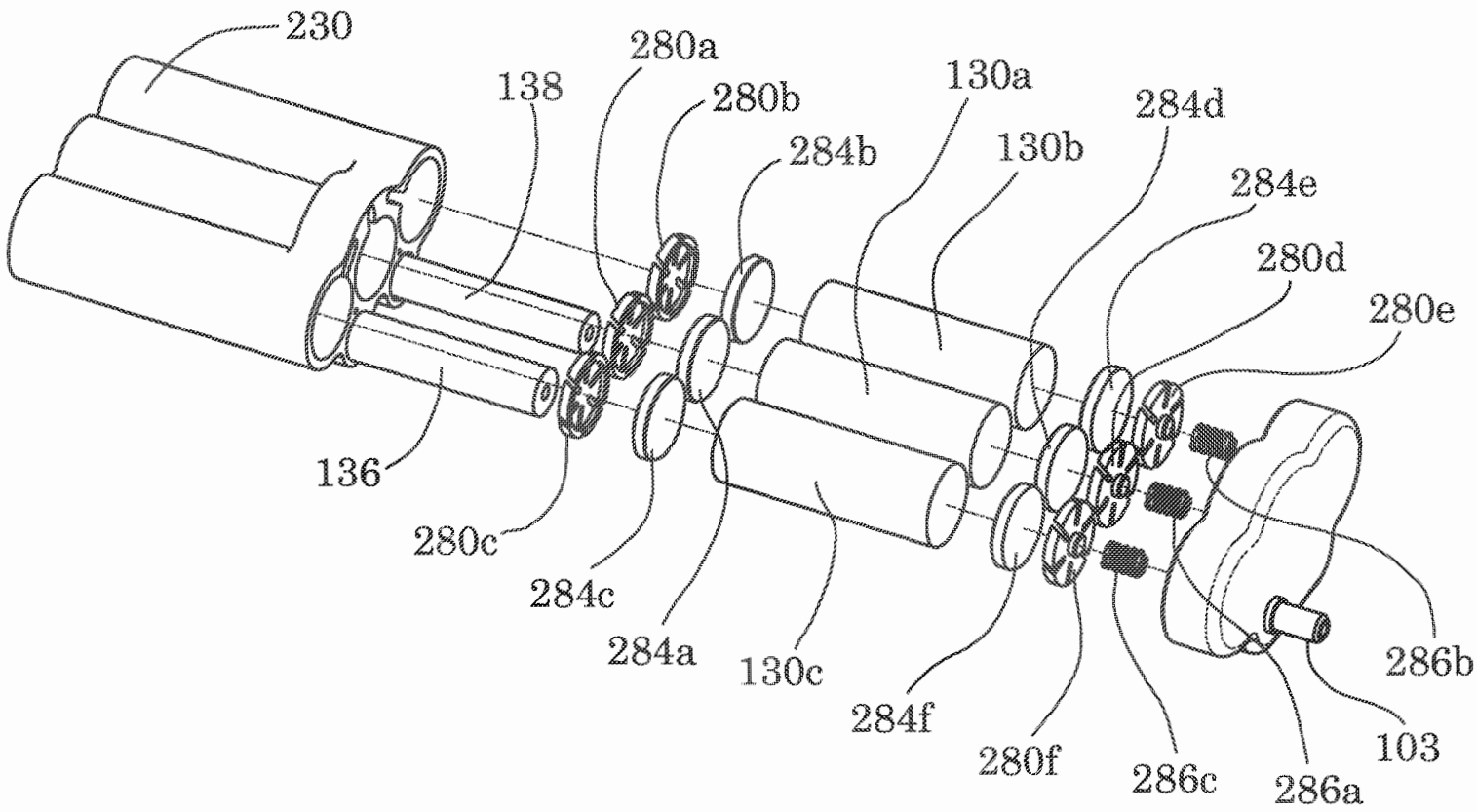

230
138
280a
280b
284b
130a
130b
284d
284e
280d
280e
136
280c
284c
284a
130c
284f
280f
286c
286a
103
286b

Fig 16

Fig 17

Fig 18

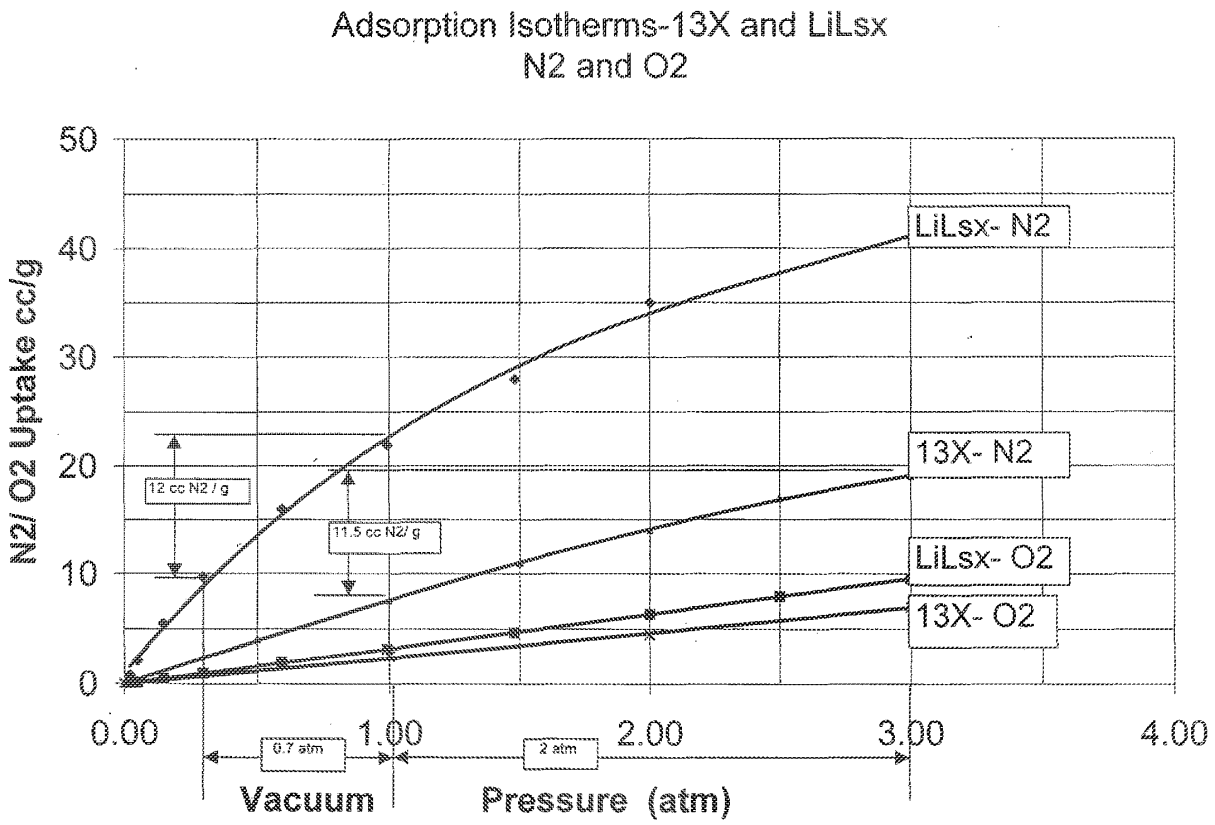

Adsorption Isotherms-13X and LiLsx
N2 and O2
N2/ O2 Uptake cc/g
50
40
30
20
10
0
0.00
1.00
2.00
3.00
4.00
LiLsx- N2
13X- N2
LiLsx- O2
13X- O2
12 cc N2 / g
11.5 cc N2/ g
0.7 atm
2 atm
Vacuum
Pressure (atm)

FIG. 19

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# DECLARATION (37 CFR 1.63) FOR UTILITY OR DESIGN APPLICATION USING AN APPLICATION DATA SHEET (37 CFR 1.76)

| Title of Invention | AMBULATORY OXYGEN CONCENTRATOR |
|---|---|

As the below named inventor, I hereby declare that:

This declaration is directed to:

- ☐ The attached application, or
- ☑ United States application or PCT international application number 13/795,977 filed on March 12, 2013

The above-identified application was made or authorized to be made by me.

I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.

I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than five (5) years, or both.

## WARNING:

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LEGAL NAME OF INVENTOR

Inventor: Theodore W. Jagger Date (Optional) :

Signature: Theodore Jagger

Note: An application data sheet (PTO/AIA/14 or equivalent), including naming the entire inventive entity, must accompany this form. Use an additional PTO/SB/A1A01 form for each additional inventor.

This collection of information is required by 35 U.S.C. 115 and 37 CFR 1.63. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 1 minute to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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V180.12-0026

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| Title of Invention | AMBULATORY OXYGEN CONCENTRATOR |
|---|---|

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This declaration is directed to:

☐ The attached application, or

☑ United States application or PCT international application number 13/795,977 filed on March 12, 2013

The above-identified application was made or authorized to be made by me.

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LEGAL NAME OF INVENTOR

Inventor: Nicholas P. Van Brunt Date (Optional): 5-8-13

Signature: N P Van Brunt

Note: An application data sheet (PTO/AIA/14 or equivalent), including naming the entire inventive entity, must accompany this form. Use an additional PTO/SB/AIA01 form for each additional inventor.

This collection of information is required by 35 U.S.C. 115 and 37 CFR 1.63. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 1 minute to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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- ☑ United States application or PCT international application number 13/795,977 filed on March 12, 2013.

The above-identified application was made or authorized to be made by me.

I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.

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LEGAL NAME OF INVENTOR

Inventor: John A. Kivisto Date (Optional): 4/30/13

Signature:

Note: An application data sheet (PTO/AIA/14 or equivalent), including naming the entire inventive entity, must accompany this form. Use an additional PTO/SB/AIA01 form for each additional inventor.

This collection of information is required by 35 U.S.C. 115 and 37 CFR 1.63. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 1 minute to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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# DECLARATION (37 CFR 1.63) FOR UTILITY OR DESIGN APPLICATION USING AN APPLICATION DATA SHEET (37 CFR 1.76)

| Title of Invention | AMBULATORY OXYGEN CONCENTRATOR |
|---|---|

As the below named inventor, I hereby declare that:

This declaration is directed to:

- ☐ The attached application, or
- ☑ United States application or PCT international application number 13/795,977 filed on March 12, 2013

The above-identified application was made or authorized to be made by me.

I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.

I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than five (5) years, or both.

## WARNING:

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LEGAL NAME OF INVENTOR

Inventor: Perry B. Lonnes Date (Optional):

Signature: Perry B Lonnes

Note: An application data sheet (PTO/AIA/14 or equivalent), including naming the entire inventive entity, must accompany this form. Use an additional PTO/SB/AIA01 form for each additional inventor.

This collection of information is required by 35 U.S.C. 115 and 37 CFR 1.63. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 1 minute to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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Doc Code: PA..

Document Description: Power of Attorney

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NOTE: This form is to be submitted with the Power of Attorney by Applicant form (PTO/AIA/82B or equivalent) to identify the application to which the Power of Attorney is directed, in accordance with 37 CFR 1.5. If the Power of Attorney by Applicant form is not accompanied by this transmittal form or an equivalent, the Power of Attorney will not be recognized in the application.

| | |
|---|---|
| Application Number | |
| Filing Date | |
| First Named Inventor | Theodore W. Jagger |
| Title | REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR |
| Art Unit | |
| Examiner Name | |
| Attorney Docket Number | 67853-012 PUS4 |

**SIGNATURE of Applicant or Patent Practitioner**

| | | | |
|---|---|---|---|
| Signature | /Timothy J. Murphy/ | Date | May 14, 2020 |
| Name | Timothy J. Murphy | Telephone | 248 988 8360 |
| Registration Number | 64,613 | | |

**NOTE**: This form must be signed in accordance with 37 CFR 1.33. See 37 CFR 1.4(d) for signature requirements and certifications.

■ *Total of 1 forms are submitted.

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# POWER OF ATTORNEY TO PROSECUTE APPLICATIONS BEFORE THE USPTO

I hereby revoke all previous powers of attorney given in the application identified in the attached statement under 37 CFR 3.73(c).

I hereby appoint:

[X] Practitioners associated with Customer Number: 26096

**OR**

[ ] Practitioner(s) named below (if more than ten patent practitioners are to be named, then a customer number must be used):

| Name | Registration Number | Name | Registration Number |
|---|---|---|---|
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

As attorney(s) or agent(s) to represent the undersigned before the United States Patent and Trademark Office (USPTO) in connection with any and all patent applications assigned only to the undersigned according to the USPTO assignment records or assignment documents attached to this form in accordance with 37 CFR 3.73(c).

Please change the correspondence address for the application identified in the attached statement under 37 CFR 3.73(c) to:

[X] The address associated with Customer Number: 26096

**OR**

[ ]

| | | |
|---|---|---|
| Firm or individual name | | |
| Address | | |
| City | State | Zip |
| Country | | |
| Telephone | Email | |

Assignee name and address: VBOX, INCORPORATED

**A copy of this form, together with a statement under 37 CFR 3.73(c) (Form PTO/AIA/96 or equivalent) is required to be filed in each application in which this form is used. The statement under 37 CFR 3.73(c) may be completed by one of the practitioners appointed in this form, and must identify the application in which this Power of Attorney is to be filed.**

**SIGNATURE of Assignee of Record**

The individual whose signature and title is supplied below is authorized to act on behalf of the assignee.

| | |
|---|---|
| Signature | Date 4/11/18 |
| Name Robert J Folkes | Telephone 612 355 5202 |
| Title CFO | |

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# STATEMENT UNDER 37 CFR 3.73(c)

Applicant/Patent Owner: Theodore W. Jagger

Application No./Patent No.: _____ Filed/Issue Date: _____

Titled: REMOVABLE CARTRIDGE FOR OXYGEN CONCENTRATOR

VBOX, Incorporated, a corporation

(Name of Assignee) (Type of Assignee, e.g., corporation, partnership, university, government agency, etc.)

states that, for the patent application/patent identified above, it is (choose **one** of options 1, 2, 3 or 4 below):

1. ☑ The assignee of the entire right, title, and interest.

2. ☐ An assignee of less than the entire right, title, and interest (check applicable box):

   ☐ The extent (by percentage) of its ownership interest is _____%. Additional Statement(s) by the owners holding the balance of the interest must be submitted to account for 100% of the ownership interest.

   ☐ There are unspecified percentages of ownership. The other parties, including inventors, who together own the entire right, title and interest are:

   Additional Statement(s) by the owner(s) holding the balance of the interest must be submitted to account for the entire right, title, and interest.

3. ☐ The assignee of an undivided interest in the entirety (a complete assignment from one of the joint inventors was made). The other parties, including inventors, who together own the entire right, title, and interest are:

   Additional Statement(s) by the owner(s) holding the balance of the interest must be submitted to account for the entire right, title, and interest.

4. ☐ The recipient, via a court proceeding or the like (*e.g.*, bankruptcy, probate), of an undivided interest in the entirety (a complete transfer of ownership interest was made). The certified document(s) showing the transfer is attached.

The interest identified in option 1, 2 or 3 above (not option 4) is evidenced by either (choose **one** of options A or B below):

A. ☑ An assignment from the inventor(s) of the patent application/patent identified above. The assignment was recorded in the United States Patent and Trademark Office at Reel 016390, Frame 0364, or for which a copy thereof is attached.

B. ☐ A chain of title from the inventor(s), of the patent application/patent identified above, to the current assignee as follows:

   1. From: _____ To: _____

      The document was recorded in the United States Patent and Trademark Office at

      Reel _____, Frame _____, or for which a copy thereof is attached.

   2. From: _____ To: _____

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This collection of information is required by 37 CFR 3.73(b). The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450**.

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## STATEMENT UNDER 37 CFR 3.73(c)

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☐ Additional documents in the chain of title are listed on a supplemental sheet(s).

☐ As required by 37 CFR 3.73(c)(1)(i), the documentary evidence of the chain of title from the original owner to the assignee was, or concurrently is being, submitted for recordation pursuant to 37 CFR 3.11.

[NOTE: A separate copy (i.e., a true copy of the original assignment document(s)) must be submitted to Assignment Division in accordance with 37 CFR Part 3, to record the assignment in the records of the USPTO. See MPEP 302.08]

The undersigned (whose title is supplied below) is authorized to act on behalf of the assignee.

| /Timothy J. Murphy/ | May 14, 2020 |
|---|---|
| Signature | Date |
| Timothy J. Murphy | 64613 |
| Printed or Typed Name | Title or Registration Number |