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(54) Method for producing gaseous oxygen and a cryogenic plant in which said method can be carried out.

(57) A method for producing gaseous oxygen, preferably at pressures in excess of 35 bars A, by the fractional distillation of air using a conventional double column having a high pressure column (6) and a low pressure column (11). Liquid oxygen is withdrawn from the bottom of the low pressure column (11) and is pressurized to just above the desired product pressure by pump (13). The liquid oxygen is then vapourized in heat exchanger (15) using nitrogen from the top of the high pressure column (6). The nitrogen is compressed in compressor (28) to 41 bars A and in compressors (32 and 34) to 80 bars A. The stream (35) is sub-cooled in heat exchanger 15 and, after expansion at valve (36), the liquid nitrogen is returned to the high pressure column as reflux. The stream (31) is cooled in heat exchanger (15) and is expanded in an expander (40). The gas leaving the expander (40) is used to cool the stream (35) in heat exchanger (15) and is subsequently recycled to the inlet of compressor 28.

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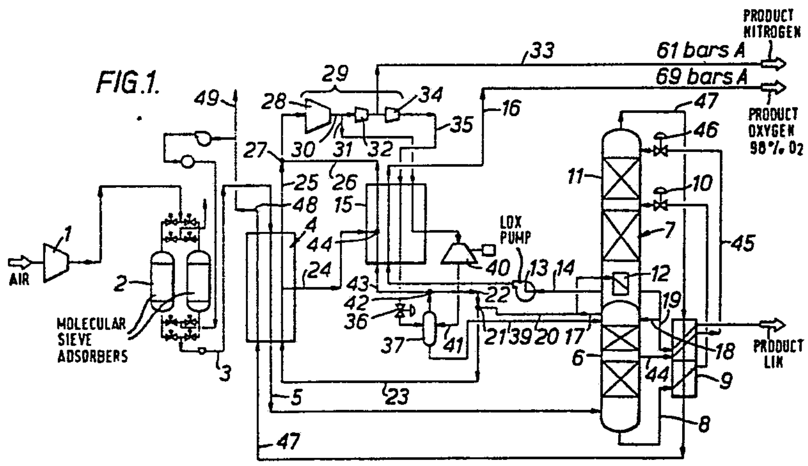


FIG.1.
AIR
MOLECULAR SIEVE ADSORBERS
LOX PUMP
PRODUCT NITROGEN
61 bars A
69 bars A
PRODUCT OXYGEN 98% O2
PRODUCT LIN

This invention relates to a method for producing gaseous oxygen and to a cryogenic plant in which said method can be carried out.

In our UK Patent Application No. 7936637 filed 23 October 1979, we have described a method of producing gaseous oxygen which method comprises the steps of fractionating dry compressed air in a double distillation column having a high pressure column operating at between 5 and 9 bars absolute and a low pressure column operating at between 1.3 and 3 bars absolute, wherein liquid oxygen is withdrawn from the bottom of the low pressure column, is pressurized and is evaporated against a stream of substantially pure nitrogen which has been removed from the top of the high pressure column and compressed to such a pressure that it is at least partially liquified by the evaporation of the oxygen, and wherein at least part of the liquified nitrogen is expanded and returned to the high pressure column as reflux.

The preferred embodiment described in the said UK Patent Application is specifically designed for producing gaseous oxygen at 2.8 bars absolute although it is indicated that the general method is applicable for much higher pressures.

We have found that if the gaseous oxygen is required at pressures in excess of 35 bars, the amount of power consumed by the preferred embodiment described in the said UK Patent Application increases rapidly over a conventional air separation unit provided with a gaseous oxygen compressor and in such cases the value of the additional power outweighs the saving in capital cost.

In order to reduce the amount of power consumed by our cryogenic plant we have found it advantageous to compress the nitrogen rich gas from the high pressure column and divide it into a first sub-stream and a second sub-stream. The first sub-stream is cooled against the vaporising liquid oxygen and is then expanded to produce a mainly (on a molar basis) liquid product at least part of which is returned to the high pressure column as reflux. The second sub-stream is cooled, expanded in an expander, and at least part of the product leaving the expander is used to assist cooling the first sub-stream and is then recycled to the compressor. The nitrogen rich gas is preferably taken from the top of the high pressure column where it is substantially pure. This enables a substantially pure stream of nitrogen to be drawn off the plant at an elevated pressure if desired.

Whilst this method will work at all pressures it is particularly advantageous at pressures of 35 bars A and above.

The first sub-stream and the second sub-stream may be compressed to the same pressure although normally the first sub-stream will be compressed to a pressure 7 to 40 bars above the second sub-stream.

We have found it particularly desirable to arrange for fluid leaving the expander to contain up to 15 mole percent liquid and preferably between 8 and 10 mole percent liquid. In such a case the liquid is preferably separated from the gas and the liquid returned to the high pressure column as additional reflux.

Typically, the pressure of the first sub-stream will be between 40 and 80 bars absolute whilst the pressure of the second sub-stream will be between 30 and 50 bars absolute.

The present invention also provides a cryogenic plant for producing gaseous oxygen, which plant comprises a high pressure column and a low pressure column for distilling air, a heat exchanger in which liquid oxygen can evaporate, a pump for, in use, receiving liquid oxygen from the bottom of said low pressure column and introducing it into said heat exchanger, a compressor, a conduit for carrying nitrogen rich gas from said high pressure column to said compressor, a conduit for conveying a first sub-stream of compressed nitrogen rich gas from said compressor through said heat exchanger in countercurrent flow to said liquid oxygen, an expansion valve for, in use, expanding cold nitrogen rich gas leaving said heat exchanger and a conduit for conveying liquified nitrogen rich gas from said expansion valve to said high pressure column, characterized in that a further conduit is provided for conveying a second sub-stream of compressed nitrogen rich gas from said compressor to said heat exchanger to cool said compressed nitrogen rich gas, an expander for allowing compressed nitrogen rich gas from said heat exchanger to expand, a conduit for conveying nitrogen rich gas from said expander to said heat exchanger to assist in cooling said first sub-stream of compressed nitrogen rich gas and a conduit for carrying said nitrogen rich gas from said heat exchanger to said compressor.

There is also provided a cryogenic plant for producing gaseous oxygen, which plant comprises a high pressure column and a low pressure column for distilling air, a heat exchanger in which liquid oxygen can evaporate, a pump for, in use, receiving liquid oxygen from the bottom of said low pressure column and introducing it into said heat exchanger, a compressor, a conduit for carrying substantially pure gaseous nitrogen from the top of said high pressure column to said compressor, a conduit for conveying a first sub-stream of substantially pure compressed nitrogen from said compressor through said heat exchanger in countercurrent flow to said liquid oxygen, an expansion valve for, in use, expanding substantially pure cold nitrogen leaving said heat exchanger and a conduit for conveying substantially pure liquid nitrogen from said expansion valve to said high pressure column, characterized in that a further conduit is provided for conveying a second sub-stream of substantially pure compressed nitrogen from said compressor to said heat exchanger to cool said compressed nitrogen, an expander for allowing substantially pure compressed nitrogen from said heat exchanger to expand, a conduit for conveying substantially pure gaseous nitrogen from said expander to said heat exchanger to assist in cooling said first sub-stream of substantually pure compressed nitrogen and a conduit for carrying said substantially pure gaseous nitrogen from said heat exchanger to said compressor.

The present invention further provides a cryogenic plant for producing gaseous oxygen which plant comprises a high pressure column and a low pressure column for distilling air, a first heat exchanger in which liquid oxygen can evaporate, a pump for, in use, receiving liquid oxygen from the bottom of said low pressure column and introducing it into said first heat exchanger, a compressor, a conduit for carrying nitrogen rich gas from said high pressure column to said compressor, a conduit for conveying a first sub-stream of compressed nitrogen rich gas from said compressor through said first heat exchanger in countercurrent flow to said liquid oxygen, an expansion valve for, in use, expanding cold nitrogen rich gas leaving said heat exchanger and a conduit for conveying liquified nitrogen rich gas from said expansion valve to said high pressure column, characterized in that a further conduit is provided for conveying a second sub-stream of compressed nitrogen rich gas from said compressor to a second heat exchanger to cool said compressed nitrogen rich gas, an expander for allowing compressed nitrogen rich gas from said second heat exchanger to expand, a conduit for conveying nitrogen rich gas from said expander to said first heat exchanger to assist in cooling said first sub-stream of compressed nitrogen rich gas, and a conduit for carrying said nitrogen rich gas from said first heat exchanger to said compressor.

There is also provided a cryogenic plant for producing gaseous oxygen which plant comprises a high pressure column and a low pressure column for distilling air, a first heat exchanger in which liquid oxygen can evaporate, a pump for, in use, receiving liquid oxygen from the bottom of said low pressure column and introducing it into said first heat exchanger, a compressor, a conduit for carrying substantially pure gaseous nitrogen from the top of said high pressure column to said compressor, a conduit for conveying a first sub-stream of substantially pure compressed nitrogen from said compressor through said first heat exchanger in counter-current flow to said liquid oxygen, an expansion valve for, in use, expanding substantially pure cold nitrogen leaving said heat exchanger and a conduit for conveying substantially pure liquid nitrogen from said expansion valve to said high pressure column, characterized in that further conduit is provided for conveying a second sub-stream of substantially pure compressed nitrogen from said compressor to a second heat exchanger to cool said substantially pure compressed nitrogen, an expander for allowing substantially pure compressed nitrogen from said second heat exchanger to expand, a conduit for conveying substantially pure gaseous nitrogen from said expander to said first heat exchanger to assist in cooling said first sub-stream of substantially pure compressed nitrogen, and a conduit for carrying said substantially pure gaseous nitrogen from said first heat exchanger to said compressor.

Preferably, the first heat exchanger is a wound coil heat exchanger and the second heat exchanger is a plate-fin heat exchanger.

For a better understanding of the invention reference will now be made, by way of example, to the accompanying drawings, in which:

Figure 1 is a simplified flowsheet of one embodiment of a cryogenic plant in accordance with the invention; and

Figure 2 is a second embodiment of such a cryogenic plant.

Referring to Figure 1, air at a flowrate of 5934 Kg moles/hour is compressed to 6.5 bars absolute (bars A) in compressor 1 and is subsequently passed through molecular sieve adsorbers 2 where water, carbon dioxide and any heavy hydrocarbons present are adsorbed. The air leaves the molecular sieve adsorbers 2 through conduit 3 at 6.1 bars A and 10°C. It is then cooled to -172°C in heat exchanger 4 which it leaves through conduit 5 at a condition close to saturation. The stream in conduit 5 is then introduced into the high pressure column 6 of a double column 7.

A crude liquid oxygen stream is withdrawn from the bottom of the high pressure column 6 through conduit 8 and after being subcooled to -177°C in subcooler 9 is expanded to 1.5 bars A at valve 10 and introduced into low pressure column 11 which is provided with a reboiler 12. Liquid oxygen at 98 molar % purity accumulates in the bottom of the low pressure column 11 and passes to the inlet of pump 13 through conduit 14. The liquid is then pumped at a rate of 1162 Kg moles/hour to heat exchanger 15 where it vaporizes and leaves through conduit 16 at 69 bars A and 20°C to form the gaseous oxygen product stream.

Gaseous nitrogen is withdrawn from the top of the high pressure column 6 through conduit 17. A portion of this nitrogen is condensed in reboiler 12 and returned to the high pressure column 6 through conduit 18 and the remainder through conduit 19 and sub-cooler 9 to product at the rate of

17 kg. moles/hour. The remaining nitrogen in conduit 17 passes through conduit 20. It is joined at junction 21 by nitrogen from conduit 22 and the combined stream is passed through conduit 23 to heat exchanger 4. After being warmed to -125°C, a portion of the nitrogen is withdrawn through conduit 24 and the balance leaves the heat exchanger 4 through conduit 25 at 6°C. It is joined by nitrogen from conduit 26 at junction 27 and the combined stream is introduced into the first stage 28 of a compressor 29. The nitrogen is compressed to 41 bars A and leaves the first stage 28 through conduit 30. A second sub-stream 31 is then withdrawn and the balance is compressed to 61 bars A in second stage 32 and product nitrogen at a flowrate of 1367 Kg moles/hour is withdrawn through conduit 33. The remaining nitrogen is compressed to 80 bars A in the third stage 34 of compressor 29 which is leaves through conduit 35 as a first sub-stream.

The first sub-stream is cooled to -169°C in heat exchanger 15 which it leaves as a sub-cooled vapour as it is above its critical pressure. It is then expanded to 5.9 bars A at valve 36 and the two phases thus formed are separated in phase separator 37.

Liquid nitrogen is withdrawn from the bottom of the phase separator 37 and is returned to the high pressure column 6 through conduit 39 as reflux.

Turning now to the second sub-stream 31, the nitrogen at 41 bars A is cooled to -123°C in heat exchanger 15 and is then expanded through generator loaded expander 40 to 5.9 bars A. The two phase nitrogen leaving the expander 40 through conduit 41 contains 8 mole percent liquid and is introduced into phase separator 37. The gaseous nitrogen in the phase separator 37 passes to junction 42. Gaseous nitrogen is passed through conduit 22 to junction 21 whilst the balance is introduced into the cold end of heat exchanger 15 through conduit 43. After being warmed to -125°C the nitrogen is joined by nitrogen from conduit 24 at junction 44. The combined stream is then warmed and enters conduit 26.

Of the remaining features a nitrogen-rich liquid fraction is taken from the high pressure column 6 through conduit 44 to sub-cooler 9 which it leaves through conduit 45. It is then expanded at valve 46 and the resulting two phase mixture is introduced into the low pressure column 11. Waste nitrogen leaves the top of the low pressure column 11 through conduit 47 and after being warmed in sub-cooler 9, enters heat exchanger 4 via conduit 47. The waste nitrogen leaves heat exchanger 4 through conduit 48 and a portion is used for regenerating the molecular sieves 2. The remainder is vented to atmosphere through conduit 49.

Various modifications to the cryogenic plant described are currently envisaged, for example, whereas heat exchangers 4 and 15 will normally be plate-fin heat exchangers where pressure permit, it may be necessary for heat exchanger 15 to be a wound coil heat exchanger (generally where the pressure of the oxygen stream exceeds 81 bars A). However, since wound coil heat exchangers are relatively expensive, it may be desirable to use a plate-fin heat exchanger in combination with a wound coil heat exchanger in place of a single wound coil heat exchanger 15.

In this connection reference is made to Figure 2 which shows a cryogenic plant which is generally similar to that shown in Figure 1 except that the liquid oxygen is pumped to 98 bars A by pump 13. In this particular embodiment the second and third stages of the compressor have been combined into a single second stage 51 which compresses the nitrogen to 74 bars A. For this embodiment the heat exchanger 15 is of the wound coil type. Because of the cost and complexity of building a wound coil heat exchanger with the flows arranged as shown in Figure 1, the second sub-stream 31 is cooled in a separate plate-fin heat exchanger 52 before being expanded as before. Cooling of the second sub-stream is effected by a stream 53 taken from conduit 24, passed through plate-fin heat exchanger 52 and returned to conduit 26 as shown.

If desired, in either of the cryogenic plants described, heat exchanger 4 may be replaced with a reversing heat exchanger the and molecular sieve adsorbers omitted. In such an embodiment it will be necessary to place a hydrocarbon absorber in conduit 8 downstream of subcooler 9.

If desired the second stage 32 and/or third stages 34 of the compressor 29 could be separate and distinct from the first stage 28 and driven, for example completely, or in part, by expander 40.

CLAIMS

1. A method for producing gaseous oxygen which method comprises the steps of fractionating dry compressed air in a double distillation column having a high pressure column operating at between 5 and 9 bars absolute and a low pressure column operating at between 1.3 and 3 bars absolute, wherein liquid oxygen is withdrawn from the bottom of the low pressure column, is pressurized and is evaporated against a stream of nitrogen rich gas which has been removed from the high pressure column and compressed in a compressor characterized in that the compressed nitrogen rich gas is divided into a first sub-stream and a second sub-stream; the first sub-stream is cooled against the vaporising liquid oxygen and is then expanded to produce a mainly (on a molar basis) liquid product at least part of which is returned to the high pressure column as reflux; and the second sub-stream is cooled, expanded in an expander, and at least part of the product leaving the expander is used to assist cooling the first sub-stream and is then recycled to said compressor.

2. A method for producing gaseous oxygen which method comprises the steps of fractionating dry compressed air in a double distillation column having a high pressure column operating at between 5 and 9 bars absolute and a low pressure column operating at between 1.3 and 3 bars absolute, wherein liquid oxygen is withdrawn from the bottom of the low pressure column, is pressurized and is evaporated against a stream of substantially pure nitrogen which has been removed from the top of the high pressure column and compressed in a compressor characterized in that the compressed nitrogen is divided into a first sub-stream and a second sub-stream; the first sub-stream is cooled against the vaporising liquid oxygen and is then expanded to produce a mainly (on a molar basis) liquid product at least part of which is returned to the high pressure column as reflux; and the second sub-stream

is cooled, expanded in an expander, and at least part of the product leaving the expander is used to assist cooling the first sub-stream and is then recycled to said compressor.

3. A method according to Claim 1 or 2, characterized in that the first sub-stream is compressed to a higher pressure than the second sub-stream.

4. A method according to Claim 1 or 2, characterized in that said second sub-stream leaving said expander contains up to 15 mole percent liquid.

5. A method according to Claim 1, 2, 3 or 4, characterized in that the pressure of said first sub-stream is in excess of 40 bars absolute.

6. A method according to Claim 1, 2, 3, 4, or 5, characterized in that the pressure of said second sub-stream is in excess of 30 bars absolute.

7. A cryogenic plant for producing gaseous oxygen, which plant comprises a high pressure column and a low pressure column for distilling air, a heat exchanger in which liquid oxygen can evaporate, a pump for, in use, receiving liquid oxygen from the bottom of said low pressure column and introducing it into said heat exchanger, a compressor, a conduit for carrying nitrogen rich gas from said high pressure column to said compressor, a conduit for conveying a first sub-stream of compressed nitrogen rich gas from said compressor through said heat exchanger in countercurrent flow to said liquid oxygen, an expansion valve for, in use, expanding cold nitrogen rich gas leaving said heat exchanger and a conduit for conveying liquified nitrogen rich gas from said expansion valve to said high pressure column, characterized in that a further conduit is provided for conveying a second sub-stream of compressed nitrogen rich gas from said compressor to said heat exchanger to cool said compressed nitrogen rich gas, an expander for allowing compressed nitrogen rich gas from said heat exchanger to expand, a conduit for conveying nitrogen rich gas from said expander to

said heat exchanger to assist in cooling said first sub-stream of compressed nitrogen rich gas and a conduit for carrying said nitrogen rich gas from said heat exchanger to said compressor.

8. A cryogenic plant for producing gaseous oxygen, which plant comprises a high pressure column and a low pressure column for distilling air, a heat exchanger in which liquid oxygen can evaporate, a pump for, in use, receiving liquid oxygen from the bottom of said low pressure column and introducing it into said heat exchanger, a compressor, a conduit for carrying substantially pure gaseous nitrogen from the top of said high pressure column to said compressor, a conduit for conveying a first sub-stream of substantially pure compressed nitrogen from said compressor through said heat exchanger in countercurrent flow to said liquid oxygen, an expansion valve for, in use, expanding substantially pure cold nitrogen leaving said heat exchanger and a conduit for conveying substantially pure liquid nitrogen from said expansion valve to said high pressure column, characterized in that a further conduit is provided for conveying a second sub-stream of substantially pure compressed nitrogen from said compressor to said heat exchanger to cool said compressed nitrogen, an expander for allowing substantially pure compressed nitrogen from said heat exchanger to expand, a conduit for conveying substantially pure gaseous nitrogen from said expander to said heat exchanger to assist in cooling said first sub-stream of substantually pure compressed nitrogen and a conduit for carrying said substantially pure gaseous nitrogen from said heat exchanger to said compressor.

9. A cryogenic plant for producing gaseous oxygen which plant comprises a high pressure column and a low pressure column for distilling air, a first heat exchanger in which liquid oxygen can evaporate, a pump for, in use, receiving liquid oxygen from the bottom of said low pressure column and introducing it into said first heat exchanger, a compressor,

a conduit for carrying nitrogen rich gas from said high pressure column to said compressor, a conduit for conveying a first sub-stream of compressed nitrogen rich gas from said compressor through said first heat exchanger in counter-current flow to said liquid oxygen, an expansion valve for, in use, expanding cold nitrogen rich gas leaving said heat exchanger and a conduit for conveying liquified nitrogen rich gas from said expansion valve to said high pressure column, characterized in that a further conduit is provided for conveying a second sub-stream of compressed nitrogen rich gas from said compressor to a second heat exchanger to cool said compressed nitrogen rich gas, an expander for allowing compressed nitrogen rich gas from said second heat exchanger to expand, a conduit for conveying nitrogen rich gas from said expander to said first heat exchanger to assist in cooling said first sub-stream of compressed nitrogen rich gas, and a conduit for carrying said nitrogen rich gas from said first heat exchanger to said compressor.

10. A cryogenic plant for producing gaseous oxygen which plant comprises a high pressure column and a low pressure column for distilling air, a first heat exchanger in which liquid oxygen can evaporate, a pump for, in use, receiving liquid oxygen from the bottom of said low pressure column and introducing it into said first heat exchanger, a compressor, a conduit for carrying substantially pure gaseous nitrogen from the top of said high pressure column to said compressor, a conduit for conveying a first sub-stream of substantially pure compressed nitrogen from said compressor through said first heat exchanger in countercurrent flow to said liquid oxygen, an expansion valve for, in use, expanding substantially pure cold nitrogen leaving said heat exchanger and a conduit for conveying substantially pure liquid nitrogen from said expansion valve to said high pressure column, characterized in that further conduit is provided for conveying a second sub-stream of substantially pure compressed nitrogen

from said compressor to a second heat exchanger to cool said substantially pure compressed nitrogen, an expander for allowing substantially pure compressed nitrogen from said second heat exchanger to expand, a conduit for conveying substantially pure gaseous nitrogen from said expander to said first heat exchanger to assist in cooling said first sub-stream of substantially pure compressed nitrogen, and a conduit for carrying said substantially pure gaseous nitrogen from said first heat exchanger to said compressor.

11. A cryogenic plant according to Claim 9 or 10, characterized in that said first heat exchanger is a wound coil heat exchanger and said second heat exchanger is a plate-fin heat exchanger.

For the applicants,

Brian Lucas

Brian Lucas
CHARTERED PATENT AGENT
EUROPEAN PATENT ATTORNEY

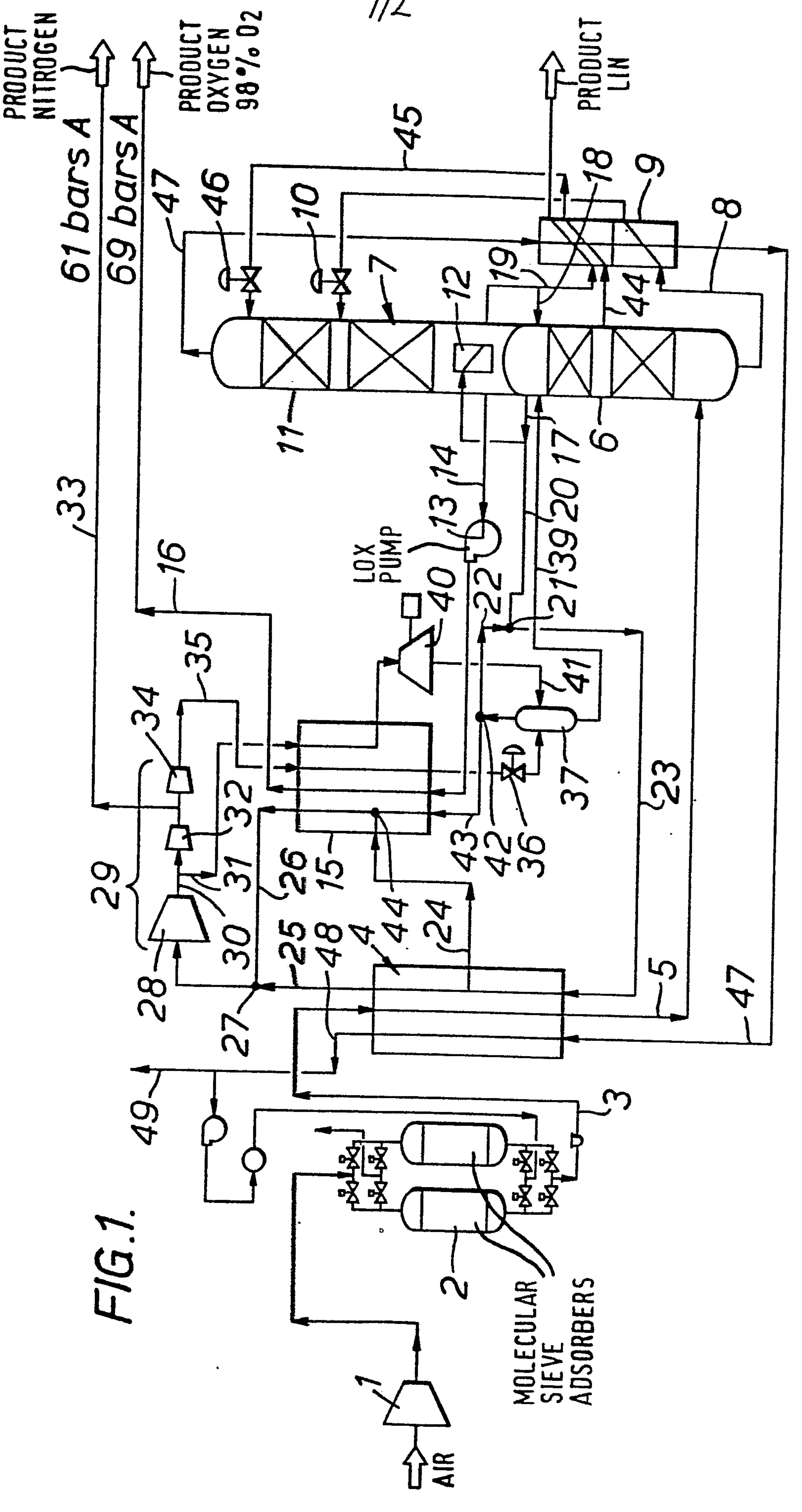
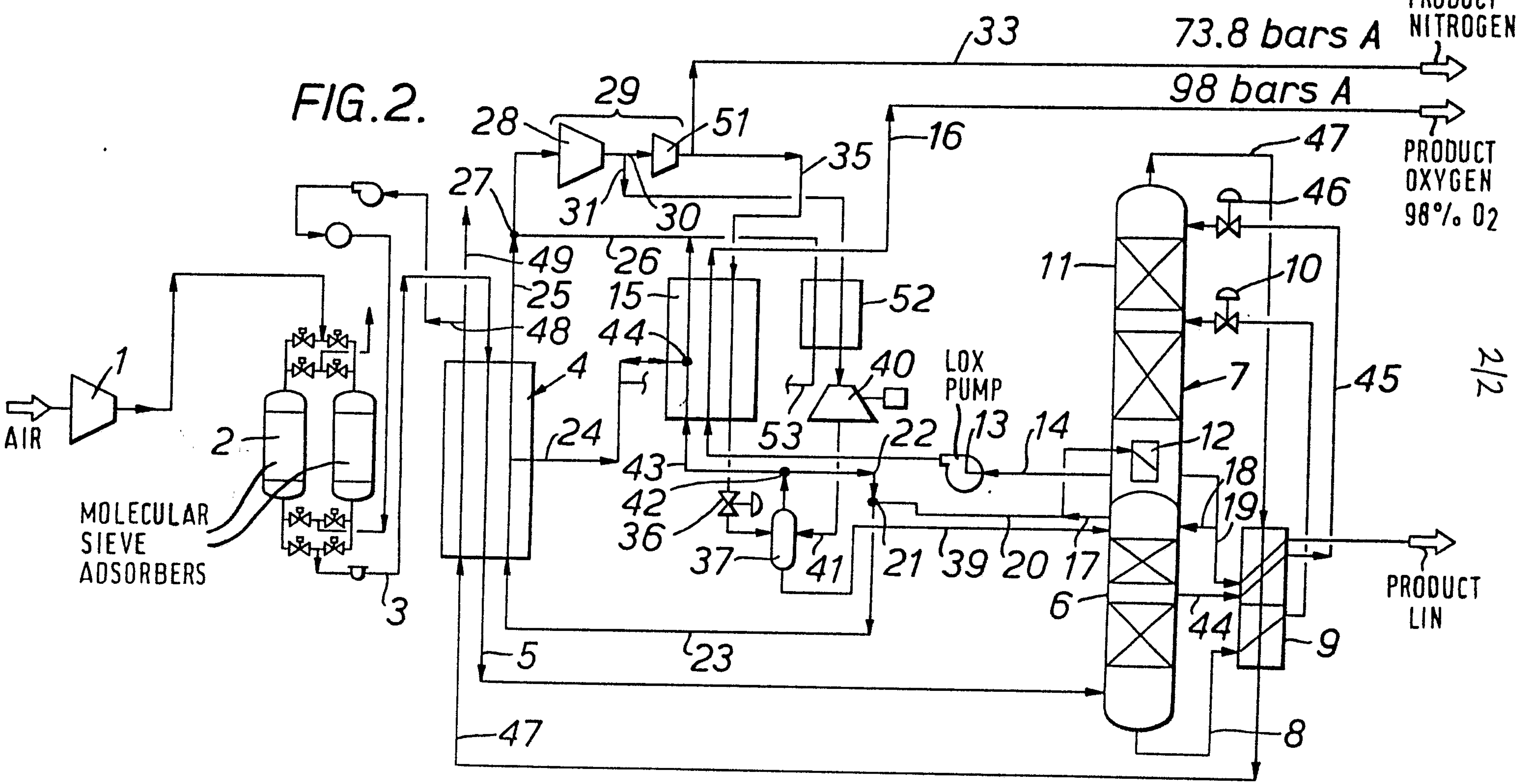

PRODUCT NITROGEN
PRODUCT OXYGEN 98% O2
PRODUCT LIN
61 bars A
69 bars A
LOX PUMP
MOLECULAR SIEVE ADSORBERS
AIR

FIG.1.

FIG.2.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98 bars A
PRODUCT OXYGEN 98% O2
PRODUCT LIN
LOX PUMP
AIR
MOLECULAR SIEVE ADSORBERS
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Application number

EP 81 30 2417

European Patent Office

EUROPEAN SEARCH REPORT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.³)
P,E	EP - A - 0 024 962 (L'AIR LIQUIDE) * abstract; page 1, lines 1-3; page 2, lines 2-16; page 6, lines 1-26; figure 2 * --	1,2,7, 8	F 25 J 3/04
	US - A - 3 605 422 (J.A.PRYOR et al.) * abstract; column 1, lines 26-37; lines 61-72; column 2, line 14 - column 6, line 14; figure * --	1,2,7, 8	
			TECHNICAL FIELDS SEARCHED (Int. Cl.³)
A	US - A - 3 083 544 (F.JAKOB) * column 1, lines 11-16; column 1; line 34 - column 2, line 10; column 2, line 47 - column 3, line 36; figure 1 * --	1,7	F 25 J 3/04
D,E	EP - A - 0 029 656 (AIR PRODUCTS AND CHEMICALS) ------------		

CATEGORY OF CITED DOCUMENTS

X: particularly relevant
A: technological background
O: non-written disclosure
P: intermediate document
T: theory or principle underlying the invention
E: conflicting application
D: document cited in the application
L: citation for other reasons

&: member of the same patent family, corresponding document

X The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
The Hague	24-09-1981	SIEM

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