FORM 1

622669

REGULATION 9

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952

APPLICATION FOR A STANDARD PATENT

We, THE BOC GROUP plc, an English Company, of Chertsey Road, Windlesham, Surrey GU20 6HJ, England, hereby apply for the grant of a Standard Patent for an invention entitled:-

"AIR SEPARATION"

which is described in the accompanying Complete Specification.

Our address for service is:

SHELSTON WATERS
55 Clarence Street
SYDNEY, N.S.W. 2000.

DATED this 11th day of MAY, 1989
THE BOC GROUP plc

by Leon K. Allen.
Fellow Institute of Patent Attorneys of Australia
of SHELSTON WATERS

To: The Commissioner of Patents
WODEN A.C.T. 2606

File: D.B.B128

Fee: $265.00

S007392 11/05/89

(NON-CONVENTION—Company)

88B102/MW

FORM 7—REGULATION 12 (1)

COMMONWEALTH OF AUSTRALIA

PATENTS ACT, 1952-1973

DECLARATION IN SUPPORT OF AN APPLICATION FOR A PATENT.

(a) Here Insert (in full) Name of Company.

In support of the Application made by [a] THE BOC GROUP Plc

(hereinafter referred to as "Applicant") for a patent for an invention entitled:

(b) Here Insert Title of invention.

[b] "AIR SEPARATION"

(c) Here Insert Full Name and Address of Company Official authorised to make declaration.

I, [c] MICHAEL WICKHAM,

of CHERTSEY ROAD, WINDLESHAM,

SURREY GU20 6HJ, UNITED KINGDOM.

do solemnly and sincerely declare as follows:

1. I am authorised by Applicant to make this declaration on its behalf.

(d) Here Insert (in full) Name and Address of Actual Inventor(s).

2. [d] THOMAS RATHBONE
12 BROOMLEAF ROAD,
FARNHAM,
SURREY GU9 8DG UNITED KINGDOM is/are the actual inventor(s) of the invention and the facts upon which Applicant is entitled to make the Application are as follows:

~~Applicant is the Assignee of the said Inventor(s)~~.

THE BOC GROUP Plc ARE THE ASSIGNEES OF THE BOC LIMITED (CRYOPLANTS) WHOSE RIGHT TO THE INVENTION IS BY VIRTUE OF SECTION 39(1) OF THE PATENTS ACT 1977 IN THE UNITED KINGDOM.

Declared at Windlesham, Surrey, UK

this 3rd day of April 19 89.

(e) Personal Signature of Declarant (c) (no seal, witness or legalisation).

For The BOC Group plc

[e] M C Wickham

Michael Wickham
By Power of Attorney

To THE COMMISSIONER OF PATENTS.

SHELSTON WATERS
PATENT ATTORNEYS
55 CLARENCE STREET, SYDNEY
AUSTRALIA
Cables: 'Valid' Sydney Telex: 24422

(12) PATENT ABRIDGMENT (11) Document No. AU-B-34696/89
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 622669

(54) Title
AIR SEPARATION

International Patent Classification(s)
(51)[4] **F25J 003/04**

(21) Application No. : **34696/89** (22) Application Date : **11.05.89**

(43) Publication Date : **29.11.90**

(44) Publication Date of Accepted Application : **16.04.92**

(71) Applicant(s)
THE BOC GROUP PLC

(72) Inventor(s)
THOMAS RATHBONE

(74) Attorney or Agent
SHELSTON WATERS , 55 Clarence Street, SYDNEY NSW 2000

(56) Prior Art Documents
EP 328239
EP 259070

(57) Claim

1. A method of separating air, comprising the steps of:

a) passing a stream of air into a first distillation column;

b) withdrawing an oxygen-rich liquid from a bottom region of the first distillation column and passing it to a top region of a mixing zone;

c) passing nitrogen-rich vapour from the first distillation column to a bottom region of the mixing zone;

d) establishing through the mixing zone a downward flow of liquid that becomes progressively richer in nitrogen in the direction of liquid flow and an upward flow of vapour that becomes progressively richer in oxygen in the direction of vapour flow;

.../2

e) passing liquid nitrogen from the mixing zone to the first distillation column to act as reflux;

f) withdrawing an oxygen-depleted mixed stream comprising oxygen and nitrogen from a chosen level of the mixing zone;

g) employing the mixed stream to support combustion of a fluid fuel and expanding the resulting combustion products in a turbine;

h) withdrawing from the first distillation column a stream of argon-containing fluid whose argon concentration is greater than that of the air stream, and separating an argon product from the argon-containing stream in a second distillation column; and

i) withdrawing an oxygen product from one or both of the distillation columns.

COMMONWEALTH OF AUSTRALIA 622669

FORM 10

PATENTS ACT 1952

COMPLETE SPECIFICATION

FOR OFFICE USE:

Class Int.Class

Application Number:
Lodged:

Complete Specification Lodged:
Accepted:
Published:

Priority:

Related Art:

Name of Applicant: THE BOC GROUP plc

Address of Applicant: Chertsey Road, Windlesham, Surrey GU20 6HJ, England

Actual Inventor: THOMAS RATHBONE

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

Complete Specification for the Invention entitled:

"AIR SEPARATION"

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

AIR SEPARATION

DESCRIPTION

This invention relates to a method and apparatus for separating argon from air.

Traditionally, in separating air, if argon is to be obtained as a product gas, the incoming air is separated into relatively pure streams of oxygen, nitrogen and argon. European Patent Application 136 926A relates to the operation of a conventional double column with argon "side-draw" for producing nitrogen, oxygen and argon products. It is the object of the invention disclosed in that European Patent Application to take advantage of a temporary fall in the oxygen demand in order to increase the production of one or more of the other products, for example argon. A liquid is thus taken from one of the two columns forming the double column and is passed to the top of an auxiliary column or mixing column operating at substantially the pressure of low pressure column. A gas whose oxygen content is less than that of the liquid is taken from the low pressure column and is passed to the bottom of the auxiliary column. A liquid collected at the bottom of the auxiliary column is passed as reflux into

the low pressure column at substantially the level from which the said gas is taken. As more oxygen-rich liquid is taken from the double column and passed to the auxiliary column so more reflux may be provided for the low pressure column, thereby making possible an increase in the rate of argon production. However, this method involves substantial inefficiencies which makes it unsuitable for use in a plant for producing argon as the primary or sole product of air separation. In particular we have discovered that the mixing of liquid oxygen and nitrogen vapour is preferably performed at a pressure of at least 3 atmospheres absolute, whereas the low pressure column is normally operated at a pressure of about 1 1/2 atmosphere absolute.

Our UK patent application 2 174 916 A relates to a method of separating argon from air in which improvements in the operation of the auxiliary or mixing zone are made possible resulting in an improvement in the argon yield and in the efficiency at which it is produced. However, this gain in yield and efficiency is gained at the expense of oxygen and nitrogen production. If oxygen or nitrogen is also taken as a product, there is a decline in the yield and efficiency with which the argon is produced. Moreover, it is necessary to withdraw a pressured mixed waste stream from the mixing zone, so for maximum

efficiency another process is needed to be operated to utilise the pressure of the waste stream.

European patent application 81 178 A relates to a process in which a single distillation column is used to separate air into nitrogen and oxygen products. In addition, a nitrogen waste stream containing some oxygen is produced. The nitrogen waste stream is used to support combustion of a hydrocarbon fuel to power a turbine driving the compressor that raises the incoming air to a pressure suitable for its introduction into a distillation column. The advantage of this arrangement is that the separation is performed with a relatively low specific power consumption. However, this process has drawbacks, chief among which are that it produces no argon and that the single column operates relatively inefficiently.

It is an aim of the present invention to provide a method and apparatus for separating air which as well as making possible the production of argon in relatively high yield also makes possible the production of oxygen.

According to the present invention there is provided a method of separating air, comprising the steps of:

(a) passing a stream of air into a first distillation column;

(b) withdrawing an oxygen-rich liquid from a bottom region of the first distillation column and passing it to a top region of a mixing zone;

(c) passing nitrogen-rich vapour from the first distillation column to a bottom region of the mixing zone;

(d) establishing through the mixing zone a downward flow of liquid that becomes progressively richer in nitrogen in the direction of liquid flow and an upward flow of vapour that becomes progressively richer in oxygen in the direction of vapour flow;

(e) passing liquid nitrogen from the mixing zone to the first distillation column to act as reflux therein;

(f) withdrawing an oxygen-depleted mixed stream comprising oxygen and nitrogen from a chosen level of the mixing zone;

(g) employing the mixed stream to support combustion of a fluid fuel and expanding the resulting combustion products in a turbine;

(h) withdrawing from the first distillation column a stream of argon-containing fluid whose argon concentration is greater than that of the air stream, and separating an argon product from the argon-containing stream in a second distillation column; and

(i) withdrawing an oxygen product from one or both of the distillation columns; (preferably from the first distillation column).

The invention also provides apparatus for separating air, comprising:

(a) means for passing a stream of air into a first distillation column;

(b) means for withdrawing an oxygen-rich liquid from a bottom region of the first distillation column and passing it to a top region of a mixing zone;

(c) means for passing nitrogen rich vapour from the first distillation column to a bottom region of the mixing zone;

(d) liquid-vapour contact means for establishing through the mixing zone a downward flow of liquid that becomes progressively richer in nitrogen in the direction of liquid flow and an upward flow of vapour that becomes progressively richer in oxygen in the direction of vapour flow;

(e) means for passing liquid nitrogen from the mixing zone to the first distillation column to act as reflux;

(f) means for withdrawing an oxygen-depleted mixed stream comprising oxygen and nitrogen from a chosen level of the mixing zone;

(g) means for burning a fluid fuel using the mixed stream to support the combustion

h) means for withdrawing from the first distillation column a stream of argon-containing fluid whose argon concentration is greater than that of the air

stream said means communicating with a second distillation column for separating an argon product from the argon containing stream; and

(i) means for withdrawing an oxygen product from one or both of the distillation columns

The turbine is preferably employed to drive at least one compressor that compresses the air upstream of the first distillation column. The fuel is preferably a gaseous hydrocarbon such as methane. Preferably the fuel is preheated by heat exchange with the combustion products downstream of their exit from the turbine.

By integrating such a power generation system with the air separation greater efficiency of conversion of fuel into power is possible than in a conventional power station.

Preferably, the mixed stream contains not less than 15% by volume of oxygen.

Preferably, condensation is provided for oxygen-rich vapour at the top of the mixing zone. Such condensation helps to improve the efficiency at which the mixing zone operates. A further improvement in this efficiency may be achieved by withdrawing a vapour stream from a level of

the mixing zone above that of the level from which said mixed stream is withdrawn, but below the top of the mixing zone, condensing the vapour stream in heat exchange with a stream of boiling liquid from one of the distillation columns, returning a stream of thus-formed condensate to the mixing zone, and returning boiled liquid to its respective distillation column.

Efficient operation of the mixing zone and first distillation column is enhanced by choosing an operating pressure for them of above 3 atmospheres absolute. Typically, the first distillation column and the mixing zone are operated at pressures in the order of 5 atmospheres. It is, however, usually desirable to operate the second distillation column at a pressure in the range of 1 to 2 atmospheres absolute. Accordingly, it is preferred that the second distillation column operates at a lower pressure than the first distillation column and that the said argon-containing stream be withdrawn from the first column as liquid, be sub-cooled, and be passed into the second distillation column through a throttling valve. This arrangement makes possible efficient operation of the argon column at any pressure selected within a relatively wide range of operating pressures. With such an arrangement, it becomes convenient to take a stream of nitrogen from the mixing zone and employ it to

reboil the liquid in or from a bottom region of the second distillation column, thereby condensing the nitrogen. Resulting condensate is then preferably introduced into the top region of the first distillation column as reflux.

In order to reboil liquid at or from the bottom of the first distillation column and to condense oxygen at or from the top of the mixing zone, nitrogen may be employed as the working fluid. In addition, it is generally desirable to take the argon product in the liquid phase and accordingly it is preferred to condense argon at or from the top of the second distillation column. One portion of the condensed argon is used as reflux of the second column and a second portion is taken as product. Typically, a working fluid comprising nitrogen is employed to condense the argon.

In order to enhance the efficiency at which the argon column operates a stream of vapour is preferably taken from a level of the second distillation column intermediate that at which the argon-containing stream is introduced into such column on the top of the second column, the stream of vapour is then condensed and returned to the second column. Again, nitrogen is preferably employed to condense such stream.

Accordingly, in preferred embodiments of the invention, nitrogen is typically required at five different pressures to perform heat pumping duties for the apparatus according to the present invention and the apparatus according to the invention preferably includes a nitrogen distribution and refrigeration system to meet this need.
The nitrogen is desirably taken from the top of the first distillation column where the gaseous phase typically contains from 0.5 to 1% by volume of oxygen (and a balance of nitrogen).

In another embodiment of the invention, the argon-rich vapour condenser associated with the second distillation column is amalgamated with the reboiler for the first distillation column in a condenser-reboiler.

As in conventional air separation processes, it is desirable to remove low volatility impurities from the air stream prior to its introduction into the first distillation column. Such low volatility impurities may for example be removed from the air stream in a reversing heat exchanger or heat exchangers, or by adsorbers.

The argon product, which is preferably produced in the liquid phase, may if desired be subjected to further purification as it typically contains up to 2% by volume of oxygen.

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

Figure 1 is a simplified circuit diagram showing an arrangement of liquid-vapour contact columns for use in generating argon in accordance with the invention and,

Figure 2 is a circuit diagram of an argon generator employing the arrangement of columns shown in Figure 1. Referring to Figure 1, an air stream from which low volatility constituents and impurities such as carbon dioxide and water vapour have been removed is introduced into a single distillation column 10 through an inlet 2 at a pressure of typically at 5 atmospheres absolute and at a temperature typically at its dew point. A distillation column 10 is provided with a suitable number of liquid-vapour contact trays (not shown) to enable the incoming air to be separated into an oxygen-enriched liquid which collects at the bottom of the column 10 and a nitrogen-enriched vapour which collects at the top of the column 10. Liquid nitrogen reflux for the column 10 is provided through inlet 16 at the top of the column and reboil for the column is provided by a reboiler 14 in the bottom region thereof. The properties of the fluid mixture in the column 10 are such that a maximum

concentration of argon is obtained in the liquid and vapour phases at a level below that of the inlet 2, and whereas the incoming air contains in the order of 0.9% by volume of argon, a liquid fraction typically containing in the order of 8% by volume of argon may be withdrawn from the column 10 through the outlet 4.

In order to form the reflux and reboil for the distillation column 10, it is necessary to do heat pumping work. To reduce the amount of work from an external source that it is required, another liquid-vapour contact column 20 is employed to mix liquid oxygen and gaseous nitrogen fractions from the distillation column 10 and thus produce liquid nitrogen which is returned to the column 10 as reflux. A liquid oxygen stream is withdrawn from the bottom of the column 10 through an outlet 6 and is mixed with a gaseous oxygen stream passing out of the column 20 through outlet 32. The resulting mixed stream flows through a condenser 30 and the condensate is introduced into the top of the column 20 through an inlet 22. Preferably the liquid oxygen that enters the column 20 through the inlet 22 is not pure. The use of the condenser 30 in association with the mixing column 20 is described in our UK patent application No. 2 174 916 A. Gaseous nitrogen is taken from the top of the distillation column 10 through the outlet 8 and is passed into an

inlet 24 at the bottom of the mixing column 20. The mixing column 20 operates at substantially the same pressure as the distillation column 10 and is provided with a number of liquid-vapour contact trays (not shown) to enable intimate contact to take place between the liquid and vapour phases. It is desirable that the relationship between the liquid and the vapour on each tray is relatively close to equilibrium, and accordingly, the mixing column typically has a relatively large number of trays, for example 30.

As the liquid descends the column 20 so it becomes progressively richer in nitrogen. Thus a liquid nitrogen stream is able to pass out of the column 20 through an outlet 26 to form part of the liquid nitrogen reflux stream that enters the column 10 through the inlet 16. A mixed stream comprising from 85% by volume of nitrogen and 15% by volume of oxygen is withdrawn from an intermediate location in the column 20 through an outlet 28. The stream withdrawn through the outlet 28 is relatively lean in argon compared with the air entering the distillation column 10 through the inlet 2 since most and preferably substantially all of this argon is subsequently withdrawn again through the outlet 4. The stream withdrawn through the outlet 28 is then warmed to ambient temperature in heat exchanger 29, which is

desirably integrated with heat exchangers that affect cryogenic coding of other streams passing through the plant. The stream is then heated to a temperature of at least 150°C. by heat exchange in heat exchanger 31 with hot gas exiting a turbine 37. After passage through the heat exchanger 31, the mixed stream is introduced into a burner 33 to support combustion of hydrocarbon fuel, e.g. methane, introduced into the burner 33 through an inlet 35. The burner 33 is operated at the pressure of the mixed stream and hence of the columns 20 and 10. Since the combustion products from the burner 33 are employed in the turbine 37 to generate power, it is desirable that the columns 10 and 20 are operated at a pressure of at least 3 atmospheres absolute and preferably at a pressure in the range 5 to 10 atmospheres absolute. The composition of the mixed stream may be selected so as to make possible stable combustion at a not-excessive temperature (eg. about 1000°C) in the burner 33. After leaving the turbine 37 the combustion products are used to preheat the mixed stream in the heat exchanger 31, as aforesaid, and are then vented to the atmosphere. The turbine 37 may be coupled directly to the drive of an air compressor used to compress the incoming air or may be used to generate electricity to drive the compressor.

By employing a mixed stream relatively depleted in oxygen in comparison with the analogues mixed stream in the processes described in our UK patent application 2 174 916 A it becomes possible to withdraw an oxygen product stream from the distillation column 10 without adversely affecting the yield of argon product, or the efficiency with which it is produced. Accordingly, an oxygen product stream is withdrawn from the column 10 through an outlet 39.

Oxygen in the gaseous phase is withdrawn from the top of the mixing column 20 through the outlet 32 and is condensed in a condenser 30, the resulting liquid oxygen being combined with the liquid oxygen being withdrawn from the distillation column 10 through the outlet 6 and then being fed to the mixing column 20 through the inlet 22. Preferably the liquid oxygen that enters the column 20 through the inlet 22 is not pure. The use of the condenser 30 in association with the mixing column 20 is described in our UK patent application No. 2 174 916 A.

In order to maintain the operating conditions in the column 20 close to the equilibrium, a second stream of vapour may be taken from a level of the column 20 intermediate the level of the outlet 28 and the top of the column and be condensed in a condenser 40. The resulting

condensate is returned to the column at a level below that at which the vapour for condensation is taken from the column. The level at which the condensate from the condenser 40 is returned to the column 20 is selected so that the composition of the condensate corresponds approximately to that of the liquid into which it is reintroduced. In order to provide cooling for the condenser 40, a stream of liquid is withdrawn from the column 10 through an outlet 38 at a level below that of the inlet 2. The liquid that is withdrawn from the column 10 through the outlet 38 is reboiled in the condenser 40 and resulting vapour is returned to the distillation column 10 at a level such that its composition corresponds approximately to that of the vapour into which it is reintroduced. This "intermediate" reboiling of the liquid withdrawn from the column 10 through the outlet 38 also helps to improve the efficiency with which the distillation column 10 operates.

The argon enriched liquid oxygen that is withdrawn from the distillation column 10 through the outlet 4 is subjected to further distillation or rectification in the column 50. Whereas in conventional air separation plants the column that is employed to distil argon-enriched oxygen stream is operated at substantially the same pressure as the distillation column from which the stream

is taken, in preferred methods and plants according to the present invention, the column 50 is operated at a lower pressure than the column 10, for example, at a pressure a little above atmospheric. Accordingly, the liquid withdrawn through the outlet 4 is sub-cooled in a heat exchanger 94 and is then passed through a throttling valve 44 and enters the column 50 through an inlet 46 as liquid. The column 50 is provided with liquid-vapour contact trays (not shown) in order to facilitate mass exchange between the liquid and vapour phases. The column 50 is further provided with a reboiler 52 at the bottom region thereof and a condenser 54 associated with the top thereof. A liquid oxygen fraction collects at the bottom of the column 50 and is reboiled by the reboiler 52. Argon-enriched gas collects at the top of the column 50 and is withdrawn therefrom through an outlet 58 leading to the condenser 54 where it is condensed. Some of the resulting condensate is returned to the column 50 through an inlet 60 at its top and the remainder is withdrawn as a crude argon product through outlet 62.

In accordance with an unique feature of preferred methods according to the invention, the reboil for the argon column 50 is provided by taking a portion of the gaseous nitrogen leaving the top of the distillation column 10 through the outlet 8 and passing it through the

reboiler 52, the nitrogen thereby being condensed. The resultant liquid nitrogen is returned to the column 10, being united with the liquid nitrogen that leaves the mixing column 20 through the outlet 26. Accordingly, the reboiler 52 also acts as a condenser providing reflux for the distillation column 10.

In a plant embodying the column system shown in Figure 1, cooling for the condensers 30 and 54 and for the sub-cooler 94 may be provided by nitrogen generated in the distillation column 10. Similarily such nitrogen may be employed as the source of heat for the reboiler 14. One such plant is illustrated in Figure 2 of the accompanying drawings. In the description of Figure 2, the same reference numerals as used in Figure 1 shall be employed to indicate items of plant that are common to both Figures. Moreover, the operation of those parts of the plant that are shown in Figure 1 will not be described again in any detail.

The arrangement of columns employed in the plant shown in Figure 2 is generally similar to that shown in Figure 1. In order to assist the flow of liquid oxygen from the bottom of the distillation column 10 to the top of the mixing column 20, a pump 70 is employed, and a similar pump 72 is used to pump the liquid stream from the

outlet 38 of the distillation column 10 through the condenser-reboiler 40. In addition an additional condenser 74 is employed in association with the argon column 50. Vapour is taken from the column 50 through an outlet above that of the inlet to the column for the argon-enriched oxygen withdrawn from the distillation column 10. This vapour is then condensed in the condenser 74 and is returned to liquid in the column 50 at a level where the composition of the liquid corresponds approximately to that of the condensate. Moreover, liquid oxygen from the bottom of the column 50 is passed to the top of the mixing column 20 as will be described below. In other respects the arrangement of columns shown in Figure 2 is generally similar to that shown in Figure 1.

The plant shown in Figure 2 does, however, contain a number of features not shown in Figure 1 or described with respect to thereto. In particular, the plant shown in Figure 2 has the following features:

a) a nitrogen distribution and refrigeration system which in addition to providing a working fluid, comprising nitrogen, to the reboiler 52 of the argon column 50, also provides nitrogen to cool the condensers 54, 74 and 30 and nitrogen to heat the reboiler 14;

b) a reversing heat exhanger system for purifying and cooling the incoming air.

The nitrogen distribution system includes five nitrogen distribution pots, 80, 82, 84, 86 and 88, all operating at different pressures from one another. Each of the pots, 80, 82, 84, 86 and 88 receives and distributes gaseous and liquid nitrogen streams performing heat pumping duty. The pots 80 and 82 provide nitrogen at higher pressure than the operating pressure of the columns 10 and 20 to respectively the reboiler 14 and the condenser 30. The pressure in the pot 80 is higher than that of the pot 82. The pot 82 houses the condenser 30. The pot 84 operates at approximately the same pressure as that of the columns 10 and 20 and provides an intermediate region of the vapour path from the outlet 26 of the mixing column 20 to the reboiler 14 of the distillation column 10 and also an intermediate region of the liquid path from the reboiler 14 of the column 10 to the inlet 8 to the column 10.

The pots 86 and 88 operate at lower pressures than those at which the columns 10 and 20 operate. Pot 86 provides cooling for the condenser 74 associated with the argon column 50 while the pot 88, which operates at a lower pressure than the pot 86, provides cooling for the condenser 54 associated with the argon column 50.

The condensers 74 and 54 are located in the pots 86 and 88 respectively.

In addition to providing gaseous nitrogen to the reboiler 14 and receiving liquid nitrogen therefrom, the pot 80 receives a compressed gaseous nitrogen stream from a multistage compressor 90. In order to provide cooling for nitrogen supplied to the pots 80, 82, 84, 86 and 88, a sequence of heat exchangers 92, 94, 96 and 98 is provided. A compressed nitrogen stream leaving the conmpressor 90 flows through the heat exchanger 92 from its warm end at about ambient temperature and is cooled to about its dew point and is then introduced into the pot 80. A stream of liquid nitrogen is withdrawn from the bottom of the pot 80 (at a rate equal to that which the compressed nitrogen is introduced into the pot 80), and is then divided in two. One part of the stream is expanded through valve 100 and is then returned through the heat exchanger 92 countercurrently to the aforesaid compressed nitrogen stream. After being warmed to about ambient temperature, this nitrogen is then returned to the highest pressure stage of the compressor 90 for recompression.

That part of the liquid nitrogen stream withdrawn from the bottom of the pot 80 that is not expanded through the valve 100 is further reduced in temperature in the heat

exchanger 94: it enters the heat exchanger 94 at its warm end, is withdrawn from an intermediate region thereof, is passed through an expansion valve 102 and is then introduced as liquid into the pot 82.

The pot 82, as well as providing a liquid nitrogen stream to condense the oxygen in the condenser 32 associated with the mixing column 20 and receiving the resultant vaporised nitrogen, also provides a gaseous nitrogen stream which provides cooling for the heat exchangers 94 and 92 and is then recompressed in a stage of the compressor 90. Thus, the gaseous nitrogen stream is withdrawn from the top of the pot 82 and is introduced into the heat exchanger 94 at a region intermediate its cold and warm ends and then flows through the heat exchanger 94 leaving the heat exchanger at its warm end. This nitrogen stream then passes through the heat exchanger 92 from its cold end to its warm end, being recompressed in the compressor 90.

A liquid nitrogen stream is also withdrawn from the pot 82, and, after passage through the heat exchanger 94 from its warm to its cold end, is expanded through valve 104 into the pot 84. The pot 84, as well as receiving nitrogen from the outlet 26 of the mixing column 20, passing nitrogen to the condenser 14, receiving return

nitrogen from the condenser 14 and returning nitrogen to the top of the distillation column 10 through the inlet 16, also provides liquid nitrogen to the pots 86 and 88 and returns gaseous nitrogen to the compressor 90. Thus, a gaseous nitrogen stream is withdrawn from the top of the pot 84 and flows through the heat exchangers 94 and 92, passing through each heat exchanger from its cold end to its warm end, and is then compressed in a stage of the compressor 90. Thus gaseous nitrogen stream is mixed with some liquid withdrawn from some of the pot 84. Further liquid from the bottom of the pot 84 passes through a heat exchanger 96 flowing from its warm to its cold end. Part of this liquid nitrogen is then expanded through valve 106 into the pot 86, while the remainder flows through the heat exchanger 98 from its warm to its cold end and is expanded through valve 108 into the pot 88. A gaseous nitrogen stream is withdrawn from the top of the pot 86 and is returned to the compressor 90 flowing through the heat exchangers 96, 94 and 92 in sequence. Similarly, a gaseous nitrogen stream is withdrawn from the top of the pot 88 and flows through the heat exchangers 98, 96, 94 and 92, in sequence, and is recompressed in the compressor 90.

As well as providing cooling and warming of the nitrogen streams, the heat exchanger 94 is employed to sub-cool the argon-enriched oxygen stream withdrawn from the column 10 through the outlet 42. In addition, liquid oxygen withdrawn from the argon column 50 through the outlet 56 is pumped by a pump 110 through the heat exchanger 94 countercurrently to the flow of the argon-enriched liquid oxygen stream and is then mixed with the liquid oxygen stream pumped from the outlet 6. The resulting mixture is introduced into a pot 112 where it is mixed with gaseous oxygen leaving the top of the mixing column 20 through the outlet 32. The resulting 2-phase mixture is withdrawn from the pot 112 and is fully condensed in the condenser 30 before being returned to the column 20 through the inlet 22.

In order to provide cooling and cleaning for the incoming air stream, reversing heat exchangers 114 and 116 are provided. The air is cooled to its dew point by passage through the heat exchangers 114 and 116. Refrigeration for the heat exchangers is provided by taking the product oxygen stream and the mixed nitrogen-oxygen stream withdrawn from the column 20 through the outlet 28 and passing through the heat exchange rs 116 and 114 countercurrently to the incoming air. (The heat exchangers 116 and 114 are used instead of the heat

exchanger 29 shown in Figure 1). A part of the aforesaid nitrogen-oxygen stream is however divided from the main stream upstream of the cold end of the heat exchange 116 and is passed through the heat exchanger 116 countercurrently to the incoming air stream. It is then expanded to a pressure a little above atmospheric pressure in an expansion turbine 118 with the performance of external work. The resulting nitrogen stream provides some refrigeration for the heat exchanger 92 and is then returned through the heat exchanger 116 flowing cocurrently with the incoming air stream. The expanded air is then returned through the heat exchanger 116 countercurrently to the incoming air flow and then passes through the heat exchanger 114 from the cold to the warm end thereof. The nitrogen-oxygen streams that leave the warm end of the heat exchanger 114 may be further expanded to recover work.

During its passage through the heat exchanger 114 and 116, carbon dioxide, water vapour and other low volatility impurities are deposited. In a manner well known in the art, once the cleaning ability of the reversing heat exchanger 114 and 116 begins to decline, the passages traversed by the incoming and returning air streams are switched so that the returning air streams can be used to resublime solid impurities deposited on the heat exchange

surfaces. Thus, the heat exchangers 114 and 116 may be used continuously to provide purified air to the inlet of the distillation column 10. It is desirable to employ relatively high and low pressure streams to effect the cleaning of the heat exchangers 116 and 114 as difficulties can arise if just a relatively high pressure air stream is used, that is if none of the air is expanded through the turbine 118.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS;

1. A method of separating air, comprising the steps of:

a) passing a stream of air into a first distillation column;

b) withdrawing an oxygen-rich liquid from a bottom region of the first distillation column and passing it to a top region of a mixing zone;

c) passing nitrogen-rich vapour from the first distillation column to a bottom region of the mixing zone;

d) establishing through the mixing zone a downward flow of liquid that becomes progressively richer in nitrogen in the direction of liquid flow and an upward flow of vapour that becomes progressively richer in oxygen in the direction of vapour flow;

e) passing liquid nitrogen from the mixing zone to the first distillation column to act as reflux;

f) withdrawing an oxygen-depleted mixed stream comprising oxygen and nitrogen from a chosen level of the mixing zone;

g) employing the mixed stream to support combustion of a fluid fuel and expanding the resulting combustion products in a turbine;

h) withdrawing from the first distillation column a stream of argon-containing fluid whose argon concentration is greater than that of the air stream, and separating an argon product from the argon-containing stream in a second distillation column; and

i) withdrawing an oxygen product from one or both of the distillation columns.

2. A method as claimed in claim 1, in which the turbine is employed to drive at least one compressor that compresses the air upstream of the first distillation column.

3. A method as claimed in claim 1 or claim 2, in which the mixed stream is preheated upstream of the combustion zone by heat exchange with the combustion products downstream of their passage through the turbine.

4. A method as claimed in any one of the preceding claims, in which the mixed stream contains not less than 15% by volume of oxygen.

5. A method as claimed in any one of the preceding claims, in which the mixing zone and first distillation column operate at pressures of at least 3 atmospheres absolute.

6. A method as claimed in claim 5, in which the mixing zone and first distillation column operate at pressures in the range of 5 to 10 atmospheres absolute.

7. A method of separating air, substantially as herein described with reference to Figure 1 or Figure 2 of the accompanying drawings.

8. Apparatus for separating air, comprising:

a) means for passing a stream of air into a first distillation column;

b) means for withdrawing an oxygen-rich liquid from a bottom region of the first distillation column and passing it to a top region of a mixing zone;

c) means for passing nitrogen rich vapour from the first distillation column to a bottom region of the mixing zone;

d) liquid-vapour contact means for establishing through the mixing zone a downward flow of liquid that becomes progressively richer in nitrogen in the direction of liquid flow and an upward flow of vapour that becomes progressively richer in oxygen in the direction of vapour flow;

e) means for passing liquid nitrogen from the mixing zone to the first distillation column to act as reflux;

f) means for withdrawing an oxygen-depleted mixed stream comprising oxygen and nitrogen from a chosen level of mixing zone;

AUSTRALIAN PATENT OFFICE MB

g) means for burning a fluid fuel using the mixed stream to support combustion and a turbine for expanding the resulting combustion products;

h) means for withdrawing from the first distillation column a stream of argon-containing fluid whose argon concentration is greater than that of the air stream, said means communicating with a second distillation column for separating an argon product from the argon containing stream; and

i) means for withdrawing an oxygen product from one or both of the distillation columns.

9. Apparatus as claimed in claim 8 in which the turbine is coupled to the drive of a compressor adapted to compress the air upstream of the first distillation column.

10. Apparatus as claimed in claim 8 or claim 9, including a heat exchanger for preheating the mixed stream intermediate the mixing zone and the means for burning a fluid fuel by heat exchange with the combustion products of the fluid fuel burning means downstream of the turbine.

AUSTRALIAN PATENT OFFICE

11. Apparatus for separating air substantially as described herein with reference to Figures 1 and 2 of the accompanying drawings.

DATED this 10th day of February 1992

THE BOC GROUP plc

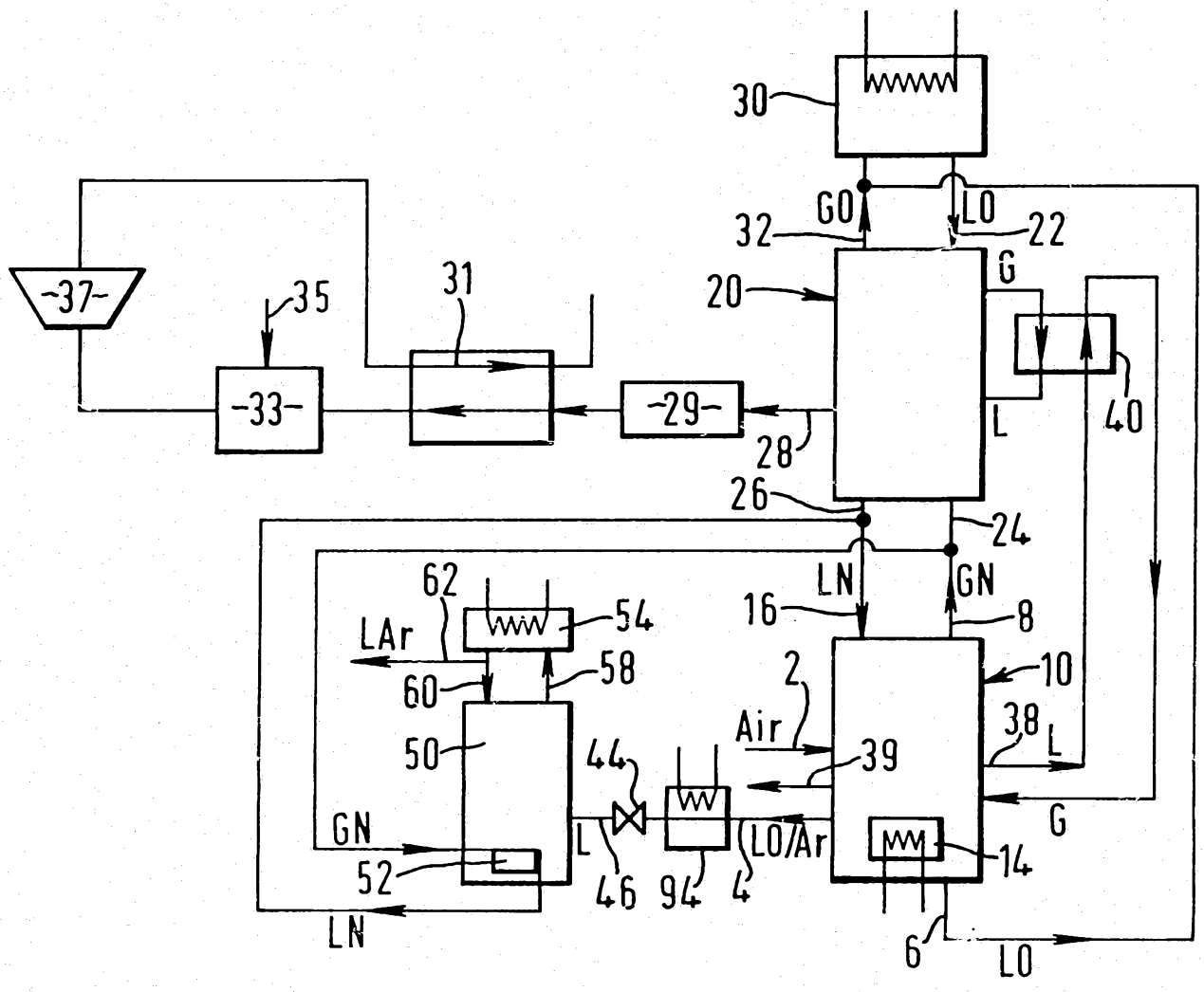
Attorney: LEON K. ALLEN

Fellow Institute of Patent Attorneys of Australia

of SHELSTON WATERS

AUSTRALIAN PATENT OFFICE MTS

FIG. 1.



34 696/89

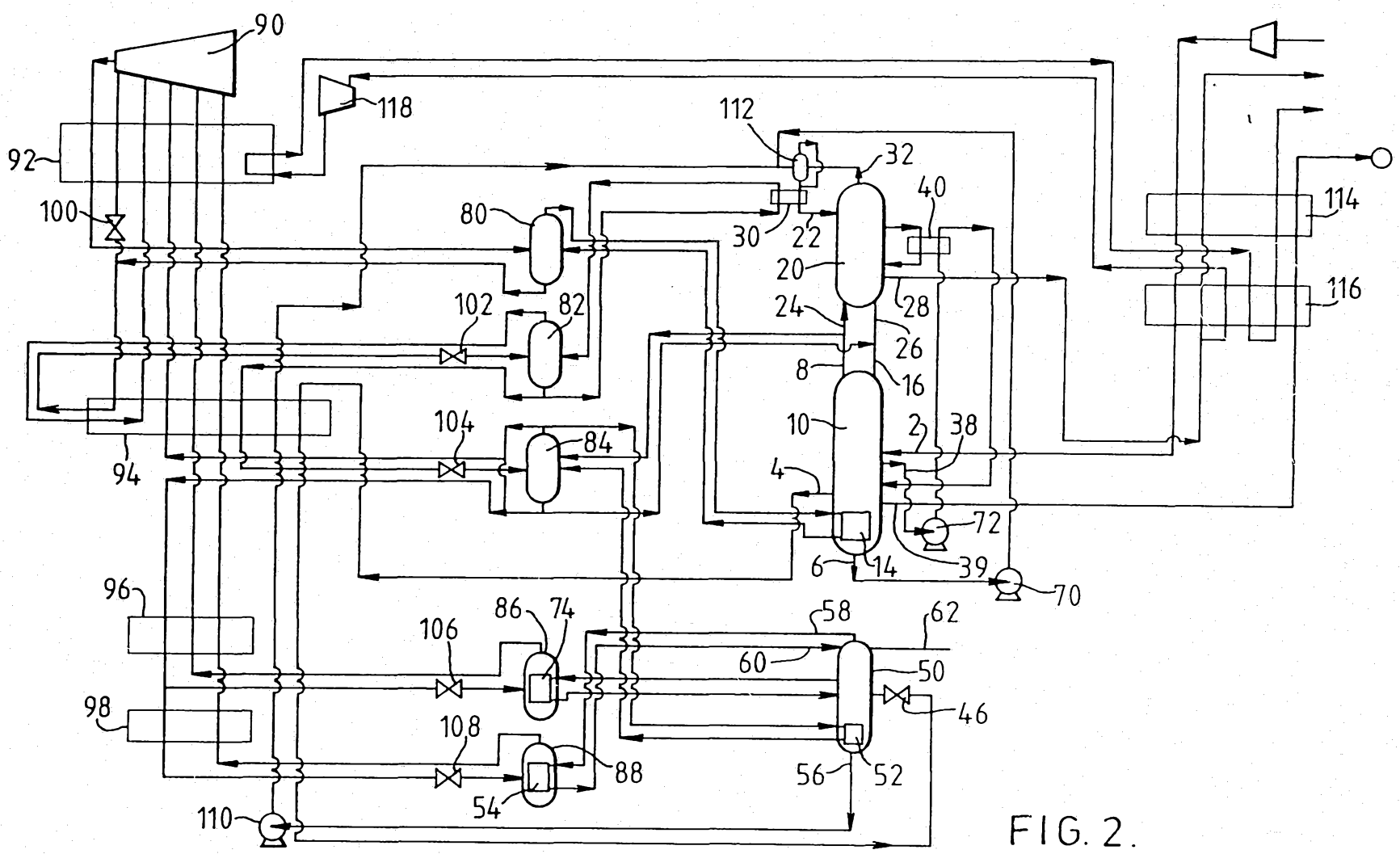

90
118
92
100
112
32
40
114
116
80
30
22
20
28
24
26
102
82
8
16
10
2
38
104
84
4
94
72
6
14
39
70
96
86
74
58
62
106
60
50
98
46
108
88
56
52
54
110

FIG. 2.