INSTRUCTIONS

(a) If Convention application insert "Convention"

(a) CONVENTION

AUSTRALIA

642991

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(b) Delete one

APPLICATION FOR A (b) STANDARD/~~PETTY~~ PATENT

(c) Insert FULL name(s) of applicant(s)

I/~~WE~~ (c) L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCEDES GEORGES CLAUDE

(d) Insert FULL address(es) of applicant(s)

of (d) 75, quai d'Orsay - 75321 PARIS CEDEX 07 (FRANCE)

(e) Delete one

hereby apply for the grant of a (e) Standard/~~Petty~~ Patent for an invention entitled

(f) Insert TITLE of invention

(f) " PROCESS AND APPARATUS FOR THE PRODUCTION OF GASEOUS NITROGEN AND SYSTEM FOR SUPPLYING CORRESPONDING NITROGEN "

(g) Insert "complete" or "provisional" or "petty patent"

which is described in the accompanying (g) complete specification.

(Note: The following applies only to Convention applications)

Details of basic application(s)

(h) Insert number, country and filing date for the/or each basic application

	Application No.	Country	Filing Date
(h)	90 04 566	FRANCE	April 10, 1990

Address for Service: PHILLIPS ORMONDE AND FITZPATRICK
Patent and Trade Mark Attorneys
367 Collins Street
Melbourne, Australia 3000

(i) Insert date of signing

Dated (i) March 5, 1991

(j) Signature of applicant(s) (For body corporate see headnote*)

(k) Corporate seal if any

(k) L'AIR LIQUIDE
Société Anonyme pour l'Etude et l'Exploitation des Procédés Georges Claude
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(France)

Jacques R. BEGHAIN
General Counsel

Note: No legalization or other witness required

PHILLIPS ORMONDE AND FITZPATRICK
Patent and Trade Mark Attorneys
367 Collins Street
Melbourne, Australia

F17/2/83

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DECLARATION FOR A PATENT APPLICATION

▼ INSTRUCTIONS

(a) Insert "Convention" if applicable
(b) Insert FULL name(s) of applicant(s)

In support of the (a) convention application made by
(b) L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCEDES GEORGES CLAUDE

(c) Insert "of addition" if applicable
(d) Insert TITLE of invention

(hereinafter called "applicant(s) for a patent (c) for an invention entitled (d)
" PROCESS AND APPARATUS FOR THE PRODUCTION OF GASEOUS NITROGEN AND SYSTEM FOR SUPPLYING CORRESPONDING NITROGEN "

(e) Insert FULL name(s) AND address(es) of declarant(s) (See headnote*)

I/~~We~~ (e) Jacques R. BEGHAIN
of L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCEDES GEORGES CLAUDE - 75, quai d'Orsay- 75321 PARIS CEDEX 07 (FRANCE)

do solemnly and sincerely declare as follows:

1. ~~I am/We are the applicant(s)~~
(or, in the case of an application by a body corporate)
1. I am/~~We are~~ authorized to make this declaration on behalf of the applicant(~~s~~).
2. ~~I am/We are the actual inventor(s) of the invention~~
(or, where the applicant(s) is/are not the actual inventor(s))

(f) Insert FULL name(s) AND address(es) of actual inventor(s)

2. (f) . François DARCHIS of Liquid Air Corporation-California Plaza- 2121 North California Bld - 94404 WALNUT CREEK - CA 94596 (U.S.A.);
. François VENET of 36, rue Jouffroy- 75017 PARIS (FRANCE);
. Jean-Louis GIRAULT of 16, quai de Rome- B 4000 LIEGE (BELGIQUE);
. Maurice GRENIER of 3, rue Camille Tahan - 75018 PARIS (FRANCE);
. Patrick JOZON of 123, rue Anatole France- 92300 LEVALLOIS-PERRET (FRANCE)

(g) Recite how applicant(s) derive(s) title from actual inventor(s) (See headnote**)

~~is~~/are the actual inventor(s) of the invention and the facts upon which the applicant(~~s~~) is/are entitled to make the application are as follows:
(g) The applicant is the assignee of the invention from the actual inventor.

(Note: *Paragraphs 3 and 4 apply* only *to Convention applications)*

(h) Insert country, filing date, and basic applicant(s) for the/or EACH basic application

3. The basic application(~~s~~) for patent or similar protection on which the application is based is/~~are~~ identified by **country, filing date, and basic applicant(~~s~~)** as follows:
(h) FRANCE
April 10, 1990 under n° 90 04 566
by L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCEDES GEORGES CLAUDE

4. The basic application(~~s~~) referred to in paragraph 3 hereof was/~~were~~ the first application(~~s~~) made in a Convention country in respect of the invention the subject of the application.

(k) Insert PLACE of signing
(l) Insert DATE of signing
(m) Signature(s) of declarant(s)

Note: No legalization or other witness required

Declared at (k) PARIS

Dated (l) March 5, 1991

L'AIR LIQUIDE (m)
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General Counsel

To: The Commissioner of Patents

P18/7/78

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367 Collins Street
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AU9174181

(12) PATENT ABRIDGMENT (11) Document No. AU-B-74181/91
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 642991

(54) Title
PROCESS AND APPARATUS FOR THE PRODUCTION OF GASEOUS NITROGEN AND SYSTEM FOR SUPPLYING CORRESPONDING NITROGEN

International Patent Classification(s)
(51)[5] **C01B 021/04**

(21) Application No. : **74181/91** (22) Application Date : **09.04.91**

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90 04566	**10.04.90**	**FR FRANCE**

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L'AIR LIQUIDE SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCEDES GEORGES CLAUDE

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(56) Prior Art Documents
EP 190355
FR 2225705

(57) Claim

1. Process for producing gaseous nitrogen with variable flow by means of air distillation equipment, the equipment including an HPN air-separation column for producing gaseous nitrogen in a product line, and having a head portion and a bottom portion which is fed with compressed air, and liquid nitrogen storage means supplying a flow of liquid nitrogen to the head portion of the column, wherein a nominal flow of liquid nitrogen from the storage means is supplied to the head portion of the column after the column produces a nominal flow of gaseous nitrogen, including the steps of sensing the pressure of the gaseous nitrogen in the product line and of selectively modulating the flow of liquid nitrogen supplied from the storage means to the head portion in a range above said nominal flow depending on the value of the sensed pressure to accommodate variations in the demand for gaseous nitrogen.

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COMPLETE SPECIFICATION
(ORIGINAL)

Class Int. Class

Application Number:
Lodged:

Complete Specification Lodged:
Accepted:
Published:

Priority

Related Art:

Applicant(s):

L'Air Liquide, Societe Anonyme pour l'Etude et l'Exploitation des Procedes Georges Claude
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367 Collins Street
Melbourne 3000 AUSTRALIA

Complete Specification for the invention entitled:

PROCESS AND APPARATUS FOR THE PRODUCTION OF GASEOUS NITROGEN AND SYSTEM FOR SUPPLYING CORRESPONDING NITROGEN

Our Ref : 209713
POF Code: 1290/43509

The following statement is a full description of this invention, including the best method of performing it known to applicant(s):

6006

BACKGROUND OF THE INVENTION

(a) Field of the Invention

The present invention relates to the production of gaseous nitrogen. It is more particularly concerned with satisfying moderate (typically 100 to 1000 Nm^3/h) and variable needs in nitrogen of high purity, i.e. typically containing less than 0.1% oxygen. In the present specification, flows are considered in terms of weight.

(b) Description of the Prior Art

High purity nitrogen is normally obtained by cryogenic means. Where small consumptions of nitrogen are required, construction of known self-contained production units is prohibitively expensive, in the case of automated installations, and slightly less expensive in non-automated installations, where, however, high labour costs increase the price of nitrogen for the consumer.

A more economical solution consists in utilizing an evaporator, i.e. a container for liquid nitrogen with large capacity, for example many tens of thousands of liters, from which liquid nitrogen is withdrawn and vaporized. This solution is not very satisfactory on any energy point of view, because the frigorific energy contained in liquid nitrogen is lost, and, in addition, it requires the presence, at a relatively small distance, of a unit for the production of liquid nitrogen, so that the cost of supplying the evaporator with a tank-truck remains moderate.

SUMMARY OF THE INVENTION

The invention aims at providing technology which produces variable and moderate quantities of gaseous nitrogen at reduced cost, and at increased distances from a unit for the production of liquid nitrogen.

For this purpose, the invention aims at a process

for producing gaseous nitrogen with variable flow by means of air distillation equipment, the equipment including an HPN air-separation column for producing gaseous nitrogen in a product line, and having a head portion and a bottom portion which is fed with compressed air, and liquid nitrogen storage means supplying a flow of liquid nitrogen to the head portion of the column, wherein a nominal flow of liquid nitrogen from the storage means is supplied to the head portion of the column after the column produces a nominal flow of gaseous nitrogen, including the steps of sensing the pressure of the gaseous nitrogen in the product line and of selectively modulating the flow of liquid nitrogen supplied from the storage means to the head portion in a range above said nominal flow depending on the value of the sensed pressure to accommodate variations in the demand for gaseous nitrogen.

In the present description, the term "an HPN air-separation column" means a simple distillation column provided with a head condenser. In such a column, the air to be treated, compressed under a pressure of the order of 6 to 12 bars, from which water and CO_2 have been removed and cooled to the vicinity of its dew point, is introduced at the base of the column. The "rich liquid" (oxygen enriched air) collected in the vat portion of the column is expanded and vaporized in the head condenser, after which it is removed as a residue. The gaseous nitrogen produced is withdrawn from the head portion of the column.

According to advantageous characteristics of the invention:

- said nominal flow is introduced during a period of time at least equal to a predetermined length of time which is sufficient to ensure a predetermined level of refrigerating liquid in the head condenser of the column;
- to produce a flow of gaseous nitrogen which is higher than the nominal flow, a flow of make-up liquid nitrogen originating from said source is vaporizd outside the column;
- at least a portion of the make-up flow is

vaporized by heat exchange with the incoming air upstream of the inlet of this air in the compressor of the apparatus.

It is also an object of the invention to provide an apparatus for the production of gaseous nitrogen with variable flow, adapted for the operation of such process. This apparatus including:

- an air distillation column having a head portion with a gaseous nitrogen outlet discharging in a product line, a liquid nitrogen inlet, and a bottom portion with a feed air inlet;
- a pressure sensor in the product line, capable of furnishing a pressure signal;
- air feed means for feeding air to the feed air inlet via a feed air line including a compressor;
- a liquid nitrogen storage means having an outlet and
- a first liquid nitrogen line between the outlet of the storage means and the liquid nitrogen inlet of the column and including flow control means responsive to said pressure signal.

It is also an object of the present invention to provide a system for supplying gaseous nitrogen to a plurality of users, this system including:

- a central unit for the production of large quantities of liquid nitrogen;
- at least one tank truck for remote dispatch of liquid nitrogen produced at the central unit;
- within a first radius around the central unit, at least one vaporizing unit for vaporizing liquid nitrogen, having a liquid nitrogen tank for supplying gaseous nitrogen to a first user;
- within a range between the first radius and a second radius greater than the first radius, at least one apparatus for the production of gaseous nitrogen with variable flow, for supplying gaseous nitrogen to a second user comprising:
- an air distillation column having a head portion with a gaseous nitrogen outlet

discharging in a product line, a liquid nitrogen inlet, and a bottom portion with a feed air inlet;

- a pressure sensor in the product line, capable of furnishing a pressure signal;
- air feed means for feeding air to the feed air inlet via a feed air line including a compressor;
- a liquid nitrogen storage means having an outlet, and
- a first liquid nitrogen line between the outlet of the storage means and the liquid nitrogen inlet of the column and including flow control means responsive to said pressure signal, whereby the liquid nitrogen tank of the vaporizing unit and the liquid nitrogen storage means of the apparatus are periodically refilled from the central unit by the tank with liquid nitrogen.

-in a first radius around the production unit, a series of liquid nitrogen vaporizers which can be supplied by the tank-truck;

-between the first radius and the second radius, which is more important than the first one, a series of apparatuses as defined above, wherein the container of these installations can be supplied by the tank-truck.

BRIEF DESCRIPTION OF THE DRAWINGS

An embodiment of the invention will now be described with reference to the annexed drawings, in which:

-Figure 1 is a schematic illustration of a system for the production of gaseous nitrogen according to the invention;

-Figure 2 is a schematic representation of an apparatus according to the invention;

-Figure 3 is a diagram illustrating the process according to the invention.

DESCRIPTION OF PREFERRED EMBODIMENTS

The system for supplying gaseous nitrogen represented in Figure 1 essentially comprises:

-a unit 1 for the production of liquid nitrogen;

-within a radius R1 around this unit, a certain number of liquid nitrogen vaporizers 2, each consisting of a liquid nitrogen storage 3 of large capacity provided with a liquid withdrawing duct 4 connected to a utilization duct 5 via vaporizer 6, for example of the atmospheric type. Such vaporizers are well known in the art;

-between radius R1 and radius R2 > R1 around unit 1, a certain number of apparatuses 7 such as those of Figure 2, each of these apparatuses comprising a container for liquid nitrogen 8;

-at least one tank-truck 9, and generally a fleet of such trucks, adapted to supply the vaporizers 2 and containers 8 of the apparatuses 7 with liquid nitrogen produced by unit 1; and possibly

-a teletransmission system (not represented) connecting each vaporizer 2 and each apparatus 7 to unit 1 to ensure the management of the deliveries of liquid nitrogen by the tank-truck(s).

The apparatus 7 represented in Figure 2 essentially comprises:

-the above-mentioned container 8;

-a cold box 9 containing on the one hand an air distillation column 10 of the type HPN (High Purity Nitrogen), on the other hand a heat exchange line 11;

-a device 12 for purifying air by adsorption;

-an auxiliary heat exchanger 13;

-an air compressor 14; and

-an air refrigerating device 15.

The operation of apparatus 7 will now be described with reference to Figures 2 and 3. On the diagram of Figure 3 the time t has been shown in abscissae, and a plurality of parameters, whose meaning will appear hereinbelow, are given as ordinates.

The first description will be the novel operation of the apparatus, i.e. at permanent rate where column 10 via withdrawing duct 16, exiting in the head portion of the column, produces a constant flow of gaseous nitrogen which is equal to the nominal flow DN for which the column has been

designed. Duct 16 ends in a utilization duct 17 provided with a buffer capacity 18 and, downstream of the latter, a pressure pick-up 19.

In this operation (corresponding to $t < t0$ on Figure 3), the nitrogen consumption C (Fig. 3(a)) is constant and equal to the nominal flow DN, and the pick-up 19 indicates a constant pressure P (Figure 3(e)). Via a duct 20 provided with an electromagnetic valve 21 for any kind of adjustment, a mean to small flow of liquid nitrogen, for example equal to about 5% DN (Fig. 3(b)), is introduced in the head portion of the column 10 and serves to ensure that cold conditions are maintained and also to increase the rate of reflux of the column. The heat exchanger 13 is inactive. The entering air compressed by compressor 14, precooled by air cooling device 15, purified in a device 12 and cooled to the vicinity of its dew point in exchange line 11, is introduced at the bottom of column 10. The rich liquid collected in the vat portion of the column is expanded in an expansion valve 22, vaporized in the head condenser 23 of the column, warmed with countercurrent air in the heat exchange, then used to regenerate device 12 before being withdrawn via duct 24 as residual gas of the apparatus.

It will be presumed that at instant $t0$, the consumption (or demand) of gaseous nitrogen starts to increase (Fig.3(a)). The pressure at 19 decreases (Fig.3(e), which provokes the opening of a valve 25 provided along a duct 26 which connects the bottom of container 8 to the cold end of exchanger 13. A flow DV1 of nitrogen (Fig. 3(c)) is thus vaporized by cooling with countercurrent entering air to a moderate temperature, for example, of the order of $-20^{\circ}C$, then this gaseous nitrogen is sent to a reservoir 18. Following this, the compressor sucks

an increased mass flow of air and the production DF (distilled flow) of the column increases (Fig. 3(d)). Simultaneously, the flow of liquid nitrogen which is introduced through duct 20 increases to some extent (Fig. 3(b)), to keep the level of rich liquid constant in the condenser 23.

If, from t1 to t2, the consumption continues to increase (Fig. 3(a)), an additional vaporization of liquid nitrogen (Fig. 3(c)) is carried out in an auxiliary vaporizer 27 as a result of the opening of a valve 28 without modifying the flow produced by distillation (Fig. 3(d)), after which this gaseous nitrogen is also sent to the reservoir 19. This opening of the valve 28 takes place when the pressure reaches a low value P1 (Fig. 3(e)). The total vaporized flow DV2, which is the sum of the flows vaporized in the exchanger 13 and in vaporizer 27, corresponds to the make-up nitrogen required to satisfy the demand. This vaporization of liquid nitrogen brings back the pressure at 19 to nominal value P (Fig. 3(e)).

It should be noted that after a certain period of time, frosting may start to form in the exchanger 13. This is detected by a temperature detector 29 which is disposed at the nitrogen outlet of this exchanger and closes valve 25.

After a phase of stabilization (from t2 to t3), when consumption decreases, the pressure at 19 increases, which provokes a stop of the vaporization of nitrogen (closing of valves 25 and 28), then, when the pressure reaches a high value P2, the apparatus is stopped, for example through compressor 14 (instant t4).

When the consumption of gaseous nitrogen resumes (instant t5), the pressure decreases, and when it reaches nominal value P1 (instant t6), a

self-starting electromagnetic valve 30 mounted as a bypass of the electromagnetic valve 21 and normally closed, is opened. This electromagnetic valve 30 is such that, in opened position, it allows for the passage of a flow of liquid nitrogen which is at least equal to the nominal flow DN. Its closing takes place when two conditions are fulfilled:

(a) a predetermined time T has passed since its opening; and

(b) the level of rich liquid in condenser 23 is at least equal to a predetermined value.

Time T is determined so as to ensure that, whatever the condition, hot or cold, of the apparatus when starting same, cold conditions and the correct charge of liquid at each level of a column are obained. It is possible, for example, to select a time T of the order of 2 minutes.

The electromagnetic valve 30 thus closes at instant $\underline{t}7$ indicated on Fig. 3.

Figure 3 also represents instant $\underline{t}8 < \underline{t}7$ and $\underline{t}9 > \underline{t}7$ for which, respectively, the consumption C increases beyond the nominal value after which it stabilizes, the same phenomenon as described above being then automatically reproduced (vaporization of nitrogen and variations of the pressure and the flow of nitrogen produced by the column).

It will therefore be seen that the apparatus can very easily operate entirely automatically in spite of a structure and automatisation means which are very inexpensive. In particular, as soon as the apparatus is started, a flow of nitrogen at least equal to the demand is vaporized in the column, which simultaneously ensures the necessary cold input and the production of required gaseous nitrogen, and moreover prevents the entering air from

rising in the column. After this, the nitrogen introduced into the reservoir 18 has immediately the required purity.

As a variant, the two electromagnetic valves 21 and 30 may be replaced by a single cryogenic valve with variable flow.

It will be noted that for proper operation, the apparatus needs only one electrical connection, which has been illustrated symbolically on Fig. 1.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. Process for producing gaseous nitrogen with variable flow by means of air distillation equipment, the equipment including an HPN air-separation column for producing gaseous nitrogen in a product line, and having a head portion and a bottom portion which is fed with compressed air, and liquid nitrogen storage means supplying a flow of liquid nitrogen to the head portion of the column, wherein a nominal flow of liquid nitrogen from the storage means is supplied to the head portion of the column after the column produces a nominal flow of gaseous nitrogen, including the steps of sensing the pressure of the gaseous nitrogen in the product line and of selectively modulating the flow of liquid nitrogen supplied from the storage means to the head portion in a range above said nominal flow depending on the value of the sensed pressure to accommodate variations in the demand for gaseous nitrogen.

2. Process according to claim 1, including the steps of increasing the flow of liquid nitrogen supplied from the storage means to the head of the column when the sensed pressure decreases below a limited value as a result of an increase in the demand for gaseous nitrogen.

3. Process according to claim 2, further including the step of selectively withdrawing an additional flow of the liquid nitrogen from the storage means depending on the sensed pressure, and vaporizing said additional flow for addition, in the product line, to the flow of gaseous nitrogen from the column to meet a substantial increase in the demand for gaseous nitrogen.

4. Process according to claim 3, wherein said additional flow of liquid nitrogen is vaporized at least partially by heat-exchange with feed air supplied to the column.

5. Process according to claim 4, wherein said additional flow of liquid nitrogen is further vaporized at least partially in an auxiliary heat exchanger.

6. Process according to any preceding claim, wherein said nominal flow of liquid nitrogen supplied from the

storage means to the column corresponds to about 5% by weight of the nominal flow of gaseous nitrogen produced by the column.

7. Apparatus for the production of gaseous nitrogen with variable flow including:

- an air distillation column having a head portion with a gaseous nitrogen outlet discharging in a product line, a liquid nitrogen inlet, and a bottom portion with a feed air inlet;
- a pressure sensor in the product line, capable of furnishing a pressure signal;
- air feed means for feeding air to the feed air inlet via a feed air line including a compressor;
- a liquid nitrogen storage means having an outlet and
- a first liquid nitrogen line between the outlet of the storage means and the liquid nitrogen inlet of the column and including flow control means responsive to said pressure signal.

8. Apparatus according to claim 7, wherein said flow control means includes first and second control valves arranged in parallel in a branched portion of the first liquid nitrogen line.

9. Apparatus according to claim 7 or 8, including a first heat exchange means through which passes said feed air line and further including a second liquid nitrogen line between the outlet of the storage means and the product line, the second liquid nitrogen line passing through the first heat exchange means and having a third control valve responsive to said pressure signal.

10. Apparatus according to claim 9, wherein said first heat exchange means is coupled to said feed air line upstream of said compressor.

11. Apparatus according to claim 7, 8, 9, further including a third liquid nitrogen line between the outlet of the storage means and the product line, including a vaporizer and a fourth control valve responsive to said pressure signal.

12. A network for in situ supply of gaseous nitrogen to

at least two remote users including:

- a central unit for the production of large quantities of liquid nitrogen;
- at least one tank truck for remote dispatch of liquid nitrogen produced at the central unit;
- within a first radius around the central unit, at least one vaporizing unit for vaporizing liquid nitrogen, having a liquid nitrogen tank for supplying gaseous nitrogen to a first user;
- within a range between the first radius and a second radius greater than the first radius, at least one apparatus for the production of gaseous nitrogen with variable flow, for supplying gaseous nitrogen to a second user comprising:
- an air distillation column having a head portion with a gaseous nitrogen outlet discharging in a product line, a liquid nitrogen inlet, and a bottom portion with a feed air inlet;
- a pressure sensor in the product line, capable of furnishing a pressure signal;
- air feed means for feeding air to the feed air inlet via a feed air line including a compressor;
- a liquid nitrogen storage means having an outlet, and
- a first liquid nitrogen line between the outlet of the storage means and the liquid nitrogen inlet of the column and including flow control means responsive to said pressure signal, whereby the liquid nitrogen tank of the vaporizing unit and the liquid nitrogen storage means of the apparatus are periodically refilled from the central unit by the tank with liquid nitrogen.

13. Process substantially as hereinbefore described and illustrated in Figures 1 and 2.

14. Apparatus substantially as hereinbefore described and illustrated in Figures 1 and 2.

DATED: 6 August 1993

PHILLIPS ORMONDE & FITZPATRICK
Attorneys For:
L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCEDES GEORGES CLAUDE

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David B Fitzpatrick

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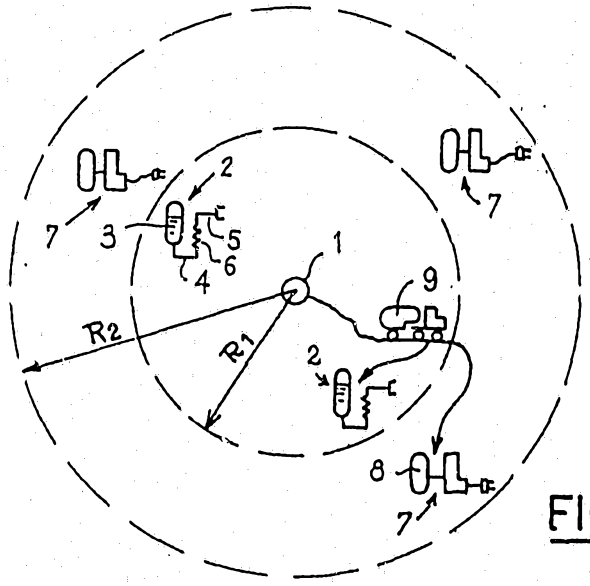


FIG.1

FIG.2

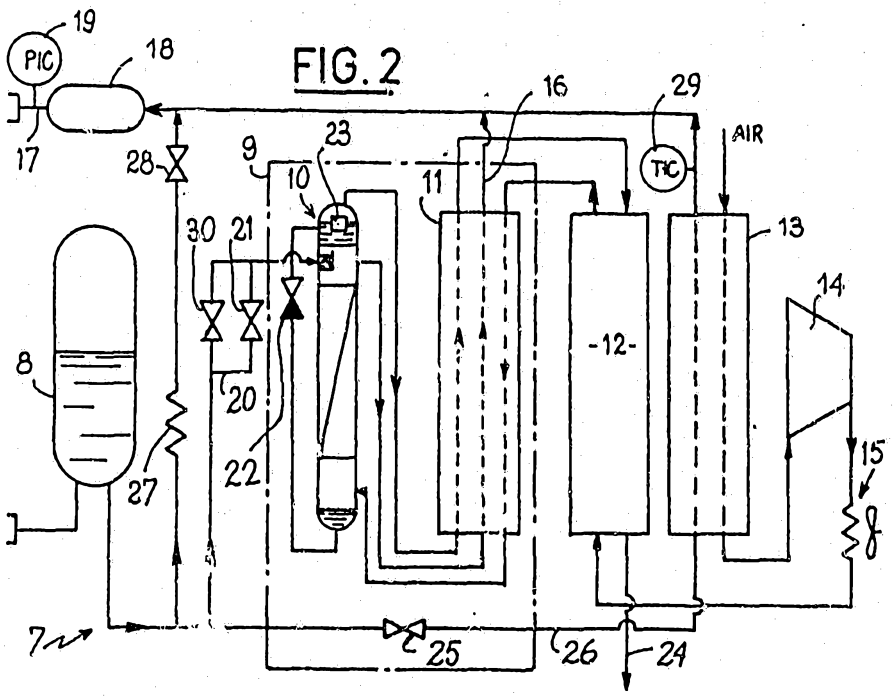
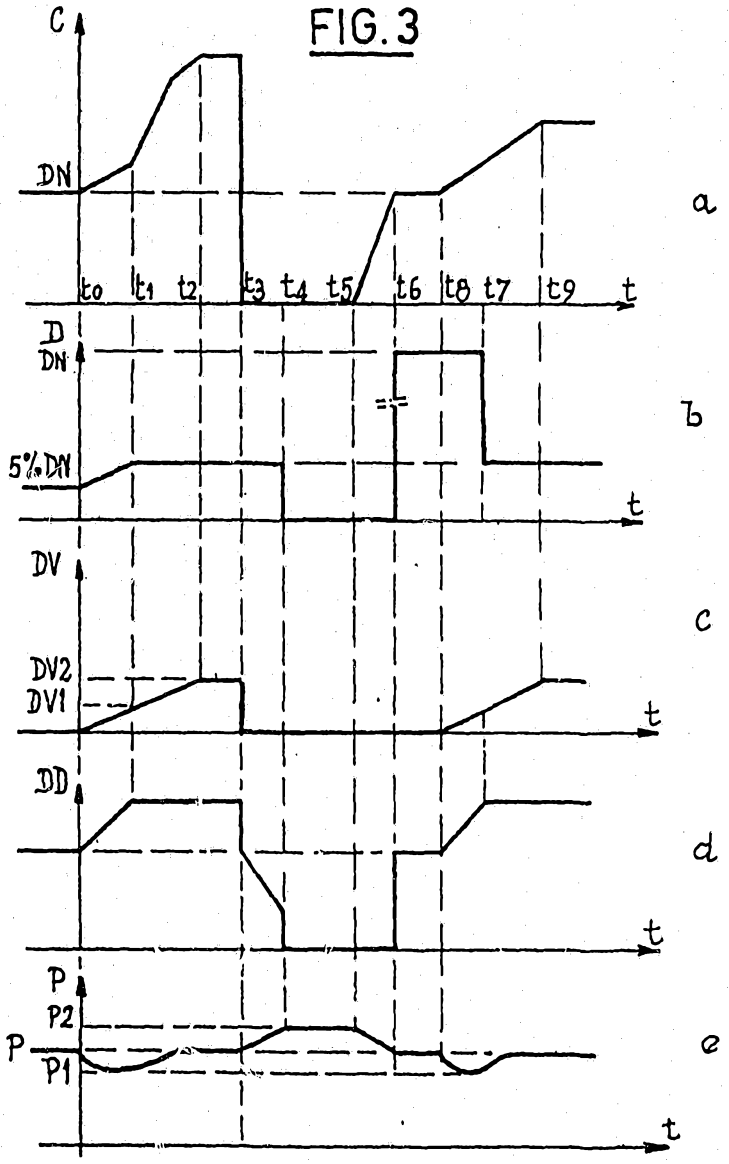


FIG. 3


C
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