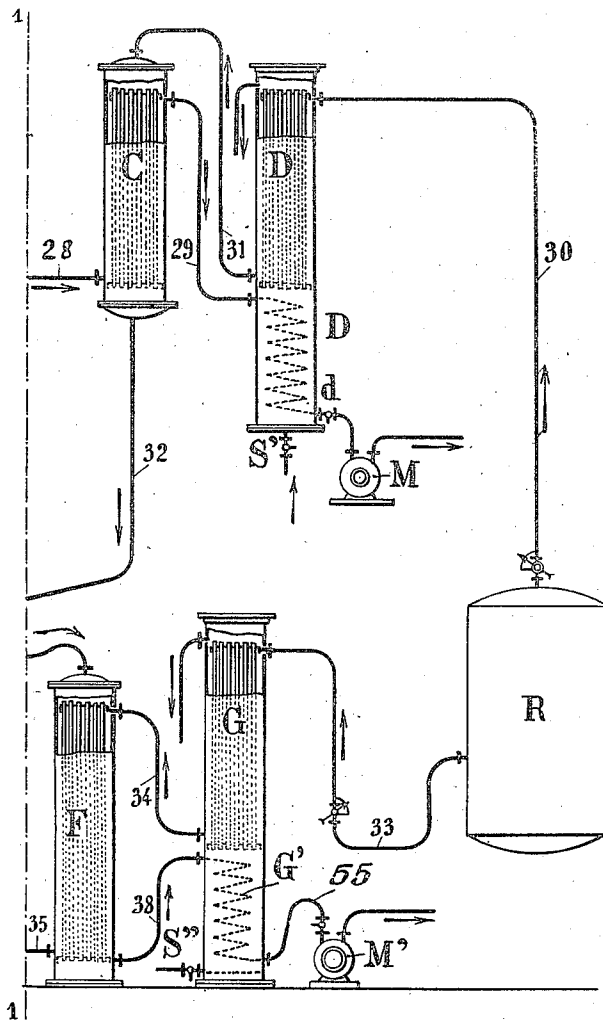


April 29, 1924.

1,492,063

E. A. BARBET
PROCESS AND APPARATUS FOR PRODUCING OXYGEN AND NITROGEN IN A
STATE OF PURITY FROM ATMOSPHERIC AIR
Filed Feb. 6, 1918 4 Sheets-Sheet 1

Fig. 1.



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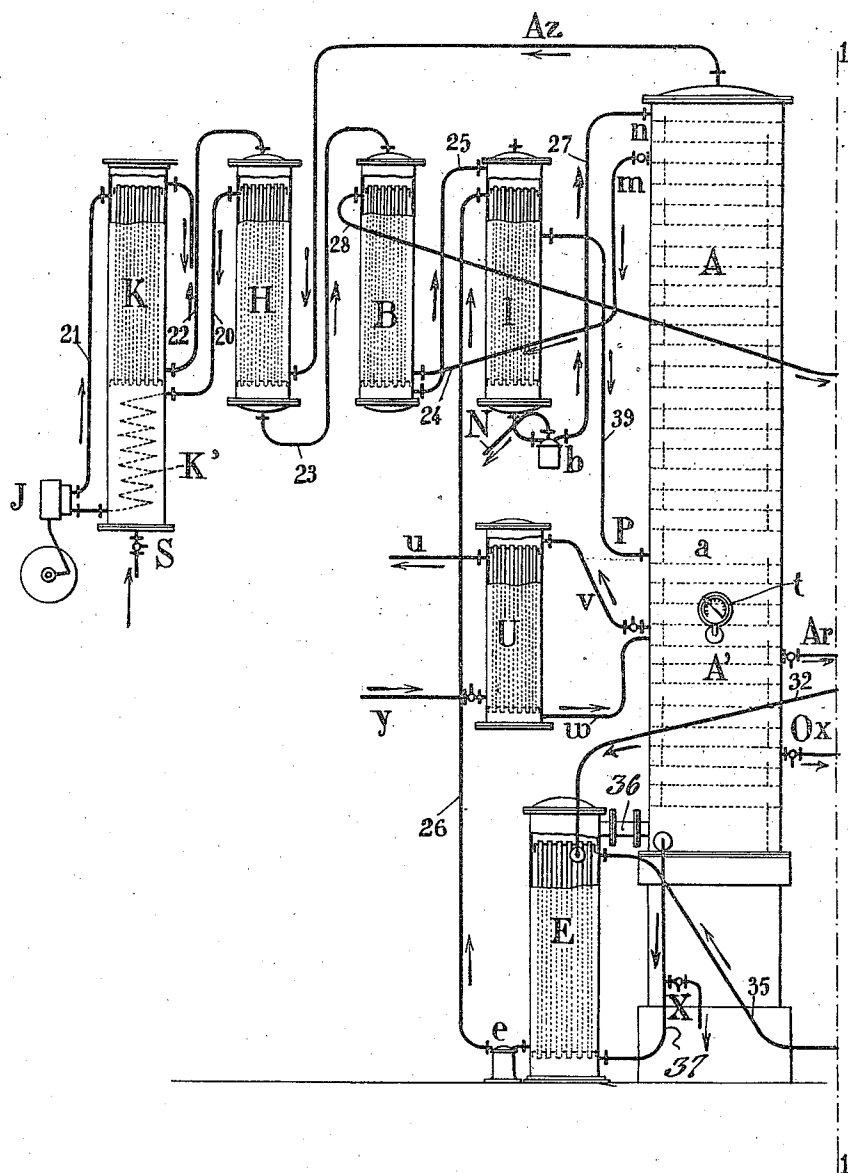
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Fig.1.



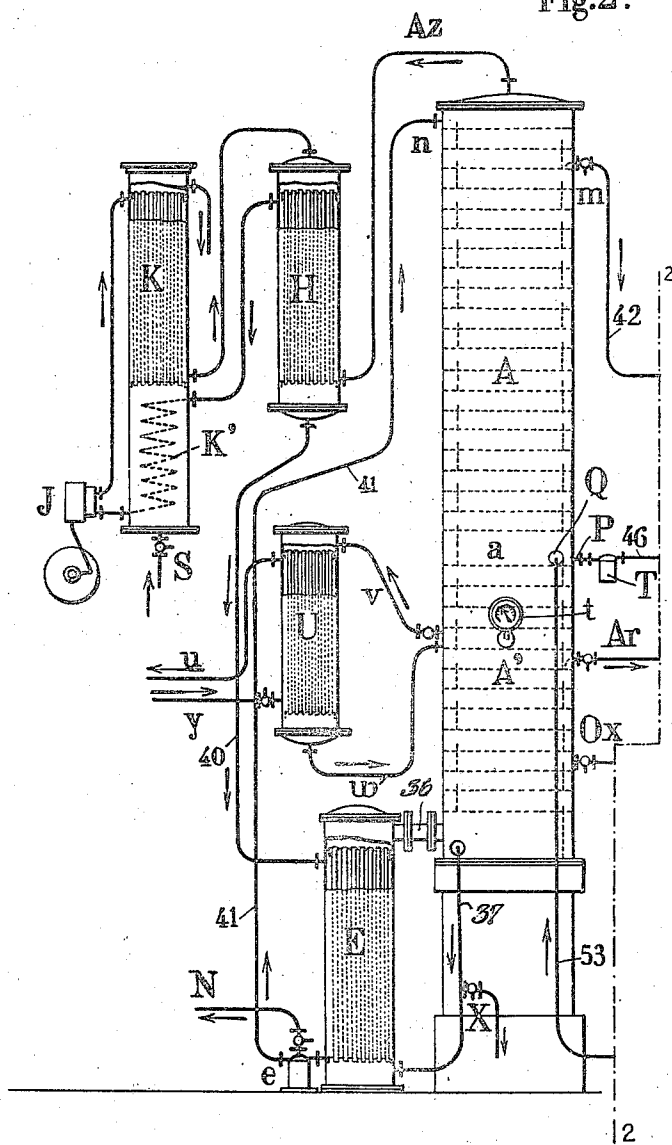
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Fig. 2.



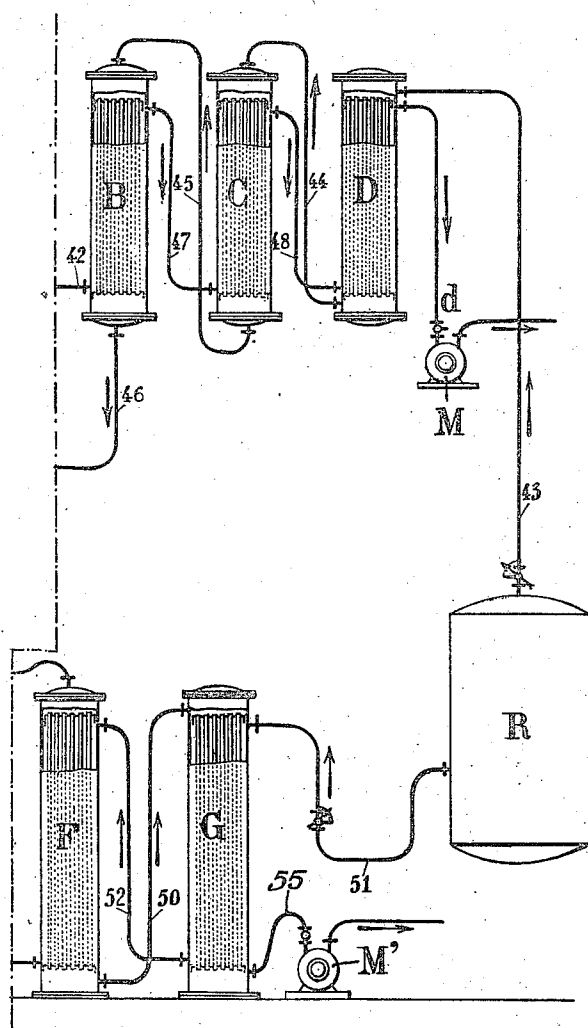
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Filed Feb. 6, 1918 4 Sheets-Sheet 4

Fig. 2.



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UNITED STATES PATENT OFFICE.

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PROCESS AND APPARATUS FOR PRODUCING OXYGEN AND NITROGEN IN A STATE OF PURITY FROM ATMOSPHERIC AIR.

Application filed February 6, 1918. Serial No. 215,679.

To all whom it may concern:

Be it known that I, EMILE AUGUSTIN BARBET, a citizen of the French Republic, residing at No. 5 Rue de l'Echelle, Paris, France, have invented certain new and useful Improvements in Processes and Apparatus for Producing Oxygen and Nitrogen in a State of Purity from Atmospheric Air, of which the following is a specification.

The present invention relates to the production of nitrogen and oxygen in a state of absolute purity and in a single continuous operation by the continuous rectification of liquid air.

The separation of the constituents of the liquid air is effected by a process analogous to that first applied in the continuous rectification of alcohols and demonstrated by Mr. E. Barbet in 1890, and in which the refining of the volatile product is effected almost entirely on the plates of the rectifying column by causing the vapours rising through the rectification column to bubble through a reflux of pure liquid returned to the top plates by the condenser. The sole action of the condenser is to take from the vapours a certain quantity of the pure product, the remainder of the condensed liquid being returned to the top plate to effect the washing of the ascending vapours. This reflux gradually and methodically forces all the less volatile constituents to the lower parts of the apparatus, permitting only the most volatile to pass over.

The heavy vapours, that is to say, vapours which are less volatile than the other vapours present are condensed in the liquid on the plates and evaporate in their stead a corresponding weight of more volatile vapour. If the vapours comprise vapours of a higher degree of volatility than those of the product desired to be obtained they will pass through the boiling liquid and not being condensed in the condenser are allowed to pass away.

In the accompanying drawing—

Figure 1 is a diagrammatic view of the device for carrying out the process of the present invention.

Figure 2 is a modification of this device.

In the operation of the apparatus, shown in Figure 1, air, previously freed from CO_2 and moisture by bubbling through NaOH solution and then through strong H_2SO_4 of $50-55^\circ$ Bé., or stronger, is supplied to the tank R, in which a pressure is maintained equal to about four atmospheres, by a suitable compressor (not shown). Air from the receptacle R passes through pipe 30, heat exchanger D, pipe 31, exchanger C, pipe 32 into the liquefaction apparatus E, from which the liquid air passes, by its own pressure, through pipe 26, into exchanger I in which it is partly re-evaporated, thence through pipe 39 into the middle *a* of the column AA' at the inlet P. Another part of the air from R passes through regulated pipe 33, exchanger G, pipe 34, exchanger F, pipe 35, into E where it mixes with the air entering the same through pipe 32.

The rectifying apparatus comprises a column having bubbling (splashing) plates AA' of any suitable type. Air is supplied at *a* as above stated. The rectification of the nitrogen is effected in the upper part A of the column; the rectification of the oxygen takes place in the lower part A' of the column.

A volume of cold gaseous nitrogen, approximately equal to twice that of the output of the apparatus, passes through the upper pipe A₂ of the rectifier, the volume being regulated by the speed of the pump J and the extent to which the valve in the connection *n* is opened. The liquefaction of this gaseous nitrogen which is to form the reflux liquid in column A is effected as follows:—The nitrogen is compressed to a pressure of two or three atmospheres by means of the pump J, and is refrigerated by cold gaseous nitrogen passing to the pump J by way of the exchanger H, pipe 20 and coil K'. The nitrogen passing through K' cools the water circulating in K several degrees, which water is then used to remove the heat of compression due to the pump. The compressed nitrogen passing from J through 21 loses its heat of compression in the cooling device K which is supplied with water through S. The compressed nitrogen then

passes through pipe 22 into the exchanger H where it is refrigerated by the nitrogen drawn in by the pump through pipe Az.

Thus we have compressed nitrogen the temperature of which is almost the same as that leaving the top of column A passing from the bottom of H by pipe 23. The nitrogen passes into the exchanger B where it comes in temperature-exchanging relation with liquid nitrogen withdrawn from column A at *m* through the pipe 24. The cooled, but not yet liquefied nitrogen passes through pipe 25 into the exchanger I in which it is liquefied by the low temperature due to liquid air supplied through the pipe 26, this liquid air being produced in another part (E) of the apparatus. The nitrogen which is liquefied in I passes through a float separator *b*, which gets rid of some of the non-liquefied traces of more volatile vapours, notably neon and helium. The liquid nitrogen rises by means of its pressure through pipe 27 and enters the top of column A at *n*.

The nitrogen reflux (i. e., that introduced in the liquid state at *n*) is not chemically pure since, owing to the rapidity of its condensation it has taken in addition to nitrogen, also neon and helium. The whole (except the portion of the neon and helium vented at N) is caused to flow back upon the head plate where this liquid is subjected anew to ebullition by the nitrogen vapours bubbling up there. In accordance with the laws of ebullition the most volatile elements are first converted back into vapour and after bubbling up on one or two plates, the reflux liquid is freed from such elements if an outlet for these is provided.

The nitrogen vapours rising from the lower plates contain very slight quantities of neon and helium, those vapours being more volatile than nitrogen, cannot be condensed and thus pass through the liquid nitrogen on the plate at *m* without rendering it impure, and it is merely necessary to draw off the desired proportion of the said liquid, (taking care not to draw off the vapour on the said plate), in order to obtain chemically pure nitrogen.

This extraction of pure liquid nitrogen is effected at *m*; it is this nitrogen which supplies the exchange element B through pipe 24. The weight of the portion so drawn off is about half that of the nitrogen which has been compressed at J. This is pure commercial nitrogen. This withdrawal of liquid nitrogen renders necessary the use of liquid air at I to complete the liquefaction of the nitrogen passing back into the top of the column through pipe 27. The other half of the liquid nitrogen continues to drop from plate to plate to wash the rising vapours in the rectification column.

The pure nitrogen is reconverted into gas in B. This very cold gas passes through pipe 28 into the bottom of exchanger C, then through pipe 29 into coil D' in the heat exchanger D which is surrounded by water. A measuring device M measures its volume and the cock *d* is manually regulated to permit of the discharge of the desired volume to the gasometer.

The cold of the pure gaseous nitrogen is used to provide a certain weight of very cold air which cooperates in supplying the rectifier. The air is drawn in a compressed state, from reservoir R through pipe 30 and passes into the exchanger D which is supplied with water through S', whereby the air is cooled, then the air passes through pipe 31 into exchanger C where it is cooled by being brought into heat-conducting relation with the gaseous nitrogen. From there it passes through pipe 32 into the tubular shell E to be liquefied by reason of its own pressure, as will be explained hereinafter.

It is possible to increase the relative power of the nitrogen reflux. It is only necessary to operate the pump J at greater speed. The same quantity of finished, pure nitrogen is continually withdrawn at *m*, but since a greater weight of reflux will be returned the volume of nitrogen passing from A will increase by exactly the same amount, and the operation will increase by exactly the same amount, and the operation will be self-regulating and accompanied with no inconvenience, since the liquefaction of larger amounts of nitrogen in I means revaporization of more of the liquid air therein, and hence the introduction of proportionately more revaporized air at P. This process permits of, as nearly as a perfect refining as the industry requires, since this degree of refining depends directly on the given proportion of the washing liquid, that is of the reflux liquid.

The liquid air dropping from plate to plate in A' becomes poorer in nitrogen and richer in oxygen. It is necessary to have a source of heat at the base of A' the function of which is to vaporize the pure oxygen which constitutes the residual liquid of the base of the rectifier, this is effected by the tubular member E.

The air which has been forced at a pressure of about 4 atmospheres into reservoir R is heated by the compression, and passes through pipe 33 into exchanger G where it is cooled by the cold water brought through S' exactly as is done for the nitrogen at K.

From G the compressed air passes through pipe 34 into the tubular exchanger F. While it is passing around the tubes in F gaseous oxygen, very cold (about 93° absolute) drawn from the bottom of A' at O_x, is caused to pass inside these same tubes.

The cooled air in F, still under pressure

finally passes through pipe 35 into the shell of E and mixes with the air delivered through 32, while in the tubes of the device E there is liquid oxygen of 93° absolute, which is the final reflux from the column A'. This apparatus is connected at its bottom to the column through pipe 37 and at its top through pipe 36. Owing to its pressure, the air, instead of liquefying at 83° absolute will be liquefiable at about 99° absolute; the liquid oxygen of 93° is thus itself able to condense it, by being itself revaporized. It is hence accordingly thus pure oxygen vapour which, on the one hand, provides the continuous withdrawal of gaseous oxygen at O_x and which, on the other hand, effects the boiling on all of the plates of the rectifier A'A.

The gaseous oxygen drawn off at O_x passing through the tubes of the exchanger F, pipe 38 and coil G', acts to refrigerate the compressed air coming from container R, as previously stated, and after passage through measuring device M' goes to the oxygen gasometer.

As to the liquefied air under pressure produced in E, (the prevention of the escape of gaseous air being prevented by the automatic float device *e*) which is to be fed to the feed plate *a*, advantage is taken of the pressure of the air to lift this liquid air through the pipe 26, into the exchanger I. It is this liquid air which has been previously referred to as the complementary agent for liquefying the nitrogen reflux.

Arriving at I the liquid air boils, a part of it being converted into vapour (richer in nitrogen than air), this being in proportion necessary to complete the liquefaction of the nitrogen. All of the excess remains in the liquid state. Then the liquid and the vapour both pass, through pipe 39 to the point P and to the feed plate *a*. The vapour portion unites with the vapours rich in nitrogen passing off upwardly from the plates A', and the two vapours rise together through the plates A to effect the rectification of the nitrogen.

It is clear that if an increased amount of nitrogen reflux is forced to the top of A a greater proportion of liquid air will consequently vaporize in I while the boiling produced at the bottom of A' by the heating element E remains constant. Hence there will be in A (as is absolutely necessary) a quantity of rectification vapour which will be increased by exactly the supplementary quantity of reflux liquid nitrogen to be evaporated. Operation is then carried on efficiently and automatically without requiring continuous regulation.

Notwithstanding heat insulation, it is not possible to prevent the entrance of a slight amount of the surrounding heat into the rectifier through the walls. If not guarded

against the entire device would become heated and no liquid would remain on the plates. Hence it is absolutely necessary to provide an excess of cold to maintain the operation at the low temperature desired.

For this purpose a tubular exchanger U is placed at the side of the rectifier through which tubular exchanger liquid air, coming from a compressor (not shown) is caused to circulate. The liquid air enters at *y* and the very cold vaporized air passes out through *u* to return to the liquefying compressor.

A certain regulated quantity of the vapours from the rectifier is drawn off at a stage in which there is already considerable richness in oxygen through the valved pipe *v*. These vapours being liquefiable at an absolute temperature higher than that of the liquid air because of their high content of oxygen, liquefy and pass in liquid state, through pipe *w*, to the same plate from which they were extracted. In order to measure the amount of cooling thus effected, a sensitive dial thermometer *t* is placed at the selected stage; if the rectifier is not sufficiently refrigerated from the liquid air in exchanger U, the thermometer *t* will rise; it is then necessary to increase the amount of liquid air passing through U. If the thermometer tends to drop the amount passing is decreased, by adjustment of the cock in pipe *y*.

Like nitrogen, oxygen contains its impurities. These are: argon which boils at -187° C.=86° absolute, that is at a temperature between those of oxygen and nitrogen, also xenon and krypton which are appreciably less volatile than oxygen.

It is evident that argon driven from the upper stages by the refluxes and forced towards the bottom of the apparatus is compelled to collect on an intermediate plate. It is sufficient to place a cock A_r, provided with an inner downwardly bent tube, for the purpose of drawing off the liquid but not the gas, at the level of maximum concentration of argon.

The cock is regulated in such a manner as to draw off an amount equal to about $\frac{1}{4}$ to $\frac{1}{3}$ of 1% of the entire amount of gas entering the apparatus so that no trace of argon will remain either in the oxygen or in the nitrogen.

As to the krypton and xenon, if the extraction of the gaseous oxygen is effected at O_x, (that is, several plates above the base of the column) these two impurities will accumulate at the base of the column and in the tubular element E. The quantity thereof is prevented from becoming an inconvenience by effecting a continuous slight withdrawal of liquid at X which consists of xenon krypton and liquid oxygen, on the pipe 37, about one fourth, one half or even

one per cent of the entire amount of air entering the apparatus according to the analysis of the oxygen. The three impurities, argon, krypton and xenon, are thus
 5 eliminated without the necessity of resorting to any special condensation or other operation.

The device is, of course, provided with manometers, electric or other thermometers,
 10 and with carefully selected heat-insulating jackets or lagging.

The general arrangement of the apparatus shown in Figure 1, for liquefying the nitrogen reflux is merely given by way of example.
 15

The apparatus shown in Figure 2 is slightly simplified. The compressor J gives a slightly greater pressure to the nitrogen than the device of Figure 1.

20 The increased volume of nitrogen issuing from the top of the column exchanges its cold units with the same nitrogen that is highly compressed by J, for instance to about 5 or 6 atmospheres. The compressed
 25 nitrogen which is cooled in H is still in the gaseous state.

Instead of liquefying the nitrogen in H (as in I in the arrangement shown in Figure 1) it is delivered by pipe 40 into the
 30 shell of vessel E wherein it is wholly liquefied. As this weight of nitrogen is greater than the weight of air which was delivered into E in Figure 1 the result is a greater gasification of oxygen and consequently a
 35 greater boiling power of the rectifying apparatus, and therefore a better purification.

The nitrogen wholly liquefied in E, passes through the separator *e* and rises thence by its pressure through pipe 41 up to *n* where
 40 it is delivered upon the top plate of the rectifier in order to constitute the desired copious nitrogen reflux. The liquid nitrogen is expanded in *e* and consequently there takes place likewise in *e*, the small automatic gasification which will allow of drawing off the
 45 light gases, namely, neon and helium at N.

The pure nitrogen will be still drawn off in the liquid state at *m*. Then, through
 50 pipe 42, it passes into the casing of exchanger B where it is gasified by abstraction of the heat from the air in the tubes thereof, whereby it will liquefy an almost equal amount of air. This air comes, under
 55 pressure, from reservoir R, through pipe 43, the tubes of the exchanger D, pipe 44, the tubes of the exchanger C and pipe 45, passes through the tubes of the exchanger B and through pipe 46 to the feed P of the rectifier, through separator T.

60 The nitrogen vaporized in the exchanger B continues its travel through pipe 47, shell of exchanger C, pipe 48, shell of exchanger D, pipe 49, then measuring device M, to the gasometer.

65 As regards the removal of the gaseous

oxygen at O₂, substantially the same apparatus is used as in Figure 1 except that the air which recuperates the cold units of the pure oxygen coming from O₂, has in
 70 this example only a very small pressure, and consequently requires no water cooling device. F and G are two exchangers. Pure oxygen issues from the meter M'. This part of the apparatus operates as follows:—

An amount of air equal to about one
 75 third of that passing through pipe 43, is fed through pipe 51, past the regulating valve therein, through the casing of the exchanger G, through pipe 52, casing of exchanger F, through pipe 53, into the
 80 rectifier at Q, while still in a gaseous condition. The gaseous cold oxygen from O₂ passes through the tubes of F, through pipe 50 through the tubes of G, through pipe 55, and out through the meter M' to the pure
 85 oxygen gasometer.

The result is a mixed feeding, namely, about $\frac{1}{3}$ of the feed enters at P in the form of liquid air, whilst the complement enters at Q in the form of air which is very cold
 90 but is still gaseous.

The arrangement will be considerably simplified, especially at the top of the apparatus, if it is not required to extract the rare
 95 gases. As regards the purity of the oxygen, the elimination of the argon, krypton and xenon is so simple, since it comprises neither exchanger nor cooler, that it is scarcely worth while to dispense with such
 100 elimination.

As hereinbefore stated, it is necessary to employ a fairly large number of temperature recuperators or exchangers, and these recuperators must be of high efficiency in
 105 order to transfer the cold of the issuing pure gases into the fresh air which is to enter the apparatus to be liquefied and rectified.

Having now described my invention what I claim as new and desire to secure by Letters Patent is:—

1. Apparatus for obtaining pure nitrogen and oxygen from atmospheric air, comprising a plate column, acting as a rectifier; means for supplying liquid air and
 115 gaseous air to said column at the central part thereof, a compressor adapted to compress the nitrogen passing out of the top of the column, means for cooling and liquefying said nitrogen a pipe adapted for
 120 bringing the liquefied nitrogen to the first plate of the column, a device located in connection with said pipe for extraction of the rare gases contained in the nitrogen, a withdrawal device on the rectifier for withdrawing a portion of the pure liquid nitrogen; heat exchange elements for cooling
 125 and liquefying compressed air under treatment by heat exchange with said liquid pure nitrogen, an outlet for the pure gaseous ni-
 130

trogen; a draw-off for liquid containing argon, located several plates below the supply level of the rectifier; heat exchange elements communicating with the base of the rectifier; a device on the connection between the rectifier and the last mentioned heat exchange elements, for withdrawing rare gases (xenon and krypton) contained in the liquid rich in oxygen at the base of the rectifier, means for bringing a gas hotter than the said liquid rich in oxygen into heat conducting relation therewith in said heat exchange elements, a draw-off for pure gaseous oxygen; heat exchange elements in which the oxygen passing out cools the compressed air, an outlet for the pure gaseous oxygen; means for withdrawing, cooling and returning vapors to the same stage of the rectifier, located between the main supply level and to level of withdrawal of argon material.

2. Apparatus for obtaining pure nitrogen and oxygen from atmospheric air comprising a plate column provided at near its central part with means for supplying liquid and gaseous air, a draw-off for impure nitrogen at the top; heat exchange elements and a compressor in which the nitrogen passing out the top of the column is compressed, cooled and partially liquefied by the cold nitrogen passing from the top of the column; means for further cooling and liquefying said nitrogen by liquid air produced in the apparatus; a pipe for returning the liquefied nitrogen to the first plate of the column; on this pipe a device for the extraction of the rare gases contained in the nitrogen; a draw-off located lower down on the rectifier for a part of the pure liquid nitrogen; heat exchange elements in which the liquid nitrogen cools compressed air; an outlet for the pure gaseous nitrogen; an extraction device for the liquid argon some distance below the main supply level of the rectifier; a heat exchange element communicating with the base of the rectifier which contains the liquid rich in oxygen; a device for the removal of xenon and krypton on the pipe connecting the base of the rectifier with said heat exchange element, connections for conducting compressed air cooled by the nitrogen to said exchange element; a pipe for bringing the liquid air produced to the exchange element in which the nitrogen is liquefied; connections for bringing this air partly in the liquid and partly in the gaseous state to the supply column; an extraction device for pure gaseous oxygen from the temperature exchange element in which the oxygen passing off cools the compressed air which unites in the exchange element at the bottom of the column with the compressed air cooled by the nitrogen; an outlet for the pure gaseous oxygen; a device between the main point of supply to

the rectifier and the point at which the argon is extracted for drawing off vapors and cooling the same by liquid air and for returning the condensed vapors to the same stage of the rectification column.

3. Apparatus for obtaining pure nitrogen and oxygen starting with atmospheric air comprising, a plate column with a feed of liquid and a feed of gaseous air both at the center thereof; temperature exchange elements and a compressor in which the nitrogen passing off from the top of the column is compressed, cooled and liquefied utilizing the cold units of the nitrogen issuing from the top of the column and of the oxygen of the bottom of the column; a pipe for returning the liquefied nitrogen to the first plate of the column; on this pipe a device for extracting the rare gases contained in the nitrogen; a draw off device on the rectifier for drawing off a portion of the pure liquid nitrogen; heat exchange elements for causing the nitrogen to cool and liquefy compressed air on its way to said column; an outlet for the pure gaseous nitrogen; draw off connection for argon located several plates below the point of supply of the column; heat exchange elements communicating with the base of the rectifier; an outlet for xenon and krypton on the communicating pipe; an outlet for pure gaseous oxygen; heat exchange elements in which the oxygen passing off cools the compressed air on its way to the column; a separate inlet for vapors on the rectifier between its point of supply and the point at which the argon is drawn off; a means for cooling these vapors by liquid air and a conduit for the return of the condensed vapors to the same stage.

4. An apparatus for obtaining pure oxygen and nitrogen from atmospheric air, comprising a pressure reservoir; connections therefrom through heat exchange elements and delivering into the middle of a vertically elongated rectifying column; a cyclic purifier and liquefier comprising a gas outlet from the top of said column, through a heat exchange system, to a compressor and back to the top of said column, a draw-off from said cyclic purifier, for withdrawing neon and the like; a second cyclic purifier and vaporizer comprising a liquid draw-off from the bottom of said column through a heat exchange device and back to the bottom of said column, and a draw-off from said purifier for withdrawing krypton and the like; a liquid nitrogen draw-off from the upper part of the column, but below the top thereof; a gaseous oxygen draw-off from the lower part of the column, but above the bottom thereof; a liquid argon draw-off above said gaseous oxygen draw-off, a cooling element in combination with said column near its middle; and heat exchangers for

transferring heat to material leaving the column from each of the materials about to enter said column, substantially as and for the purpose described.

5 5. An apparatus as covered in claim 4, in which the cooling element is connected to said column at a level below the air inlet but above the argon outlet.

6. A process of obtaining pure oxygen
10 and nitrogen from air which comprises introducing a mixture of liquid air and gaseous air at about the same temperature into the middle of a rectifying column, and allowing the liquid portion to fall therein
15 while the gaseous portion rises, withdrawing gaseous nitrogen from the top of the column, compressing and liquefying the same, withdrawing lighter and more volatile impurities from the nitrogen liquefying system,
20 reintroducing the remaining purified nitrogen at the top of the column, drawing off a portion of the further purified liquid nitrogen at a level somewhat below that at which such liquid nitrogen is returned to the column; drawing off a small amount of
25 liquid from the level of the column at which the liquid contains a maximum amount of argon, drawing off gaseous oxygen at a level below that at which the argon-containing liquid is withdrawn, drawing off liquid
30 oxygen containing krypton and xenon at the bottom of the column, evaporating the said liquid after withdrawing a sufficient amount to prevent material rise in the percentage of xenon and krypton in the column, reintroducing the remainder of the cold gaseous oxygen, xenon and krypton into the lower part of the column and cooling the column at a point intermediate its
35 height, sufficiently to correspond with the amount of heat taken up by the column from its environment.

7. The process of claim 6 in which the pressure of the liquefied nitrogen lifts the
40 same to the top of the column.

8. In a process for separating and recovering in a single continuous operation in a state of purity, substantially the whole of the nitrogen and oxygen contained in the
45 air which consists in subjecting the air

partly in liquid state and partly in the gaseous state near its point of liquefaction to a continuous rectification through the following steps, introducing the cold air at near the middle of a rectifying column containing a number of superposed plates,
55 withdrawing the nitrogen from the top of the column, freeing it from neon, hydrogen and helium, and returning the nitrogen to the top of the column, drawing off the purified nitrogen from about the third plate of the rectifier, and drawing off the argon from the plate where this gas and its maximum concentration, drawing off the oxygen containing xenon and krypton from near the
60 bottom of the column, withdrawing a portion of the oxygen, xenon and krypton to the column and extracting the pure oxygen at a point a few plates above the bottom of the column.

9. In the separation of air into its constituents in a column apparatus, the step of drawing off from the bottom of the column, a continuous stream of liquid rich in oxygen drawing off from said stream a sufficient fraction of the same to prevent any
75 substantial accumulation of xenon and krypton in the system, evaporating the residue of said stream by the latent heat of cold compressed air to be liquefied, and returning the re-vaporized oxygen to the column at a substantial distance below the point of draw-off of pure oxygen.

10. In the separation of air into its constituents in a column apparatus, the step of
85 drawing off from the bottom of the column, a continuous stream of liquid rich in oxygen drawing off from said stream a sufficient fraction of the same to prevent any substantial accumulation of xenon and krypton in the system, evaporating the rest of the oxygen so drawn off, in heat conducting relation with in-coming gas to be introduced into the system.

In testimony whereof I have signed my
95 name to this specification.

EMILE AUGUSTIN BARBET.

Witnesses:

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CHAS. P. PRESSLY.