March 29, 1960 E. KARWAT 2,930,201

PROCESS FOR THE PRODUCTION OF OXYGEN OF LOW ARGON CONTENT

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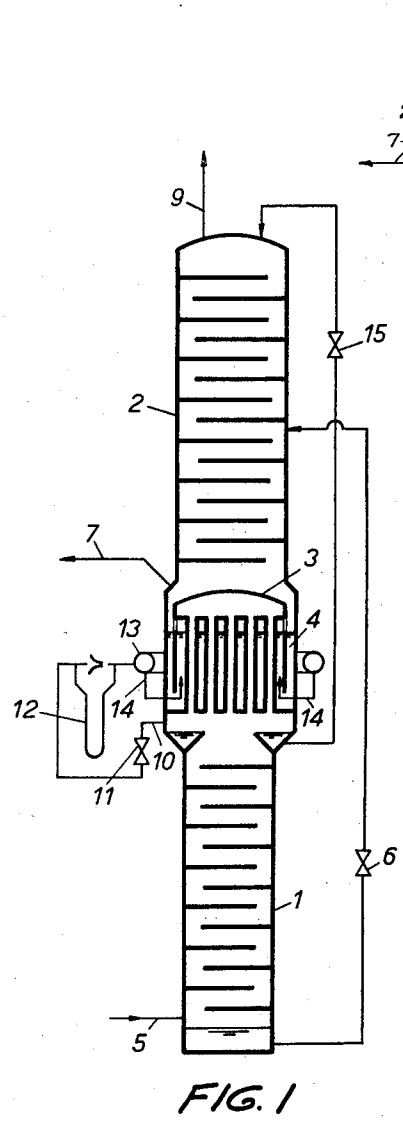


FIG. 1

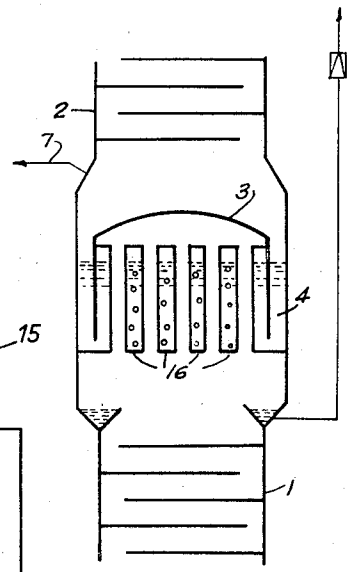


FIG. 2

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Filed Aug. 23, 1954 2 Sheets-Sheet 2

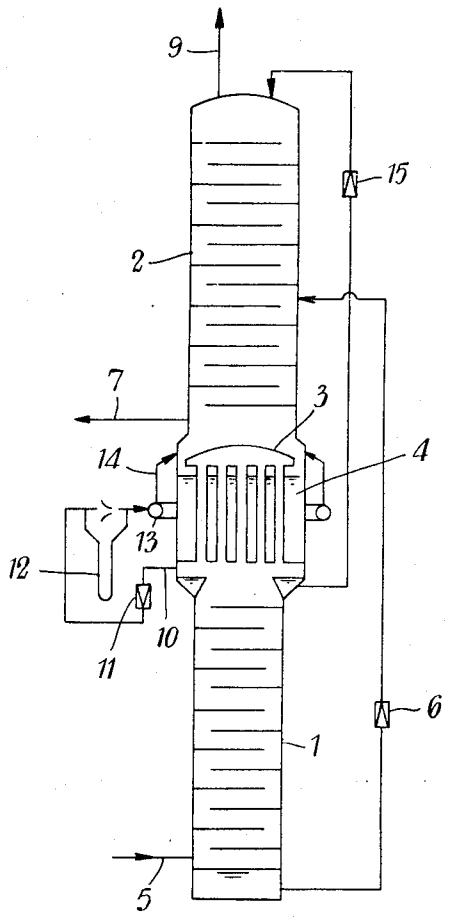


FIG. 3

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PROCESS FOR THE PRODUCTION OF OXYGEN OF LOW ARGON CONTENT

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10 Claims. (Cl. 62—22)

The present invention relates to a process and an arrangement for the production of oxygen of low argon content, the gas residue of which consists preponderantly of nitrogen.

In contrast thereto, in the oxygen produced by the usual process of air separation, the gas residue consists very preponderantly of argon with substantially only small admixtures of nitrogen. With oxygen having a degree of purity of 95% or more, the gas residue contains from 90% to 95% of argon and only a few percent of nitrogen.

If oxygen less than 95% pure is produced, the gas residue contains a larger quantity of nitrogen in proportion as the oxygen is less pure, but the gas residue then always contains, in addition to the nitrogen, substantially all the argon which has been supplied to the separator in the air to be separated. The reason for these phenomena is that the vapour-pressure of argon is closer to that of oxygen than to that of nitrogen. If the concentration by rectification of the oxygen and the nitrogen in the air, a cloud of argon enters between the rectification zones and prevents access of the nitrogen to the pure oxygen in the evaporator of a rectification column.

The present invention has for one of its objects so to influence the course of the rectification of the air treated that substantially only small admixtures of argon remain in the gas residue of the oxygen produced, said residue consisting preponderantly of nitrogen.

According to the present invention, there is provided a process for the production of oxygen of low argon content, the gas residue of which consists preponderantly of nitrogen, comprising the steps of feeding liquid air to a separating column, rectifying the liquid air in said column, separating a fraction rich in oxygen and low in nitrogen from said liquid air and feeding nitrogen to the separating column in the zone where the last stages of concentration of the said fraction take place, thereby reducing the argon content thereof.

By "zone of the last stages of concentration of the oxygen" is meant the oxygen evaporator at the base of the separating column, the gas chamber situated above it and the first plates arranged above the gas chamber in the rectification column.

It is preferable to introduce the nitrogen in the most finely divided form possible into the oxygen boiling in the evaporator of the separating column. The whirling of the liquid oxygen, its vapours and the nitrogen fed thereto can be effected more simply and more thoroughly therein at other points of the separating column.

However, the nitrogen may also be fed to the gaseous oxygen above the liquid bath or, if the nitrogen is admixed either with the gas phase or with the liquid phase on one of the plates lying above the evaporator. The latter method is applicable primarily when the oxygen produced is not taken from the vapours above the liquid bath, but is taken in the liquid state from the evaporator in a manner known per se and if desired converted into gaseous form in a supplementary evaporator.

Preferably, the nitrogen is fed to the separating column in gaseous form, because this is the simplest method of handling it. If the nitrogen is introduced into the separating column in liquid form, on the other hand, the advantage is obtained that the necessary temperature drop occurring in the oxygen bath with the replacement of the argon in the oxygen by nitrogen is promoted, since the heat of evaporation of the liquid nitrogen is promoted, since the heat of evaporation of the liquid nitrogen is liberated in the presence of oxygen.

In the most usual two-stage rectification, the nitrogen is preferably taken from the pressure column. In the case of single-stage rectification, the nitrogen is fed from the nitrogen circuit into the separating column, and, in some cases, nitrogen at normal pressure is compressed to the pressure of the separating column.

The quantity of nitrogen blown in is a multiple of the sum of the quantities of argon and nitrogen leaving the separating column, together with the oxygen produced, and is preferably from 1 to 4 times the said sum. This rule applies to oxygen of not less than 95% purity. In the case of oxygen of even lower purity, fractions of the quantities determined are sufficient to produce the required effect.

The effect of the introduction of nitrogen at the aforesaid points of the separating column is that the stream of nitrogen rising from the evaporator forces the argon away from the lower plates of the column and brings it to the head of the column.

For a better understanding of the invention and to show how the same is to be carried into effect, reference will now be made to the accompanying drawings in which

Figure 1 illustrates diagrammatically a rectification arrangement employed for practicing one embodiment of the invention;

Figure 2 is a diagrammatic elevation of a part of the rectifying column of Figure 1; and

Figure 3 illustrates a modification of Figure 1.

Situated above a pressure column, which is at a pressure of 0.5 atmospheres gauge. A condenser-evaporator 3 is situated between the two aforesaid columns, the nitrogen rising in the column 1 being condensed on the pressure side of the said condenser 7, low-pressure side thereof. 500 Nm.^{3} (Nm.^{3} indicates volume in cubic meters at 0° centigrade and at 760 mm. pressure) of low-cooled air are fed per hour to the pressure column 1 at 5 and are separated therein in a manner known per se into pressure-column liquid of high oxygen content and liquid nitrogen. The pressure-column liquid flows through an expansion valve 6 to a low-pressure column 2, the nitrogen condensed in 3 being fed through an expansion valve 15 to the top of the low-pressure column as reflux liquid. 100 Nm.^{3} of oxygen of 98% purity per hour leave at 7, and 400 Nm.^{3} of nitrogen per hour leave at 9.

According to the normal method of operation, there are 1.85 Nm.^{3} of argon and 0.15 Nm.^{3} of nitrogen in 100 Nm.^{3} of oxygen, and 2.80 Nm.^{3} of argon and 6.45 Nm.^{3} of oxygen in 400 Nm.^{3} of nitrogen.

According to the present modification, about 4 Nm.^{3} of nitrogen per hour are taken from the pressure column at 10, that is to say, double the amount of gas residue of 100 Nm.^{3} of oxygen, expanded in a valve 11 to the pressure of the upper column, measured with a gasometer 12 and introduced into a nitrogen ring conduit, which surrounds the lower end of the column 2 and from which a number of tapping pipes 14 disposed at regular intervals around the periphery of the column either lead as shown into the oxygen boiling in the condenser-evaporator 3, or open into the vapour space situated above the said condenser-evaporator 3 or open onto one of the plates of the rectification column which are situated thereabove, for example, the liquid pockets of the plates. The oxygen then taken from 7 has a purity of 98%

and contains 0.10% of argon and 1.90% of nitrogen. The quantity of oxygen produced is reduced to 97 Nm.³/h. 403 Nm.³ of nitrogen fraction leaving at 9 contain 9.4 Nm.³ of argon.

If it were desired to produce oxygen containing not more than 0.10 Nm.³ of argon to 100 Nm.³ of oxygen by the method hitherto usual, its purity would have to be adjusted to 99.87% oxygen, and the quantity of oxygen produced per hour would fall to about 72 Nm.³.

In conjunction with the displacement of the argon from the bottom plates of the column, the increase in the oxygen concentration on the rectification plates from the top downwards is somewhat impeded. If no special step were taken, somewhat more oxygen would thus also leave the column together with the nitrogen, as is shown by the figures of the foregoing example. This loss can be offset by correspondingly increasing the number of rectification plates, 3 to 4 plates being sufficient for this purpose in the case of the foregoing example.

In another constructional form (not shown) of a rectification arrangement, the tubes of the nitrogen condenser of the pressure column are provided with a number of small holes sufficient for the passage of the necessary quantity of nitrogen.

The process described in the foregoing example with reference to the diagram of a two-stage rectification, may naturally also be carried out with single-stage separating columns. The only essential requirement is that nitrogen should be additionally fed to the separating column in the zone of the last stages of concentration of the oxygen.

I claim:

1. A process for the production of oxygen having a gas residue consisting mainly of nitrogen with only a small admixture of argon, comprising the steps of feeding liquid air to a rectifying column, separating in a first portion of said column a fraction which is rich in oxygen and low in nitrogen, separating in a second portion of said column a fraction consisting of nitrogen, feeding a portion of said separated nitrogen to the zone of said first portion in which the last stages of concentration of said oxygen rich fraction takes place and removing from said zone said oxygen rich fraction which is low in nitrogen content and lower still in argon content.

2. A process according to claim 1, wherein said portion of said separated nitrogen is introduced into the oxygen fraction boiling at the base of said first portion.

3. A process according to claim 1, wherein said portion of said separated nitrogen is fed to the gaseous oxygen immediately above the boiling oxygen fraction at the base of said first portion.

4. A process as claimed in claim 1, wherein said portion of said separated nitrogen is fed to said first portion in gaseous form.

5. A process as claimed in claim 1, wherein said portion of said separated nitrogen is fed to said first portion in liquid form.

6. A rectifying column for the production of oxygen having a gas residue consisting mainly of nitrogen with only small admixture of argon, comprising a high-pressure column, a low-pressure column supported on said high-pressure column, a condenser-evaporator for boiling oxygen disposed within the base of said low-pressure column, a ring-conduit for nitrogen disposed around the base of said low-pressure column and pipes leading from said ring-conduit into a portion of the low-pressure column adjacent the condenser-evaporator.

7. Apparatus for the production of oxygen with a low argon content, comprising a high-pressure air-separating column, a low-pressure rectifying column supported vertically above said high-pressure column, conduit means connecting the base of the high-pressure column to about the middle of said low-pressure column for supplying a liquid rich in oxygen to the latter column, further conduit means connecting the top of the high-pressure column to the top of the low-pressure column for supplying a liquid rich in nitrogen to the latter column, a condenser-evaporator disposed within the base of said low-pressure column and separating the boiling liquid at the foot of the low-pressure column from the condensing vapours at the top of the high-pressure column, a collecting trough situated at about the top of the high-pressure column for collecting part of the liquid that is rich in nitrogen, and a pipe connection between said trough and the base of the evaporator part of said condenser-evaporator for supplying the gaseous nitrogen to the boiling liquid in said condenser-evaporator.

8. Apparatus for the production of oxygen of low argon content, comprising a high-pressure column for fractionating liquid air supplied to the base of said column, a low-pressure rectifying column vertically supported above said high-pressure column, a condenser-evaporator located within the base of the low-pressure column, evaporator tubes in said condenser-evaporator for receiving boiling liquid that is rich in oxygen in the foot of the low-pressure column, condenser tubes in said condenser-evaporator for receiving from the top of the high-pressure column vapours that condense in heat-exchange with said boiling liquid, a cooling dome for said condenser-evaporator separating the condenser tubes from the boiling vapours on the top of the dome, conduit means connected between the base of the high-pressure column and about the middle of the low-pressure column, further conduit means connecting the top of the high-pressure column to the top of the low-pressure column, outlets for gaseous nitrogen and oxygen at the top and bottom of the low-pressure column respectively, a collecting trough for a liquid that is rich in nitrogen located immediately below the condenser tubes at the top of the high-pressure column, and a pipe-connection between said collecting trough and the top of said evaporator tubes in said condenser-evaporator for feeding gaseous nitrogen to the vapour space above the boiling liquid in said evaporator tubes.

9. A process for the production of an oxygen fraction having a gas residue consisting mainly of nitrogen with only small admixtures of argon by the use of a 2-stage air separation apparatus including a rectifying column and a pressure column, comprising the steps of feeding liquid air to the rectifying column of the two-stage air separation apparatus, separating from said liquid air in the lower part of said column a fraction rich in oxygen, low in nitrogen and very much lower in argon, and feeding nitrogen in gaseous form from the pressure column of said apparatus to the zone in said lower part of said column in which the last stages of concentration of said fraction take place.

10. A process for the production of oxygen having a gas residue consisting mainly of nitrogen with only a small admixture of argon, comprising the steps of feeding liquid air to a rectifying column, separating in a first portion of said column a fraction which is rich in oxygen and low in nitrogen, feeding nitrogen to the zone of said first portion in which the last stages of concentration of said oxygen rich fraction takes place, removing from said zone said oxygen rich fraction which is low in nitrogen content and lower still in argon content and separating and removing in a second portion of said column the entire residual fraction consisting of nitrogen and residual argon.

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