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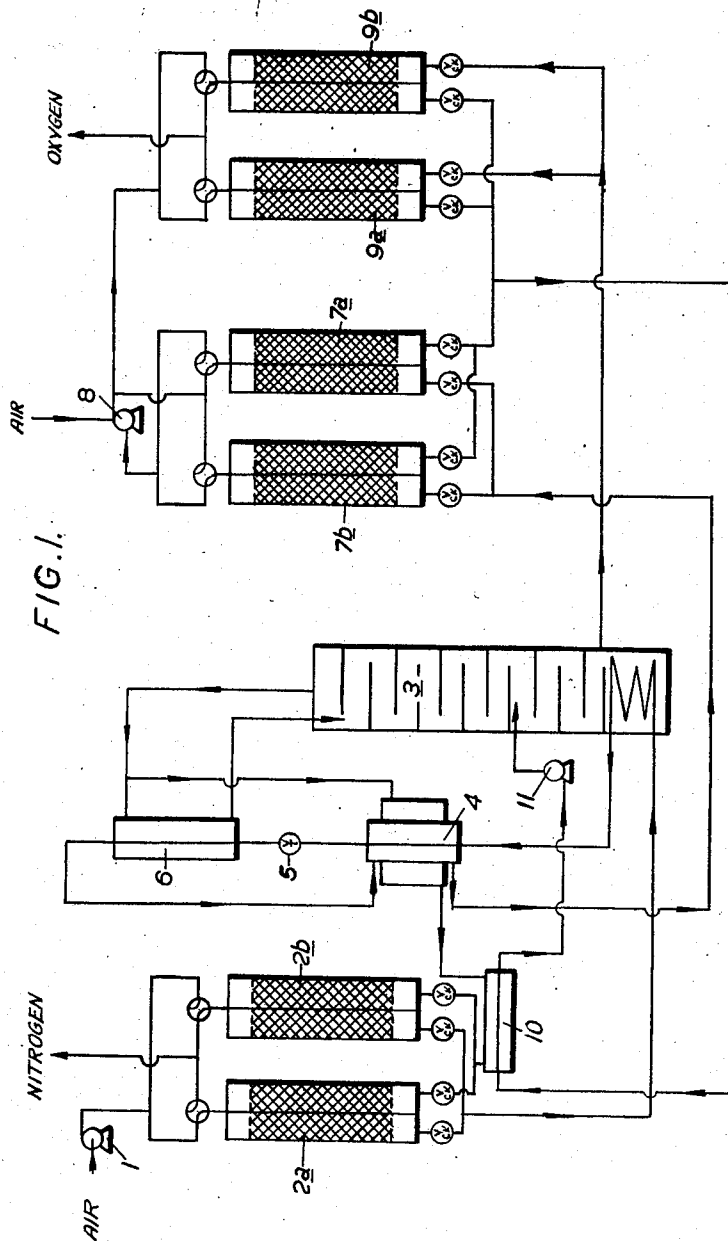
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PROCESS FOR THE SEPARATION OF GAS MIXTURES

Filed Aug. 15, 1951

4 Sheets-Sheet 1



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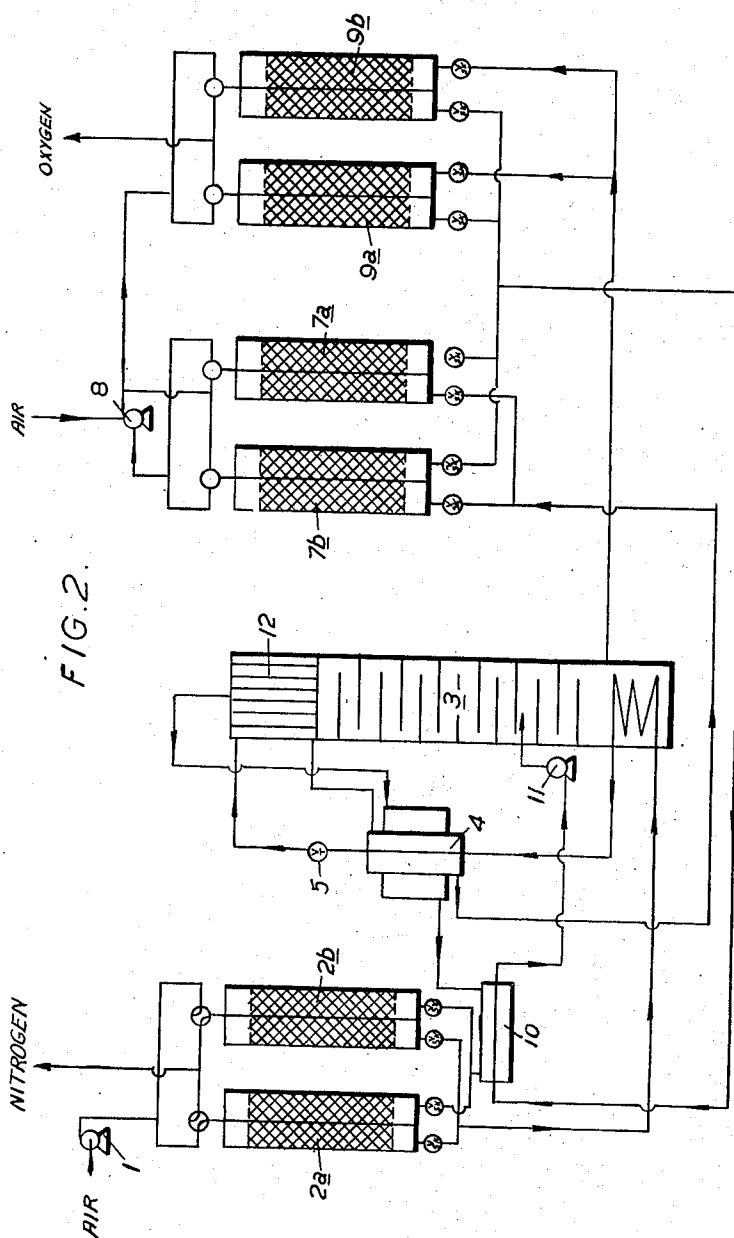
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4 Sheets-Sheet 2



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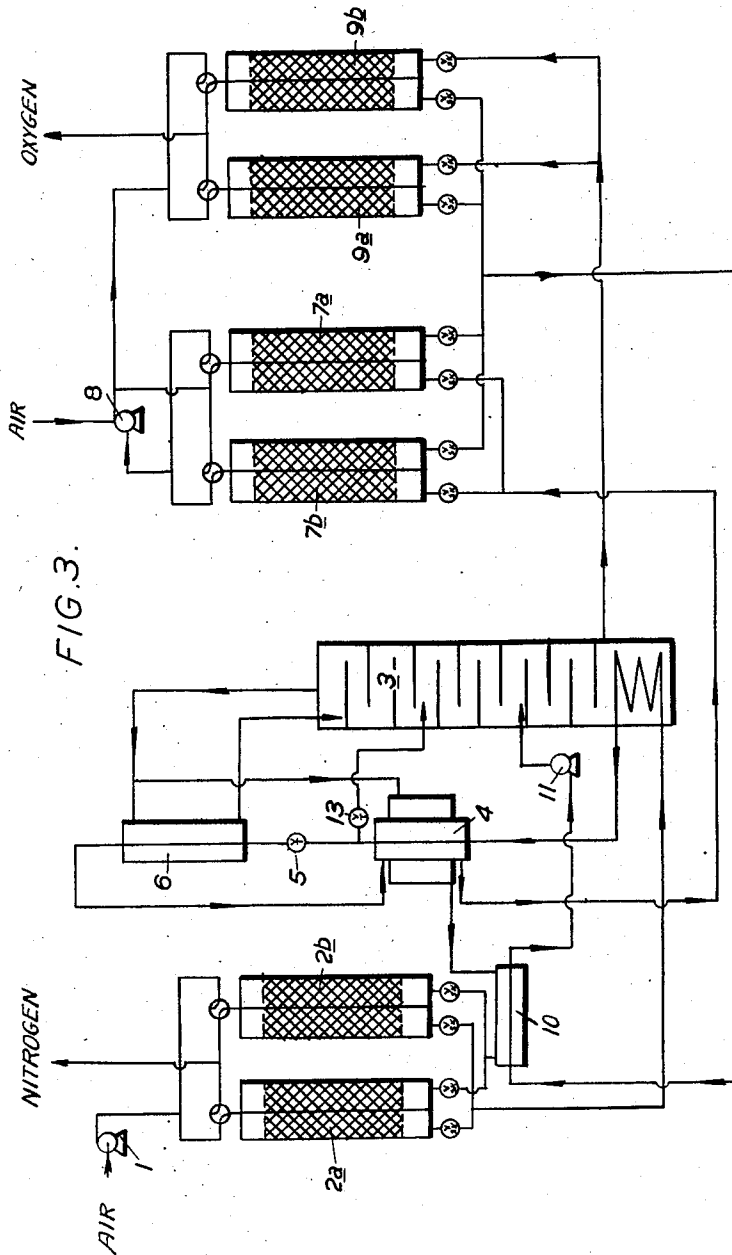
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PROCESS FOR THE SEPARATION OF GAS MIXTURES

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4 Sheets-Sheet 3



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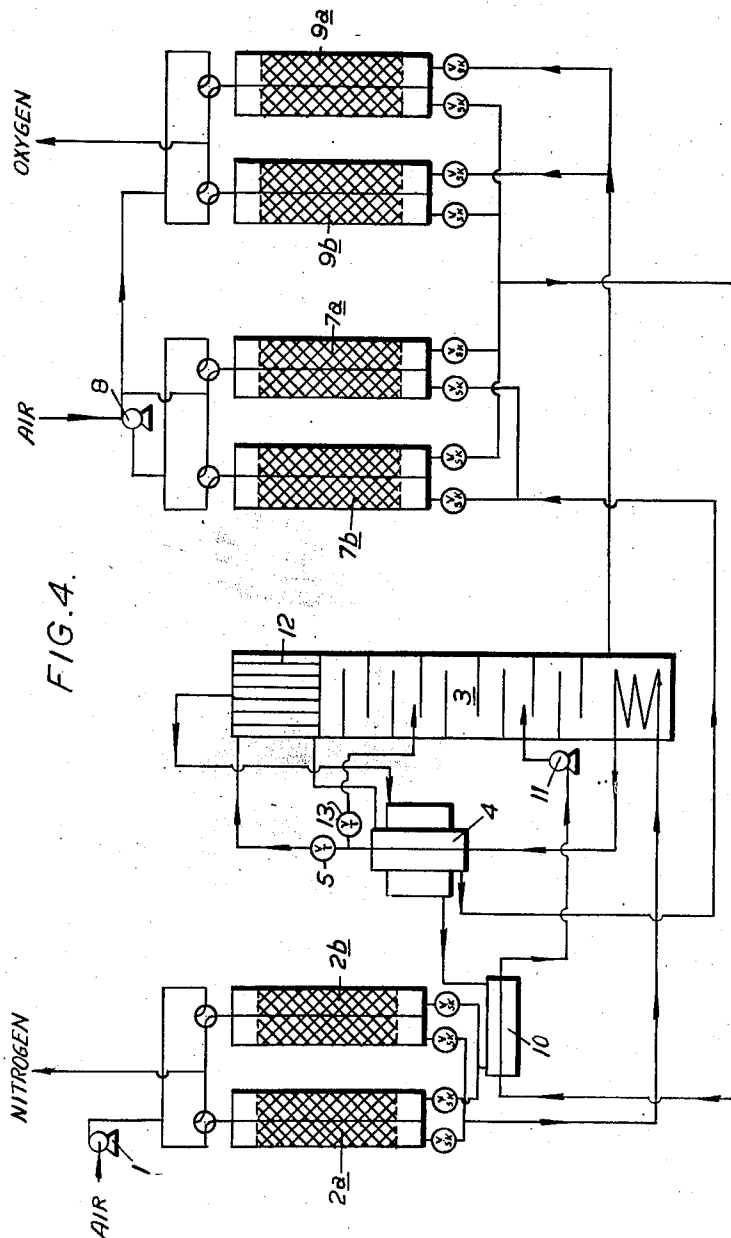
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PROCESS FOR THE SEPARATION OF GAS MIXTURES

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4 Sheets-Sheet 4



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PROCESS FOR THE SEPARATION OF
GAS MIXTURES

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11 Claims. (Cl. 62—175.5)

1

The present invention relates to the separation of gas mixtures, and more particularly to those gas mixtures which are capable of giving a homogeneous liquid phase on cooling and of being separated into two or more fractions of different boiling points. Typical of such a gaseous mixture is air, and to facilitate an appreciation of the invention herein to be described, particular reference will be made to air separation.

Previous methods of producing both oxygen and nitrogen in a substantially pure state fall into two classes; the use of the well-known double column, and methods using an auxiliary nitrogen circuit, with recompression of the nitrogen. In both cases however the nitrogen is liquefied by indirect heat-exchange with oxygen boiling at the base of a column operating at atmospheric pressure. In the first case, the nitrogen is produced by partial rectification of the air in a column or similar apparatus operating at an elevated pressure, and, after liquefaction in the manner stated above, the liquid nitrogen is expanded to atmospheric pressure for use as reflux in the main column. In the second case, nitrogen may be produced in an auxiliary apparatus, for example an "enricher" as described in British Patent No. 632,329 or may be withdrawn from the top of the main column at atmospheric pressure in gaseous form. It is then compressed to an elevated pressure such that it may be liquefied by heat exchange with the oxygen in the base of the main column as hereinbefore described.

It is an object of the present invention to provide a method for the separation of a gas mixture into a lower boiling point fraction and at least one higher boiling point fraction which uses a single rectification column and in which the lower boiling point fraction is at no time compressed to a higher pressure than that of the column.

There is thus provided by the present invention a method of separating a gas mixture into a lower boiling point fraction and at least one fraction of higher boiling point, comprising the steps of compressing the gas mixture, cooling the compressed gas mixture to a saturated vapour by heat exchange with at least part of the fraction produced, condensing at least part of this vapour by heat exchange with the boiling highest-boiling fraction, using at least part of this condensed mixture for liquefying vapour to provide reflux medium, recompressing the evaporated mixture to at least atmospheric pressure, cooling and expanding this recompressed gas mixture to saturation point, separating a stream of the gas mixture into the aforementioned fractions by

2

countercurrent contact in a rectification device with the reflux medium and recovering from all cold vapours the cold used for cooling the incoming gas stream.

The reflux medium for the rectification device may be provided by a part of the separated lower boiling fraction, liquefied by indirect heat exchange with the condensed gaseous mixture. Alternatively, the upper portion of the rectification column is formed as an enricher wherein the vapours rich in the lower boiling point constituent are condensed to form the reflux medium by indirect heat exchange with the condensed gas mixture.

This method makes it possible to use already known methods of heat exchange and of purification of the incoming gas mixture from condensable impurities.

The extra cold necessary to balance losses by heat leak, and other well-known losses, may also be obtained by any conventional means, for example, by expansion of gas in a turbine or other expansion engine.

The rectification device may conveniently operate at substantially atmospheric pressure (in which case the liquid/gas mixture evaporates under reduced pressure) or at an elevated pressure such that the liquid/gas-mixture evaporates at substantially atmospheric pressure.

The invention as applied to the separation of air will now be described in more detail with reference to Figures 1 to 4 of the accompanying drawings which show diagrammatically four methods of carrying the invention into effect. Like parts in all figures bear the same reference numeral. The direction of flow along the interconnecting pipework is everywhere shown by arrows.

In all the figures, regenerators are shown for cooling the compressed air. In all figures a pair of associated regenerators bear the same reference numeral and the members of each pair are marked respectively by the suffix *a* or *b*, the suffix *a* being applied to the cooling regenerator and the suffix *b* to the regenerator which is in course of being cooled.

To avoid undue elaboration of the description several features for carrying the process into effect which are well known per se to those skilled in the art have been omitted from the drawings. It will be appreciated, for example, that in practice there will have to be provision for removing residual traces of moisture and carbon dioxide from the cooled mixture emerging from the regenerators; that it is usually necessary to filter

condensed gases before expansion; that vapours must be superheated before isentropic expansion; that the gas flows through a pair of associated regenerators must be balanced to ensure the attainment of the required thermal conditions and the complete removal of condensates; also that a change-over valve mechanism has to be provided for sets of associated regenerators. All such features are well known and have been omitted in the interests of simplicity.

Referring to Fig. 1, air is compressed in a turbo-compressor 1 to approximately 70 p. s. i. a. and cooled to saturation point by counter-current heat exchange with the separated nitrogen fraction in one pair of regenerators 2a, 2b (the suffix "a" being applied to the cooling regenerator and the suffix "b" to that being cooled). It is then completely condensed by indirect heat exchange with the oxygen boiling at the base of the separation column 3. The liquid air is then sub-cooled in sub-cooler 4 by heat exchange with nitrogen from the top of the column 3 and with low-pressure evaporated air produced as hereafter described. The sub-cooled liquid is expanded through valve 5 to approximately 7 p. s. i. a. and evaporated by indirect heat exchange in exchanger 6 with part of the nitrogen from the top of the column, condensing this nitrogen which is returned to the column as reflux. The evaporated air is warmed slightly in sub-cooling the liquid air sub-cooler 4 and is then warmed to substantially atmospheric temperature by heat exchange with incoming air in a second pair of regenerators 1a, 1b. The low-pressure air is recompressed to approximately 35 p. s. i. a. in two stages in turbo-compressor 8, more air at atmospheric pressure being added after the first stage and compressed within the second stage. Part of this intermediate pressure air is then cooled counter-current to the low pressure air in regenerators 1a, 1b; the remainder is cooled counter-current to the oxygen stream from the base of the column in regenerators 9a, 9b. All the intermediate pressure air is then further cooled by heat exchange with the nitrogen stream from sub-cooler 4 in exchanger 10. This step does not eliminate the necessity for super-heating the air before it enters the turbine. Alternatively, the nitrogen stream may be warmed by cooling an air stream used for balancing the regenerators (not shown in the figure). This nitrogen stream then passes to the regenerators 2a, 2b. The intermediate pressure air then passes through expansion turbine 11, being thus cooled to saturation point and issuing from the turbine 11 at the column pressure; it then enters column 3 at the appropriate point as a gaseous feed.

Condensable impurities in the incoming air are deposited in both pairs of regenerators 2a, 2b and 1a, 1b and subsequently re-evaporated into the out-going streams. It has been found that in view of the difference in actual volumes between the incoming air stream and the outgoing air stream passing through regenerators 1a, 1b, substantially the whole of the condensates in the regenerators will have been revaporised by the outgoing air stream well before the end of the cycle. The cold low-pressure air leaving the regenerator during the concluding part of the cycle will in consequence be substantially free from revaporised condensates and advantage can be taken of this phenomenon to effect removal of cold from the oxygen stream. Since the tail portion contains no condensable impurities, any convenient type of exchanger can be used, without

danger of blocking the exchanger by the accumulation of deposits or of contaminating the oxygen.

The arrangement shown in Figure 2 is substantially similar to that in Figure 1, with the exception that the exchanger 6 is replaced by an enricher 12 at the top of the column 3. The liquid air after expansion through valve 5 to a pressure of approximately 12 p. s. i. a. is passed through the enricher 12 where it acts to condense vapours rich in nitrogen to form the reflux for the column 3.

Figure 3 shows an arrangement similar to that shown in Figure 1, except that in this case only a part of the sub-cooled liquid air is expanded through valve 5 to the exchanger 6, the remainder being expanded through valve 13 to substantially atmospheric pressure and introduced into the column 3 as liquid feed.

Figure 4 shows an arrangement providing for a liquid feed to column 3 as in the arrangement shown in Figure 3, combined with the use of an enricher 12 as previously described with reference to Figure 2.

In any of the cycles shown in the figures, the second pair of regenerators 8a, 8b may be eliminated, and the low-pressure air passed direct to a jet-pump after evaporation. The driving fluid for this pump is obtained as side-stream from the first pair of regenerators 2a, 2b in known manner. This side stream passes through an expansion turbine, being thus cooled to saturation point, and issues at the pressure necessary to operate the jet pump. The delivery of the pump is at substantially column temperature and is introduced into the column as gaseous feed.

It will be understood that the oxygen fraction produced by any of the methods above described, may, if desired, be further separated to produce an argon-enriched fraction, substantially pure oxygen, and/or a fraction enriched in krypton and xenon.

I claim:

1. A method of separating a gas mixture into a lower boiling point fraction and at least one fraction of higher boiling point which comprises the steps of compressing the gas mixture, cooling the compressed gas mixture to form a saturated vapour by heat exchange with at least part of the fractions produced, condensing at least part of this vapour by heat exchange with the boiling highest-boiling fraction, expanding this condensed mixture and using at least part thereof for liquefying vapour to provide reflux medium, recompressing the evaporated mixture to at least atmospheric pressure, cooling and expanding this recompressed gas mixture to saturation point, separating a stream of the gas mixture into the aforementioned fractions by counter-current contact in a rectification device with the said reflux medium, and recovering from all cold vapours the cold used for cooling the incoming stream of gas mixture.

2. A method according to claim 1 wherein the reflux medium for the rectification device is provided by a part of the separated lower boiling point fraction liquefied by indirect heat exchange with the condensed gaseous mixture.

3. A method according to claim 1 wherein the rectification device comprises a rectification column surmounted by an enricher wherein the vapours rich in the lower boiling point constituent are condensed to serve as reflux medium in the column by indirect heat exchange with the condensed gaseous mixture.

4. A method according to claim 1 wherein only

5

a part of the condensed mixture is used for liquefying the lower boiling point fraction, the remainder after expansion to the column pressure being fed to the rectification column at an appropriate point as a liquid feed.

5. A method according to claim 1 wherein the evaporated mixture prior to re-compression is used to cool the re-compressed gas mixture in alternating heat exchangers.

6. A method of separating air into a nitrogen fraction and an oxygen fraction which comprises the steps of compressing the air, cooling the compressed air to form a saturated vapour by heat exchange with at least one of the fractions produced, condensing at least part of this vapour by heat exchange with the boiling oxygen fraction, expanding this condensed vapour and using at least part thereof for liquefying vapour to serve as reflux medium, re-compressing the evaporated air to at least atmospheric pressure, cooling and expanding the re-compressed air to saturation point, separating a stream of the air into the aforementioned fractions by counter-current contact in a rectification device with the said reflux medium, and recovering from all cold vapours the cold used for cooling the incoming stream of air.

7. A method according to claim 6 wherein the reflux medium for the rectification device is provided by a part of the separated nitrogen fraction liquefied by indirect heat exchange with the condensed air.

6

8. A method according to claim 6 wherein the rectification device comprises a rectification column surmounted by an enricher wherein the vapours rich in nitrogen are condensed to serve as reflux medium in the column by indirect heat exchange with the condensed air.

9. A method according to claim 6 wherein only a part of the condensed air is used for liquefying the nitrogen fraction, the remainder after expansion to the column pressure being fed to the rectification column at an appropriate point as a liquid feed.

10. A method according to claim 6 wherein the evaporated air prior to re-compression is used to cool the re-compressed air in alternating heat exchangers.

11. A method according to claim 6 wherein the evaporated air prior to recompression is used to cool the recompressed air in alternating heat exchangers and wherein a relatively pure tail fraction of the evaporated air leaving the alternating heat exchanger is used after recompression to cool the separated oxygen fraction.

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