

May 15, 1962

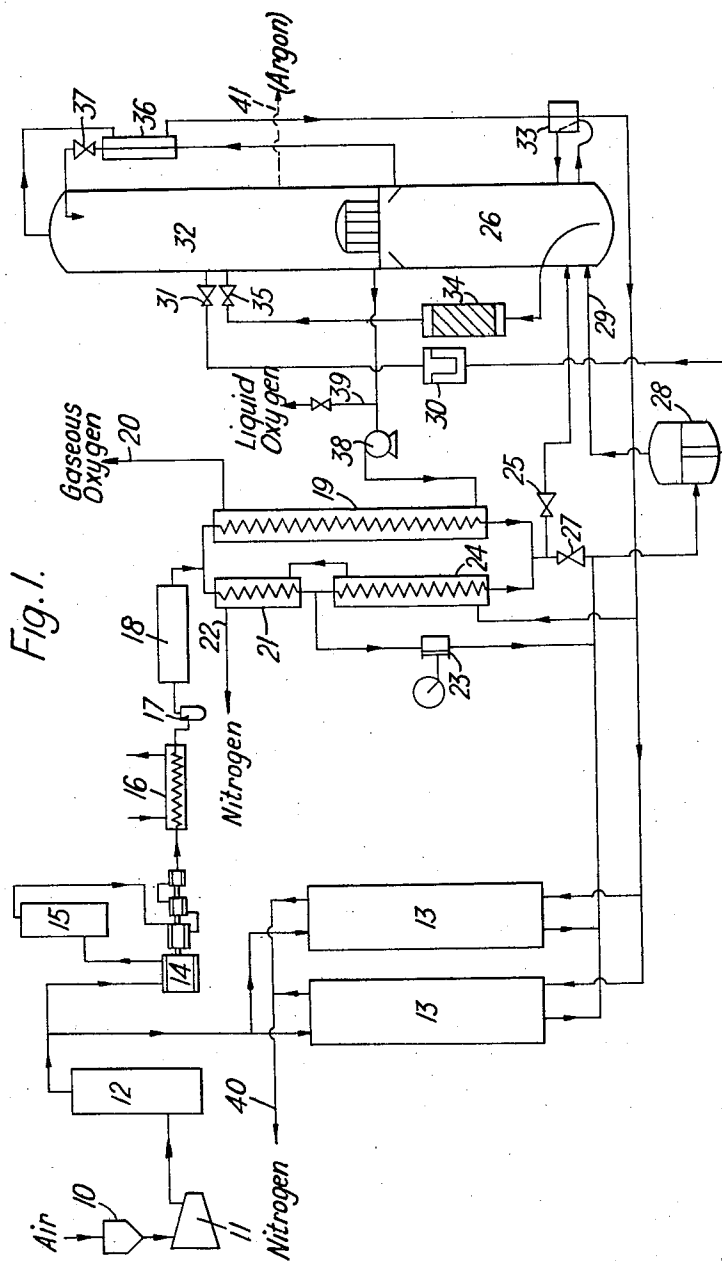
P. M. SCHUFTAN ETAL

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SEPARATION OF AIR

Filed June 5, 1959

2 Sheets-Sheet 1



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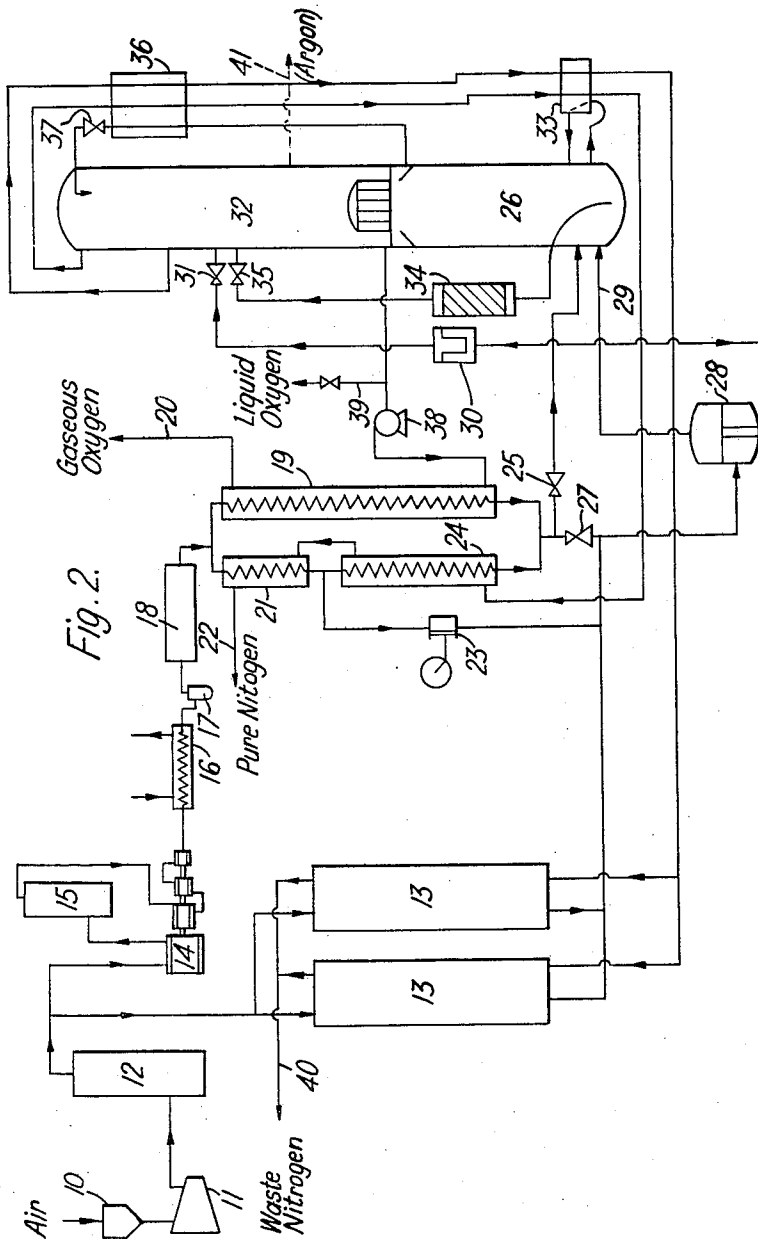
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1

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SEPARATION OF AIR

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Filed June 5, 1959, Ser. No. 818,336
5 Claims. (Cl. 62—13)

This invention relates to the low temperature separation of air. More particularly it relates to a low temperature air separation process in which air is compressed, cooled in a reversing heat exchange zone by heat exchange with a gaseous nitrogen product fraction and subjected to rectification to produce oxygen and nitrogen fractions.

The oxygen fraction is frequently required to be delivered in the gaseous phase under pressure and it has been proposed to pump under pressure a liquid oxygen fraction derived from the rectification step and to vaporize the pressurized liquid by heat exchange with incoming air, thus eliminating the necessity for gaseous oxygen compressors.

Among the objects of the instant invention are to produce a compressed gaseous oxygen fraction which is dry and not substantially contaminated with carbon dioxide, without the use of gaseous oxygen compressors; to produce a minor part of the oxygen product as liquid, when desired, without reduction in the total oxygen yield; and to produce a gaseous nitrogen fraction, which may be substantially free from oxygen or argon, which is dry and substantially uncontaminated by carbon dioxide.

According to the invention, a process for the low temperature separation of air to produce at least the major part of the oxygen product as a compressed gaseous oxygen fraction, and a part of the gaseous nitrogen product as a gaseous nitrogen fraction uncontaminated by water and carbon dioxide, comprises the steps of compressing the air to a relatively low pressure, dividing the compressed air into a major stream and a minor stream, cooling said major stream with removal of condensable constituents by heat exchange with a cold gaseous nitrogen fraction of a mass flow greater than that of said major stream in a reversing heat exchange zone, further compressing said minor stream to a substantially higher pressure with removal of condensable impurities therefrom, dividing said compressed minor stream into a first and a second sub-stream, cooling said first sub-stream by heat exchange with a pressurized liquid oxygen fraction with simultaneous vaporization of said liquid oxygen fraction to produce a compressed gaseous oxygen fraction, cooling said second sub-stream by heat exchange with a second cold gaseous nitrogen fraction, withdrawing said second gaseous nitrogen fraction after such heat exchange as a gaseous nitrogen product uncontaminated by water and carbon dioxide, expanding said first and second sub-streams, subjecting said major stream leaving said reversing heat exchange zone and said expanded first and second sub-streams to rectification in a rectification zone, withdrawing a liquid oxygen fraction from said rectification zone and pumping at least a major part of liquid oxygen fraction to provide said pressurized liquid oxygen fraction vaporized to produce said compressed gaseous oxygen fraction.

While the whole of the liquid oxygen product may be pumped under pressure, a minor part of the product may, if desired, be withdrawn prior to pumping and stored or utilized as liquid. It will be appreciated that when such minor part is retained as liquid, some adjustment of the relative proportions of the various air streams, and/or of the pressure to which the minor air stream is compressed will be required, as hereinafter more particularly described, in order to ensure that the refrigeration requirements of the separation process are met.

2

In order to increase cold production to meet the demand for liquid oxygen, a third sub-stream may be withdrawn from the second sub-stream at a point in the latter's heat exchange with the gaseous nitrogen fraction such that the third sub-stream is at the appropriate temperature, expanded with the production of external work and thereafter combined with the major stream leaving the reversing heat exchange zone.

It will be appreciated that the gaseous nitrogen fraction used to cool the second sub-stream does not pass through the reversing heat exchange zone and is therefore dry and uncontaminated with carbon dioxide. If desired, the rectification step may be arranged to produce both a gaseous nitrogen fraction substantially free from oxygen and argon, and also a relatively impure gaseous nitrogen fraction, the former being used to cool the second sub-stream and the latter to cool the major air stream in the reversing heat exchange zone and to volatilize condensable impurities deposited therein.

It is well known that for the efficient operation of a reversing heat exchange zone, for example provided by regenerators or reversing heat exchangers it is necessary to ensure that all the condensable impurities deposited from the air during its passage through the zone are completely volatilized by the gaseous nitrogen passing through the zone after reversal. Such complete volatilization may be effected by ensuring that the mass flow of gaseous nitrogen through the zone is greater than that of the major air stream passing through the zone, while the minor air stream is in heat exchange with a smaller mass of separation products.

The invention will now be more particularly described with reference to the accompanying drawings in which:

FIGURE 1 illustrates diagrammatically one arrangement of apparatus for practicing the process of this invention; and

FIGURE 2 illustrates diagrammatically a modified form of arrangement of apparatus for practicing the process of the invention.

In the two figures, like parts are indicated by the same reference numeral.

It will be understood that the drawings illustrate diagrammatically preferred apparatus for practicing this invention and that the invention may be carried out using other apparatus. For example, the reversing heat exchange zone has been shown as constituted by regenerators but it might equally be constituted by reversing heat exchangers.

For the sake of clarity and to avoid undue elaboration of the description, the change-over valve system associated with the regenerators has not been shown in the drawings, but it will be appreciated that such a system must be provided.

The drawings will be described with reference to the production of 95% oxygen, by way of example, but it will be appreciated that the apparatus may be used for the production of higher or lower oxygen purities.

Referring to FIGURE 1, air enters the separation system through a filter 10 and passes into an electrically driven multi-stage turbo-compressor 11 which delivers the air at a pressure of 67 p.s.i.g. The compressed air is then cooled in a direct cooler 12 by direct contact with cooling water to approximately the temperature of the cooling water. The air leaving the cooler 12 is divided into a major stream and a minor stream, the relative proportions of the two streams being dependent on the proportion of the oxygen product to be withdrawn as liquid. Where, for example, all the oxygen product is required as compressed gas, the major air stream may comprise 67% by volume of the total air and the minor stream 33% by volume. Where 10% of the total oxygen product is required to be withdrawn as liquid, the major stream may

comprise 62% of the total air and the minor stream 38%.

The major air stream is cooled approximately to its dew point by passage through one of two regenerators 13 of conventional type, the regenerator not in use for cooling air being itself re-cooled by passage therethrough of a cold gaseous nitrogen fraction as hereinafter described. The regenerators 13 are changed over automatically by timing gear after a suitable period, for example 8 minutes. During the passage of the major air stream through the regenerator, carbon dioxide and water vapour present in the air are deposited on the regenerator packing, from which they are volatilized during the cooling cycle by the returning nitrogen stream. Balancing of the regenerators is achieved by ensuring that the mass flow of the nitrogen product passing through the regenerators is greater than that of the major air stream. The cooled major air stream is then fed to the rectification system as hereinafter described.

The minor air stream is compressed in a four-stage piston compressor 14 to a high pressure which will depend upon the proportion of the total oxygen product required to be withdrawn as liquid. Thus, where all the oxygen product is required as compressed gas, the pressure used may be 2350 p.s.i.g. and where 10% of the oxygen product is required as liquid, the pressure may be increased to 2925 p.s.i.g.

At a convenient point in the compression of the minor air stream, for example, as in the drawing between the first and second stages, the air is passed through a scrubbing tower 15 in which its carbon dioxide is removed by scrubbing with caustic soda solution.

The high pressure minor air stream is then cooled to about 12° C. in a cooler 16 by heat exchange with an external refrigerant, such as, for example, boiling dichlorodifluoromethane. Water condensed from the air on cooling is removed in a separator 17. The remainder of the moisture in the minor air stream is then removed by passage through a drier 18 containing a suitable adsorbent such as alumina. While for the sake of simplicity, only one such drier is shown in the drawing, in practice the drier is provided in duplicate for alternate use, one drier being reactivated while the other is in use. Again, it will be appreciated that in accordance with conventional practice, an oil filter is inserted before the drier to remove any traces of oil in the air, and a dust filter after the drier to remove any alumina dust carried over by the air stream from the drier.

The minor air stream is then divided into a first and a second sub-stream, the relative proportions of the sub-streams again depending on the proportion of the total oxygen product required as liquid. Thus, when all the oxygen is required as compressed gas, the first sub-stream may comprise 80% of the minor air stream and the second sub-stream only 20%, whilst where 10% of the oxygen product is required as liquid, the first sub-stream may comprise 64% of the minor air stream and the second sub-stream 36%.

The first sub-stream is cooled to -160° C. in a heat exchanger 19 by heat exchange with a pressurized liquid oxygen fraction, which is itself vaporized and superheated to 6° C. The compressed gaseous oxygen fraction so produced is withdrawn as product at 20.

The second sub-stream is cooled to about -33° to -35° C. in a precooler 21 by heat exchange with a gaseous nitrogen fraction, which after passing through the precooler 21 is withdrawn at 22 as a dry gaseous nitrogen product uncontaminated with carbon dioxide.

From the cooled second sub-stream leaving the precooler 21, a third sub-stream is bled off and expanded to the pressure of the major air stream leaving the regenerators 13 through an expansion machine 23 with the production of external work. The expansion machine 23 may be a turbine or an expansion engine. The amount of air bled off to form the third sub-stream will again depend on the proportion of the oxygen product

required as liquid. Thus, where all the oxygen product is required as compressed gas, the proportion of the minor air stream forming the third sub-stream may be as low as 4.5%, whilst where 10% of the total oxygen product is required as liquid, the refrigeration requirements are, of course, greater and the third sub-stream may comprise 21% of the minor air stream. The expanded third sub-stream is then combined with the cooled major air stream leaving the regenerators 13.

The remainder of the second sub-stream after withdrawal of the third sub-stream is further cooled to about -170° C. in a heat exchanger 24 by heat exchange with the cold gaseous nitrogen fraction subsequently passed through the precooler 21. The first sub-stream leaving the exchanger 19 and the remainder of the second sub-stream leaving the exchanger 24 are then combined and expanded through a valve 25, and fed to the lower column 26 of a conventional double column rectification system. The column 26 operates at about 78 p.s.i.g. A small proportion (for example, about 4%) of the combined first and second sub-streams is bled off upstream the valve 25 and expanded with consequent liquefaction through a valve 27 into the major air stream leaving the regenerators 13.

The major air stream is then fed to an equalizer 28. In the drawings, the equalizer 28 is shown as located apart from the rectification columns, but if desired, it may be located at the bottom of the lower column 26. In the equalizer 28, efficient contact between the vapour and the liquid is obtained and residual higher boiling impurities, such as carbon dioxide, are precipitated. Vapour from the equalizer 28 is fed into the lower column 26 at 29, while a small liquid residue containing the precipitated higher boiling impurities is withdrawn from the bottom of the equalizer 28, passed through a filter 30 and expanded through a valve 31 into the upper column 32 of the rectification system. This upper column 32 operates at substantially atmospheric pressure. A portion of the air fed to the column 26 is withdrawn, liquefied in a condenser 33 by heat exchange with a gaseous nitrogen fraction leaving the rectification system and returned to the column 26. The amount of air so withdrawn and liquefied is adjusted so that the temperature of the gaseous nitrogen fraction leaving the condenser 33 is about -175° C.

In the column 26, the air is separated into an oxygen-enriched liquid fraction collecting at the bottom of the column and a liquid nitrogen fraction which is formed at the top of the column. The oxygen-enriched liquid is withdrawn from the column 26 and passed through an adsorber 34 in which hydrocarbon impurities, such as acetylene or traces of oil from the expansion machine 23, are removed by adsorption on a suitable adsorbent such as silica gel. While for simplicity only one adsorber 34 is shown in the drawing, in practice the adsorber is provided in duplicate so that one can be regenerated while the other is on stream. From the adsorber 34, the oxygen-enriched liquid is expanded through an expansion valve 35 into the upper column 32 of the rectification system.

The liquid nitrogen fraction formed at the top of the lower column 26 is used as reflux liquid in both columns, a part of the liquid nitrogen being withdrawn, cooled in a heat exchanger 36 against gaseous nitrogen leaving the upper column 32, and expanded through an expansion valve 37 into the top of the upper column 32.

In the upper column 32, the air is further separated into a liquid oxygen fraction collecting at the bottom of the upper column and a gaseous nitrogen fraction withdrawn from the top of the column. The liquid oxygen product is withdrawn from the bottom of the upper column and fed to a pump 38 where it is pumped to a pressure of 400-600 p.s.i.g. The pressurized liquid oxygen fraction is then passed to the heat exchanger 19

5

where it is vaporized by heat exchange with high-pressure air as previously described.

If required, a part of the liquid oxygen fraction may be withdrawn through a valve-controlled outlet 39 upstream the pump 38 and stored or used as liquid. Such stored liquid may, for example, be used to produce compressed gaseous oxygen during periods when the separation plant is shut down.

The gaseous nitrogen fraction is withdrawn from the top of the upper column and passed successively through the exchanger 36 and the condenser 33 as hereinbefore described. After leaving the condenser, the gaseous nitrogen fraction is divided into two streams, one stream passing through the regenerators 13 and being withdrawn at 40 as waste nitrogen contaminated with water vapour and carbon dioxide. The other stream passes successively through the exchanger 24 and the pre-cooler 21 and is withdrawn at 22 as a dry gaseous nitrogen product uncontaminated with carbon dioxide as previously described.

Substantially complete volatilization of condensed deposits in the regenerator 13 through which the nitrogen stream is passing is ensured by arranging that the mass flow of this stream is greater than that of the major air stream passing through the regenerators.

The alternative arrangement shown in FIGURE 2 is substantially identical in operation with the exception that two gaseous nitrogen fractions are withdrawn from the upper column 32. One fraction consisting of nitrogen substantially free from oxygen and argon is withdrawn from the top of the upper column 32, passed through the heat exchanger 36 and condenser 33 and thence passed directly to the exchanger 24. In this way a substantially pure, dry nitrogen fraction is withdrawn at 22. A second relatively impure nitrogen fraction is withdrawn from the upper column 32 at a point a few plates below the top, passed through separate paths in the exchanger 36 and condenser 33 and thence passed directly to the regenerators 13.

It will be appreciated that the figures given in the above description for the proportions of the total air forming the various air streams and sub-streams and for the pressure to which the minor air stream is compressed are only exemplary and may be varied if required, provided that sufficient refrigeration is produced to provide for the cold requirements of the process. Thus, by raising the pressure to which the minor air stream is compressed above the values suggested in the description, it is possible to reduce the amount of air passing through the expansion machine 23, or even to eliminate this machine completely, without substantially reducing the total refrigeration produced.

When the process of the instant invention is used for the production of oxygen of high purity, it may be desirable to withdraw an argon-containing fraction from the upper column (as indicated in the drawings at 41) and to recover the argon therefrom by conventional means.

We claim:

1. A process for the low temperature separation of air to produce at least the major part of the oxygen product as a compressed gaseous oxygen fraction and a part of the nitrogen product as a dry gaseous nitrogen fraction uncontaminated by carbon dioxide, comprising the steps of

6

compressing the air to a relatively low pressure, dividing the compressed air into two streams, namely a major stream and a minor stream, cooling said major stream with removal of condensible impurities by heat exchange with a first cold gaseous nitrogen fraction of a mass flow greater than that of said major stream in a reversing heat exchange zone, further compressing said minor stream to a substantially higher pressure with removal of condensible impurities therefrom, dividing said further compressed minor stream into a first and a second sub-stream, both at said substantially higher pressure, cooling said first sub-stream by heat exchange with a pressurized liquid oxygen fraction with simultaneous vaporisation of said liquid oxygen fraction to produce a compressed gaseous oxygen fraction, cooling said second sub-stream by heat exchange with a second cold gaseous nitrogen fraction, withdrawing said second gaseous nitrogen fraction after such heat exchange as a dry gaseous nitrogen product uncontaminated by carbon dioxide, dividing from said cooled second sub-stream a third sub-stream, expanding said third sub-stream with the performance of external work, and combining said expanded third sub-stream with the major stream leaving said reversing heat exchange zone, further cooling the remainder of said second sub-stream by heat exchange with said second gaseous nitrogen fraction, expanding said first sub-stream and the remainder of said second sub-stream, subjecting said combined major stream and third sub-stream and said expanded first and second sub-streams to rectification in a rectification zone, withdrawing a liquid oxygen fraction from said rectification zone and pumping at least a major part of said liquid oxygen fraction under pressure to provide said pressurized oxygen liquid oxygen fraction vaporized by heat exchange with said first sub-stream.

2. Process according to claim 1 wherein a part of said liquid oxygen fraction is withdrawn as liquid prior to pumping.

3. Process according to claim 1 wherein said second gaseous nitrogen fraction passed in heat exchange with said second sub-stream is a relatively pure gaseous nitrogen fraction withdrawn from said rectification zone and said first gaseous nitrogen fraction passed in heat exchange with said major air stream in said reversing heat exchange zone is a relatively impure gaseous nitrogen fraction withdrawn from said rectification zone.

4. Process according to claim 1, including the steps of scrubbing said combined major air stream and third sub-stream, prior to rectification with a liquid derived from said minor air stream, whereby residual higher boiling impurities in said combined major air stream and third sub-stream are precipitated, and removing said impurities by filtration of said liquid.

5. Process according to claim 1 wherein an argon-containing fraction is withdrawn from said rectification zone.

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