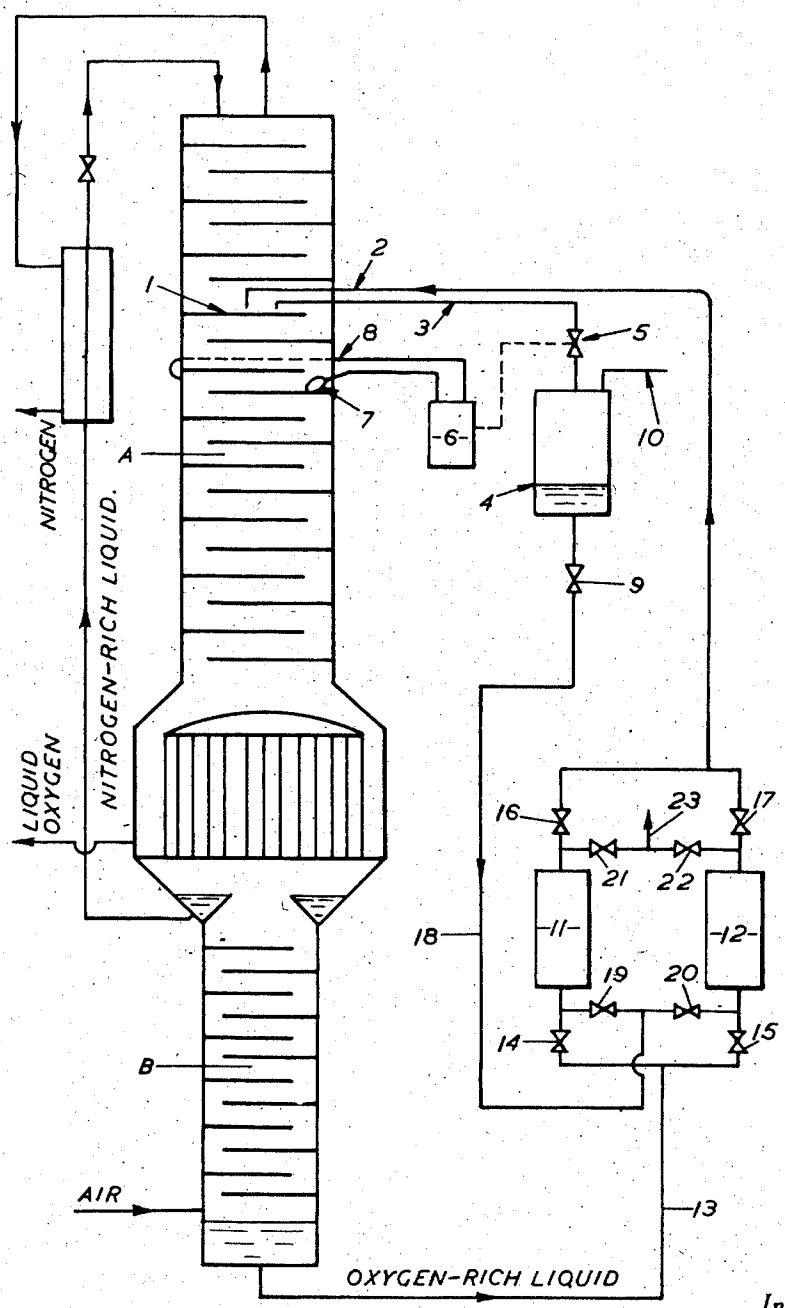
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SEPARATION OF AIR

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SEPARATION OF AIR

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1

This invention relates to the separation of air by liquefaction and subsequent rectification. While the invention can be applied to processes of the type producing gaseous oxygen as hereinafter described, it is particularly useful when applied to processes for the production of liquid oxygen of high purity.

In such processes, the yield and purity of the oxygen produced are affected to a high degree by variations in the refrigeration performance of the separation plant. The optimum conditions of yield and purity of oxygen are obtained when the refrigeration performance of the plant is such that liquefaction of the air occurs to an extent equal to the oxygen recoverable from the air. A small increase in the extent of liquefaction, however, will lead to the production of a liquid oxygen fraction of insufficient purity for commercial use. On the other hand, a decrease in the amount of liquefaction results in a decreased yield of liquid oxygen.

If the refrigeration performance of the plant is normally such that liquefaction occurs to the optimum extent, such variations may be caused by even slight disturbances in the operation of the plant, for example, by the blowing of drains or by the change-over of refrigerating driers.

In order to avoid the production of low-purity oxygen by such variations in the extent of liquefaction, it has heretofore been customary to operate the plant at a refrigeration performance below that required for optimum liquefaction with a consequent decrease in the yield of oxygen.

It is an object of the present invention to provide a process for the production of high purity liquid oxygen from air which avoids any risk of loss of purity of the oxygen by fluctuations in the refrigeration performance of the plant without any significant decrease in the oxygen yield.

According to the present invention, in a process for the separation of air by liquefaction and subsequent rectification in a rectification zone, the cold supplied to the zone is such that under normal conditions of working liquefaction of the air occurs to an extent in excess of that of the oxygen recoverable from the air, the excess liquid being automatically withdrawn from the rectification zone at an intermediate point at a rate controlled by fluctuations of the conditions in the said rectification zone due to slight variation of the plant refrigeration performance. Under these conditions slight variations in the refrigeration performance of the plant will lead only to corresponding variations in the rate of withdrawal of the excess liquid from the rectification zone, and will not cause any substantial variation in the temperature gradient in the zone and hence the purity of the oxygen product will remain substantially constant.

2

A very small proportion of the oxygen in the air being processed is lost in the excess of liquid withdrawn from the rectification zone according to the invention but because of the steady conditions which are achieved by the use of the invention it is possible to operate the plant at a higher refrigeration performance than is otherwise possible if loss of oxygen purity due to fluctuations in refrigeration performance is to be avoided. The higher refrigeration performance which is possible without loss of oxygen purity, by application of the invention enables yield of liquid oxygen from the plant to be increased and this largely compensates for the small loss of oxygen in the excess of liquid withdrawn from the rectification zone, and may even result in an increase in the net yield of liquid oxygen.

The withdrawal of the excess liquid may be arranged to be automatically controlled by the fluctuations in temperature at the point of maximum temperature gradient within the zone, caused by changes in the refrigeration performance of the plant. For example, a sensitive thermometer such as that of the vapour pressure type may be inserted in the rectification zone at the point of maximum temperature gradient and the temperature changes registered thereby arranged to operate a valve controlling the withdrawal of the excess liquid, so as to restore the temperature to its correct value. By this means the temperature gradient in the zone can be maintained within close limits. The thermometer may if desired be arranged for pressure compensation so that its reliability and sensitivity may be unaffected should the operating pressure of the rectification zone fluctuate.

Although the excess liquid may be withdrawn at any suitable point in the rectification zone, it is preferred to effect the withdrawal at the point of maximum argon concentration, whereby rectification conditions in the rectification zone are improved and the oxygen yield thereby increased.

The cold in the liquid withdrawn from the rectification zone may be recovered either continuously or intermittently. For example, it may be continuously transferred to one of the products of separation, such as the liquid oxygen fraction, to effect sub-cooling thereof, or for producing some liquid nitrogen. Alternatively the liquid may be stored for utilisation of the cold in inter-

mittently operated change-over devices, such as filter units through which a crude liquid fraction is passed in advance of the rectification zone. In order that the object of the invention should be attained, it is however essential that the purpose to which the recovered cold is put, should be such that the cold is not returned to the rectification zone.

In the drawing, the double column comprises an upper column A and a lower column B and operates in the usual manner for the production of liquid oxygen and a gaseous nitrogen fraction.

One particular application of the present invention to a plant producing liquid oxygen by rectification of liquid air, using the well known double column, will now be described with reference to the accompanying drawing, which shows diagrammatically that portion of the upper section of the double column which embodies the invention.

Referring to the drawing, "rich liquid" containing about 32% of oxygen from the lower column is led on to the feed tray 1 by pipe 2. The plant is operated with a slight excess of refrigeration, i. e. excess of liquid production, which is compensated by the continuous withdrawal of an equivalent quantity of liquid through the pipe 3 into the liquid reservoir 4.

A vapour pressure thermometer 7, is installed at the point of maximum temperature gradient in the column, i. e., about two trays below feed tray 1, in contact with the liquid on the tray. The thermometer 7 is connected to the controller 6. The gas space above the liquid in contact with the vapour pressure thermometer 7 is connected by line 8 to the controller 6. The liquid used in the vapour pressure thermometer 7, is so chosen that its vapour pressure-temperature relationship is very similar to that of the liquid composition which it is desired to maintain on the control tray in contact with the vapour pressure thermometer. In the controller 6 the differential pressure between vapour pressure thermometer 7 and the column as transmitted by line 8 is used to operate a suitable mechanism actuating withdrawal valve 5. If the column pressure rises or falls slightly the temperature of liquid in contact with the vapour pressure thermometer 7 will rise or fall correspondingly, for a fixed liquid composition on the tray at 7 and the pressure transmitted by the thermometer will rise or fall by an amount equal or almost equal to that of the column. By this means changes in column pressure which may occur in practice will not, per se, produce any reaction on the controller 6.

If the plant refrigeration increases, the liquid composition on the tray at 7 will tend to become richer in the lower boiling constituent, i. e. nitrogen, and the temperature recorded by the thermometer 7 will tend to decrease. This causes a differential pressure between 7 and 8 which brings the controller 6 into operation and increases the rate of liquid withdrawal through valve 5. Similarly, if the plant refrigeration decreases the temperature recorded by thermometer 7 will tend to increase and the rate of liquid withdrawal through valve 5 will be decreased by the controller 6. By this means the liquid composition at 7 in the column can be maintained constant within close limits and the yield and purity of the liquid oxygen produced at the base of the upper column remains unaffected by the variations in plant refrigeration.

In this application the liquid which accumulates in the reservoir 4 can be withdrawn through valve 9 and the cold utilised in precooling intermittently operated filters interposed in the "rich liquid" line to the upper column. Any vaporisation from liquid reservoir 4 is vented by pipe 10.

The intermittently operated filters are indicated in the drawing at 11 and 12. The rich liquid withdrawn from the lower column through pipe 13 passes to one or other of these filters through valve 14 or 15 and from the filters through valve 16 or 17 to the pipe 2. When valves 14 and 16 are open, valves 15 and 17 will of course be closed, the position of these pairs of valves being reversed at predetermined intervals so that the rich liquid passes alternately through the two filters 11 and 12. The entries to the filters are also connected by pipe 18 to the valve 9 through valves 19 and 20 respectively, and similarly the exits from the filters are connected through valves 21 and 22 to a liquid outlet line 23.

During the period when filter 11 is in use for filtering the rich liquid, filter 12 is precooled by passage therethrough of the excess liquid from reservoir 4, valves 19 and 21 being closed and valves 20 and 22 open.

When the filters are changed over, the positions of these valves are reversed so that the excess liquid passes through filter 12.

While the process of the invention has particular advantages when applied to air separation processes for the production of liquid oxygen, it may be applied with advantage to processes producing oxygen in the gaseous state, where such processes require a substantially constant temperature gradient in the rectification zone, for example where a side stream has to be withdrawn from the rectification zone. Such a side stream could serve either to remove a fraction relatively rich in argon and containing a preponderance of either oxygen or nitrogen, whereby the bulk of the oxygen and nitrogen are obtained at very high purity; or the side stream could serve to remove a fraction rich in argon and having a very low nitrogen content for use as the feed to an argon column. In both cases, it is an advantage if the temperature at the point of withdrawal of the side stream is fixed and controlled within narrow limits.

I claim:

1. Process for the separation of air by liquefaction and subsequent rectification in a rectification zone, wherein the cold supplied to said zone is such that under normal conditions of working liquefaction of the air occurs to an extent in excess of the oxygen content of the air, the excess liquid being automatically withdrawn from said rectification zone at an intermediate point of said zone in accordance with temperature fluctuations at a point of maximum temperature gradient within the zone such that the temperature gradient at this control point is maintained within close limits.

2. Process according to claim 1, wherein the withdrawal of said liquid is automatically controlled in accordance with temperature fluctuations at a point of maximum temperature gradient within said zone as indicated by changes in composition at this point.

3. Process according to claim 1, wherein said excess liquid is withdrawn at the point of maximum argon concentration such that rectification conditions in the rectification zone are improved by the withdrawal of the argon in the excess liquid.

4. Process according to claim 1 wherein said

excess liquid is utilised for cooling purposes external to the cold requirements of the separation process.

5. Process according to claim 1, wherein said excess liquid is used for sub-cooling a separation product after its final removal from the separation system.

6. Process according to claim 1, wherein said excess liquid is used to cool a filter unit through which a crude liquid fraction is passed in advance of the rectification zone.

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References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
1,880,981	Pollitzer	Oct. 4, 1932
2,151,248	Vaughan	Mar. 21, 1939
2,280,383	De Baufre	Apr. 21, 1942
2,501,999	Fausek	Mar. 28, 1950
2,506,350	De Baufre	May 2, 1950
2,519,892	Dennis	Aug. 22, 1950
2,541,409	Cornelius	Feb. 13, 1951
2,553,550	Collins	May 22, 1951