

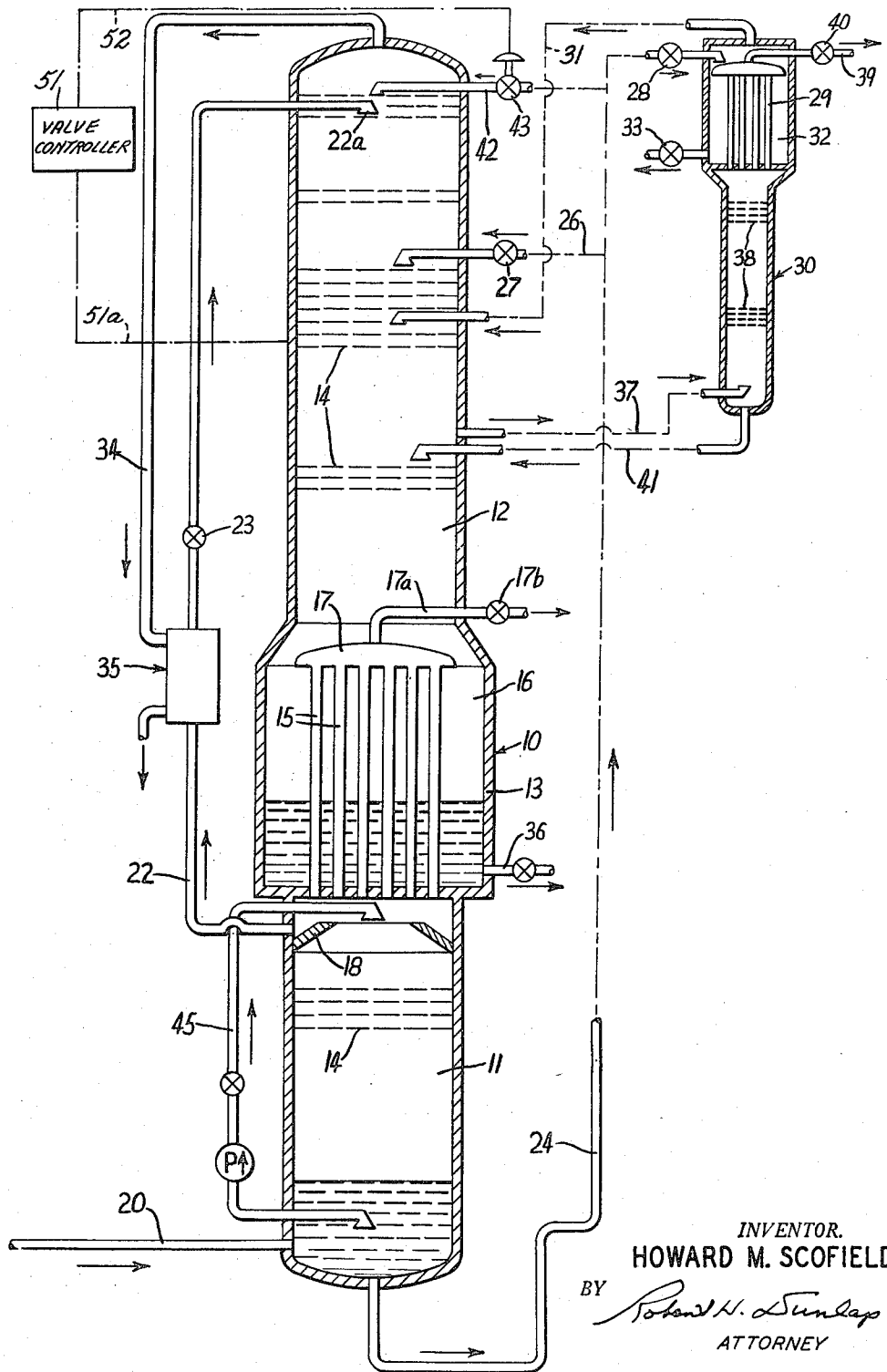
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HIGH ARGON RECOVERY USING KETTLE TOP FEED-TOP PINCH PRINCIPLE

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**HIGH ARGON RECOVERY USING KETTLE TOP
FEED-TOP PINCH PRINCIPLE****Howard M. Scofield, Kenmore, N.Y., assignor to Union
Carbide Corporation, a corporation of New York****Application August 17, 1954, Serial No. 450,452****13 Claims. (Cl. 62—21)**

This invention relates to a process of separating low boiling ternary gas mixtures and more particularly to the recovery of argon by low temperature separation of air.

Most of the argon used in the world is produced as a by-product in the production of oxygen by low temperature separation of atmospheric air into its major constituents. It has not been found practical or economical to produce it otherwise. But known oxygen plants have a relatively low argon producing capacity because a sizable fraction of the argon in the air treated is lost in the effluent nitrogen. With the greatly increased consumption of argon in recent years, presently employed processes and plants have not been able to meet the increased demands without incurring an over-production of oxygen.

The recovery of an argon product when separating air by low temperature rectification has been accomplished by withdrawing fluid from an intermediate zone of a final rectification, where the argon concentration is high and nitrogen concentration is low, and then subjecting the withdrawn fluid to auxiliary rectification. Although argon in a state of high purity has been obtained in this manner, known processes of this type have not functioned efficiently with respect to the production of argon and only a relatively low percentage of the argon present in each unit volume treated has been recovered, the unrecovered argon being lost with the oxygen and nitrogen products.

The primary object of this invention, therefore, is to provide an improved process for obtaining increased producing capacity in existing and new equipment in the production of an intermediate boiling point component in the separation of a ternary gas mixture. More particularly, the present invention has for its object to obtain increased recovery efficiency in the production of argon in air separating processes recovering both oxygen and argon. Another object of the present invention is to reduce the unit cost of argon production.

Still another object of the invention is to provide new features of control to maintain stabilized operation of the process mentioned hereinabove.

The foregoing and other objects of this invention are brought about according to the invention by making use of several correlatable principles which include:

First, that the upper part of the low pressure stage of a well known double rectification column can be made to trap argon, which has heretofore left the column with the effluent waste nitrogen top product and has been lost, and to wash such argon down the column to a zone where the fluid has relatively high argon content and very low nitrogen content. The argon trap is produced by applying an "oxygen-pinch" to the top of the low pressure rectification stage where nitrogen-rich reflux liquid is introduced, as well as to other feed points in the upper column above the argon take-off. At these zones, particularly the top zone where the oxygen-nitrogen pinch is coldest because of the cold nitrogen liquid being introduced therein, the oxygen-nitrogen driving

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force is very small and the argon-nitrogen driving force can be utilized to effect a washing down of the argon out of the waste nitrogen top product.

In the rectification art, a "pinch" refers to a reflux condition where the contacting liquid and vapor are in substantial equilibrium, there being little driving force for rectification and there being required several trays to effect a small composition change. As herein employed an "oxygen-pinch" may be defined as a reflux condition where there is a zone including several trays over which practically no change in the oxygen content of the descending liquid occurs but an effective change in the argon content is maintained. Accordingly the application of an "oxygen-pinch" at the top of the low pressure rectification stage to produce an argon trap where argon is washed out of the rising vapor is hereinafter referred to as the "top oxygen-pinch" principle.

Second, that in the separation of air by a two-stage rectification wherein nitrogen-rich reflux liquid and oxygen-enriched reflux liquid are introduced into the second rectification stage, the proper "top oxygen-pinch" can be applied by regulation of the argon content and oxygen content in the refluxing nitrogen-rich liquid introduced in the low pressure stage of the rectifying column. In other words, most favorable argon recoveries have been obtained by using a nitrogen-rich washing liquid at the top of the low pressure rectification stage that is relatively free of argon, but has proper oxygen content to effect equilibrium conditions favorable to argon enrichment and unfavorable to oxygen enrichment.

Third, that, in liquid oxygen plants, limiting and adjusting the purity of the effluent nitrogen and of the nitrogen-rich reflux liquid minimizes reduction in argon content of the descending liquid at the bottom of the top part of the upper column in the region of oxygen enrichment just above the oxygen-enriched liquid feed, where equilibrium conditions are unfavorable to argon enrichment, and accordingly increases the total amount of argon passing to the lower part of the upper column where argon is removed.

In accordance with the principles of this invention, a "top oxygen-pinch" is established in the upper, low pressure column of a double column rectification unit by washing down the rising vapor at the top region of the column with a nitrogen-rich reflux liquid having a minimum argon content and sufficient oxygen content to establish oxygen-nitrogen equilibrium conditions in such top region. In this way the oxygen-nitrogen rectification driving force is reduced or pinched while there still remains an argon-nitrogen driving force which can be utilized to rectify the argon and nitrogen.

The requirement that the nitrogen-rich reflux liquid be relatively free of argon yet still have some oxygen content was difficult to meet, for an inspection of the boiling points of oxygen (-183° C.), argon (-186° C.) and nitrogen (-196° C.) reveals that in a rectification separation, argon may be removed only after the oxygen has been separated. The present invention meets this requirement by running the high pressure column with a high reflux rate so as to wash nearly all of the oxygen and argon out of the nitrogen-rich liquid that is produced at the top of the high pressure column and subsequently transferred to the low pressure column for use as a reflux liquid and by mixing with such nitrogen-rich liquid oxygen-enriched liquid withdrawn from the lower part of the high pressure column.

In the preferred embodiment of the invention, proper oxygen content in the nitrogen-rich reflux liquid is obtained by passing part of the oxygen-enriched liquid accumulating at the bottom of the high pressure column to the top of the low pressure column. This method is advantageous for it increases the argon production capacity

of the unit without reducing the oxygen production capacity, it produces a larger available quantity of reflux liquid at the pinch zone because of the nitrogen content of the oxygen-enriched liquid, and it can be performed simply without requiring additional power equipment because the oxygen-enriched liquid under the high pressure of the high pressure column can be readily throttled as a clean liquid into the low pressure column.

According to another aspect of the invention, the argon in the nitrogen-rich transfer liquid can be reduced in the high pressure column while retaining a proper oxygen concentration for producing an oxygen pinch condition at the top of low pressure column. This is accomplished by recirculating a part of the oxygen-enriched liquid at the bottom of the high pressure column to the top of the high pressure column. In this way the argon is washed down while oxygen concentration of the nitrogen-rich liquid at the top of high pressure column remains high enough for producing the desired pinch at the top of the low pressure column. This method requires the use of a pump to recirculate the oxygen-enriched liquid.

These and other objects, features and advantages of the present invention will become apparent from the following detailed description of the accompanying drawing which shows in conventional diagrammatic form air separation apparatus illustrating the principles of the improved process as applied to a plant wherein an argon fraction, a nitrogen fraction, and a substantially pure liquid oxygen fraction are produced.

The present invention will be described in connection with the apparatus of the drawing which is particularly suitable for the separation of air to obtain a liquid oxygen product substantially free of higher boiling point impurities and a product having a high content of argon. However, modifications within the scope of the invention may be made if it is desired to recover the oxygen product in gaseous rather than in liquid form.

Referring now to the drawing, the air rectifying apparatus, except as explained hereinafter, is generally of conventional construction and includes a double column, indicated at 10, having the usual high pressure stage chamber or lower column 11, a low pressure stage chamber or upper column 12 extending above the lower column, and main condenser 13 disposed between the two columns and sealing the high pressure chamber 11 from the low pressure chamber 12. The high and low pressure chambers may contain customary column trays, such as perforated plates 14, which effect intimate contact with vapors rising in the column and reflux liquid flowing down the column. The main condenser 13 is provided with vertical tubes 15, disposed in liquid oxygen collecting chamber 16 at the bottom part of the upper column, the lower ends of these tubes opening into the upper end of the high pressure rectification chamber 11 to receive the vapor used in the rectifying action of the lower column and effect a condensing of such vapor by the action of relatively cold oxygen liquid in chamber 16 of the upper column. The upper ends of the tubes open into a sealing dome 17, from which nitrogen rich gas may be taken off, as desired, through a conduit 17a by operation of a control valve 17b. An annular shelf 18 is disposed in high pressure chamber 11 in a position underlying the radially outer tubes of the main condenser 13 for catching and collecting high purity condensed nitrogen falling from such tubes.

The bottom of the upper column or low pressure rectifying chamber 12 opens into the liquid oxygen collecting chamber 16 of the main condenser 13 for effecting the boiling of part of liquid oxygen collected therein to produce vapor for the rectifying action in the upper column. The oxygen boils at the low pressure of the upper column at a temperature which is lower than the condensing temperature of the nitrogen in the condenser which is under the pressure of the lower column.

Cooled and cleaned air under a compression of about 80 p.s.i. is fed into the bottom part of the lower column 11 through a conduit 20. This air is subjected to an initial rectification whereby an oxygen-rich liquid fraction is produced that accumulates at the base of the lower column and a nitrogen-rich gaseous fraction is produced at the top of the lower column. The nitrogen rich gas enters the tubes 15 of the main condenser 13 and is partially condensed, the condensed fraction falling back into lower column where part is received on annular shelf 18 and part is received on the top trays to serve as reflux liquid for the lower column. The vapor fraction of the nitrogen rich gas passing out of the condenser enters and accumulates in the sealing dome 17 where it can be drawn off as desired through conduit 17a as mentioned above.

The nitrogen-rich liquid collected on shelf 18 is withdrawn from the lower column through a conduit 22 and passed through a heat exchanger 35 in heat exchange relation with effluent nitrogen gas leaving the upper column 12 through a conduit 34. After it has been cooled by the effluent nitrogen it traverses a throttling valve 23 where reduction in pressure results in a further drop in temperature. It is then passed into the top part of the upper column as described hereinafter to provide cold nitrogen-rich reflux liquid for the rectifying action of the upper column.

The oxygen-enriched liquid that accumulates at the base of the lower column is withdrawn through a conduit 24. A portion of this liquid is introduced directly into the upper column by a conduit 26 controlled by throttling valve 27 and another portion is passed through a throttling valve 28 to a chamber 32 surrounding a condenser 29 in the top part of an auxiliary rectifying column 30. The oxygen-enriched liquid entering such column is vaporized and passed through a conduit 31 into the upper column for further rectification. A liquid oxygen product may be withdrawn from the upper column 12 at a valved outlet 36.

At this point it may be explained that in order to apply the "top oxygen-pinch" according to the principles of this invention, it is necessary to provide a section at the top of the upper column where the reflux liquid is used principally to wash argon from the rising vapor.

Referring now in particular to the operation of the main rectification column, oxygen pinch regions are established in the upper part of the upper, low pressure column to create equilibrium conditions favorable to argon enrichment and unfavorable to oxygen-enrichment. Of the various oxygen pinches produced in the upper column the top oxygen pinch is the most important with respect to high argon recovery, for it is in this top part of the column that the nitrogen-rich transfer liquid is coldest and argon is most effectively washed down by the coldest liquid.

In accordance with the present invention, the top oxygen pinch is established by providing a reflux liquid at the top part of the low pressure column 12 which is relatively free of argon but has a sufficient oxygen content to achieve oxygen-nitrogen phase equilibrium conditions in the column at its feed point. In a preferred practice of this invention, these conditions are satisfied by running the high pressure column 11 at a high reflux rate to wash nearly all of the argon and oxygen out of the liquid that accumulates on shelf 18, by transferring such relatively argon-free liquid to the upper column through conduit 22, introducing such liquid into the upper part of the upper column just below the top, as at 22a, and by feeding a part of the oxygen-enriched liquid withdrawn from the high pressure column to the top of the low pressure column just above the nitrogen-rich transfer feed through a conduit 42 controlled by a throttling valve 43. In this manner, the substantially argon-free reflux liquid in the high pressure column is provided with a readily available supply of oxygen for maintaining the concentration re-

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quired to produce the desired oxygen pinch at the top of the low pressure column.

Other oxygen pinches in the upper part of the upper column, which enhance the wash-out of argon from the rising vapor are provided by proper location of the feed conduits 26 and 31, respectively, to introduce oxygen-enriched liquid and oxygen-enriched vapor at points producing substantial oxygen-nitrogen equilibrium conditions in the column.

An indication of the effectiveness of the oxygen pinch is obtained by a purity measurement, taken directly or indirectly as by temperature measurement, below any of the various feed points. Accordingly, control of the oxygen pinches can be achieved, when a constant proportion of nitrogen-rich liquid is withdrawn from the high pressure column for transfer to the low pressure column, by adjustment of the amount of oxygen-enriched liquid feed to the top of the low pressure stage to keep the purity condition at the measuring station within the desired range. That is, a temperature measurement at a point in upper column 12 below the oxygen-enriched vapor feed point is transmitted by a connection 51a to the valve control mechanism 51 which in turn transmits, through a connection 52, a valve controlling impulse to actuate throttling valve 43, controlling the quantity of oxygen-enriched liquid introduced to the top of upper column 12 through conduit 42. In this manner, the oxygen pinches in upper column 12 are controlled by sensing the fluid temperature at a measuring station in such column, and adjusting the quantity of the oxygen-enriched reflux liquid in response to such sensing.

At an intermediate point of the upper column where the vapor has a relatively high content of argon and a relatively low content of nitrogen, a vapor outlet conduit 37 is connected to the upper, low pressure column to conduct argon containing vapor into a lower part of auxiliary rectifying column 30. This column may also have gas and liquid contact means such as trays 38. The argon containing vapor passes upwardly through the column 30 and is washed with a reflux liquid produced by condensation of the rising vapors in condenser 29 at the top of the column. The vapors passing through the tubes of condenser 29 are subjected to partial liquefaction, the liquid produced being rich in argon and forming a suitable reflux liquid for washing down the rising vapor in the column. The argon-rich vapor that remains undensified may be withdrawn through a conduit 39 that is provided with a control valve 40. The liquid which accumulates at the bottom of the auxiliary column is substantially reduced in its argon content and is preferably returned by a conduit 41 to the upper column 12 of the main rectification, being admitted at a point near the argon take-off.

Although a large part of the higher boiling point impurities will be removed before rectification, there is usually a gradual concentration of high boiling point impurities in the condenser 29 of the auxiliary column, and a drain valve 33 connected to a lower part of the chamber 32 is provided to allow removal of such concentrate at infrequent intervals.

Alternatively, the required oxygen concentration in a substantially argon-free refluxing liquid can also be obtained by withdrawing from the high pressure column through conduit 45 part of the oxygen-enriched liquid that accumulates at the bottom thereof, the so-called "crude oxygen," and recirculating such crude oxygen in the lower, high pressure column by feeding the withdrawn part into the top of the lower column just above the nitrogen-rich liquid collector shelf 18. A pump P is employed in conduit 45 to raise the crude oxygen withdrawn to the recirculation feed level.

The crude oxygen is recirculated to produce an oxygen pinch at the top of the lower, high pressure column which will result in a washing down of argon out of the shelf liquid while providing the required oxygen concentration

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in the shelf liquid so that when such liquid is transferred through conduit 22 to the top of upper column 12, an oxygen pinch reflux condition will be established in the top region of the upper column. It will be understood that in the practice of this invention by crude oxygen recirculation in the lower column, the crude oxygen feed to the top of the upper column is not required.

It is to be understood that the detailed description and the accompanying drawings are illustrative only and that the improvements herein disclosed may be practiced in various forms within the scope of the appended claims.

What is claimed is:

1. A process for the low temperature separation of air to obtain an effluent nitrogen product, an oxygen product and an argon-containing product, which comprises: subjecting air to a two-stage rectification which includes a rectification at a higher rectification pressure to provide at the top zone thereof a nitrogen-rich liquid and at the bottom zone thereof an oxygen-enriched liquid for a main rectification at lower pressure, feeding at least a part of the oxygen-enriched liquid to an intermediate zone of the main rectification after expansion to said lower pressure, feeding part of said nitrogen-rich liquid to the top zone of the main rectification after expansion to said lower pressure, employing the remainder of said nitrogen-rich liquid as reflux in the high pressure rectification stage, passing a portion of the oxygen-enriched liquid from the bottom zone of the high pressure rectification stage to the top zone of one of said rectification stages to provide additional refluxing liquid for said one of the rectification stages, and withdrawing from an intermediate zone of the main rectification where the argon concentration is high, a fluid comprising mainly oxygen and argon to be further distilled in an auxiliary rectification.

2. A process for the low temperature separation of air to obtain an effluent nitrogen product, an oxygen product and an argon-containing product, which comprises: subjecting air to a two-stage rectification which includes a rectification at a higher rectification pressure to provide nitrogen-rich liquid containing a minimum amount of argon and oxygen-enriched liquid for a main rectification at lower pressure, feeding oxygen-enriched liquid to the main rectification after expansion to said lower pressure, feeding nitrogen-rich liquid to the top zone of the main rectification after expansion to said lower pressure, feeding at least a portion of the oxygen-enriched liquid from the high pressure rectification to the top zone of the main rectification above the nitrogen-rich liquid feed point to effect oxygen-nitrogen equilibrium conditions so as to produce an oxygen pinch in such zone, and withdrawing from an intermediate zone of the main rectification where the argon concentration is high, a fluid comprising mainly oxygen and argon to be further distilled in an auxiliary rectification.

3. A process for the low temperature separation of air to obtain an effluent nitrogen product, an oxygen product and an argon-containing product, which comprises: subjecting air to a two-stage rectification which includes a rectification at a higher rectification pressure to provide nitrogen-rich liquid containing a minimum amount of argon and oxygen-enriched liquid for a main rectification at lower pressure, feeding oxygen-enriched liquid to the main rectification after expansion to said lower pressure, feeding nitrogen-rich liquid to the top zone of the main rectification after expansion to said lower pressure, feeding at least a portion of the oxygen-enriched liquid from the high pressure rectification to the top zone of the main rectification above the nitrogen-rich liquid feed point to effect oxygen-nitrogen equilibrium conditions so as to produce an oxygen pinch in such zone, sensing said pinch by taking a purity measurement below one of the points of feed to the low pressure rectification, maintaining the desired amount of pinch when the nitrogen-rich liquid feed to the low pressure rectification is sub-

stantially constant by varying the oxygen-enriched liquid feed to the top zone to hold the purity analysis at the measuring station within a desired range, and withdrawing from an intermediate zone of the main rectification where the argon concentration is high, a fluid comprising mainly oxygen and argon to be further distilled in an auxiliary rectification.

4. A process for the low temperature separation of air to obtain an effluent nitrogen product, an oxygen product and an argon-containing product, which comprises: subjecting air to a two-stage rectification which includes a high pressure rectification at a high reflux rate to provide nitrogen-rich liquid relatively free of oxygen and argon and oxygen-enriched liquid for a main rectification at lower pressures, feeding oxygen-enriched liquid to the main rectification after expansion to said lower pressure, feeding nitrogen-rich liquid to the top zone of the main rectification after expansion to said lower pressure, feeding at least a portion of the oxygen-enriched liquid from the high pressure rectification to the top zone of the main rectification above the nitrogen-rich liquid feed point to effect oxygen-nitrogen equilibrium conditions so as to produce an oxygen pinch in such zone, and withdrawing from an intermediate zone of the main rectification where the argon concentration is high, a fluid comprising mainly oxygen and argon to be further distilled in an auxiliary rectification.

5. A process for the low temperature separation of air to obtain an effluent nitrogen product, an oxygen product and an argon-containing product, which comprises: subjecting air to a two-stage rectification which includes a rectification at a higher rectification pressure to provide nitrogen-rich liquid and oxygen-enriched liquid for a main rectification at lower pressure, feeding oxygen-enriched liquid to the main rectification after expansion to said lower pressure, feeding nitrogen-rich liquid to the top zone of the main rectification after expansion to said lower pressure, recirculating a portion of the oxygen-enriched liquid in the high pressure rectification to wash down the argon and concomitantly increase the oxygen content of the nitrogen-rich liquid so as to provide a refluxing liquid for the low pressure rectification that has minimum argon content yet sufficient oxygen content to produce an oxygen pinch at the top zone of the low pressure rectification, and withdrawing from an intermediate zone of the main rectification where the argon concentration is high, a fluid comprising mainly oxygen and argon to be further distilled in an auxiliary rectification.

6. A process for the low temperature separation of air to obtain an effluent nitrogen product, an oxygen product and an argon product which comprises: subjecting air to a two-stage rectification which includes a rectification at a higher rectification pressure to provide nitrogen-rich liquid containing a minimum amount of argon and oxygen-enriched liquid for a main rectification at lower pressure, feeding oxygen-enriched liquid to the main rectification after expansion to said lower pressure, feeding nitrogen-rich liquid to the top zone of the main rectification after expansion to said lower pressure, mixing a portion of said oxygen-enriched liquid with the nitrogen-rich liquid of low argon concentration to provide a refluxing liquid for the main rectification that has a sufficient oxygen content to effect oxygen-nitrogen phase equilibrium conditions at the top zone of the main rectification stage so as to produce an oxygen pinch in such zone, withdrawing from a zone of the low pressure rectification where the argon concentration is high, a vapor comprising mainly oxygen and argon, subjecting such withdrawn vapor to an auxiliary rectification by washing with a reflux liquid formed by partial liquefaction of vapor of the auxiliary rectification, producing said latter reflux liquid by a heat exchange between vapor of the auxiliary rectification and oxygen-enriched liquid withdrawn from the high pressure rectification to substantially vaporize such liquid, and passing the oxygen-enriched vapor produced thereby to an inter-

mediate zone of the low pressure rectification below the level of the oxygen-enriched liquid feed point.

7. A process for the separation of air as defined by claim 6, wherein the oxygen-enriched liquid and oxygen-enriched vapor are respectively introduced into the upper part of the low pressure rectification at successively lower levels intermediate the argon take-off and the nitrogen-rich liquid feed point where such fluids will produce auxiliary oxygen pinches for further wash of argon out of the rising vapor.

8. In a process for the low temperature separation of air in which the air is subjected to two stages of rectification to obtain an effluent nitrogen product, an oxygen product and an argon-containing product from an intermediate zone of the second stage and in which the reflux feeds for the second stage in the form of crude oxygen and nitrogen-rich liquid are fed to the second stage at successively higher levels in the upper part thereof, the improvement for increasing the recovery of argon which comprises establishing an oxygen pinch reflux condition in the upper part of the second stage, to provide a zone where the reflux liquid is used chiefly to wash down argon, by running the first stage at a high reflux ratio to minimize the argon content in the nitrogen-rich liquid feed to the second stage and by feeding a portion of the crude oxygen to the second stage at a level at least as high as the nitrogen-rich liquid feed level.

9. A process for the separation of air by low temperature rectification to obtain oxygen, argon, and nitrogen, which comprises: subjecting air to a two-stage rectification wherein nitrogen-rich reflux liquid is introduced into the top zone of the second rectification stage, wherein oxygen-enriched reflux liquid is introduced into an intermediate zone of the second rectification stage and wherein an oxygen product and a nitrogen product are formed, also introducing some oxygen-containing liquid into the second rectification stage above the nitrogen-rich liquid feed point to effect oxygen-nitrogen phase equilibrium conditions and thereby produce an oxygen pinch in the top region of this second stage, and withdrawing from an intermediate zone of the second rectification stage where the argon concentration is high, a fluid comprising mainly oxygen and argon to be further distilled in an auxiliary rectification.

10. A process for the separation of air by low temperature rectification to obtain oxygen, argon, and nitrogen, which comprises: subjecting air to a two-stage rectification wherein nitrogen-rich liquid and oxygen-enriched liquid are produced in the upper and lower regions, respectively, of the high pressure stage and wherein the nitrogen-rich liquid is introduced as reflux into the top zone of the low pressure rectification stage and wherein at least a part of the oxygen-enriched liquid is introduced as reflux into an intermediate zone of the low pressure rectification stage and also wherein the nitrogen-rich reflux liquid is insufficient to produce a completely oxygen-free nitrogen product, passing another part of the oxygen-enriched liquid from the bottom zone of the high pressure rectification stage to the top zone of one of said rectification stages, to provide additional refluxing liquid for said one of the rectification stages, and withdrawing from an intermediate zone of the main rectification where the argon concentration is high, a fluid comprising mainly oxygen and argon to be further distilled in an auxiliary rectification.

11. A process for the separation of air by low temperature rectification to obtain oxygen, argon, and nitrogen, which comprises: subjecting air to a rectification stage wherein nitrogen-rich reflux liquid is introduced into a top zone of said rectification stage, wherein oxygen-enriched reflux liquid containing argon is introduced into an intermediate zone of said rectification stage and wherein an oxygen product and a nitrogen product are formed, introducing some oxygen-containing liquid from a source other than said rectification stage into the nitrogen-rich

liquid at a top zone in said rectification stage to effect oxygen-nitrogen phase equilibrium conditions and thereby produce an oxygen pinch in the top region of said rectification stage, and withdrawing from an intermediate zone of said rectification stage where the argon concentration is high, a fluid comprising mainly oxygen and argon for further distillation in an auxiliary rectification.

12. In a system for the low temperature separation of air to obtain oxygen, argon, and nitrogen, including a low pressure rectifying column and a high pressure rectifying column with means for feeding nitrogen-rich liquid from said high pressure column to a top zone of a low pressure column to serve as reflux therein and means for feeding oxygen-enriched liquid containing argon from the high pressure column to an intermediate zone in the low pressure column and means for withdrawing from an intermediate zone of the low pressure rectifying column where the argon concentration is high, a fluid comprising mainly oxygen and argon to be further distilled in a rectification, the improvement comprising an interconnection between (1) a source of oxygen-enriched liquid at a pressure higher than the pressure in the low pressure rectifying column and (2) the low pressure rectifying column for introducing oxygen-enriched liquid from such source into said top zone of the low pressure rectifying column to effect oxygen-nitrogen phase equilibrium, and thereby an oxygen pinch, in such zone.

13. In a system for the low temperature separation of

air to obtain oxygen, argon, and nitrogen, including a low pressure rectifying column and a high pressure rectifying column with means for feeding nitrogen-rich liquid from said high pressure column to a top zone of a low pressure column to serve as reflux therein and means for feeding oxygen-enriched liquid containing argon from the high pressure column to an intermediate zone in the low pressure column and means for withdrawing from an intermediate zone of the low pressure rectifying column where the argon concentration is high, a fluid comprising mainly oxygen and argon to be further distilled in a rectification, the improvement comprising an interconnection between (1) a zone in the high pressure rectifying column where oxygen-enriched liquid can be withdrawn and (2) the low pressure rectifying column for introducing oxygen-enriched liquid from said high pressure rectifying column into said top zone of the low pressure rectifying column to effect oxygen-nitrogen phase equilibrium, and thereby an oxygen pinch, in such zone.

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