Dec. 9, 1958

C. L. NEWTON

PROCESS FOR SEPARATING A GAS MIXTURE INTO ITS COMPONENTS

2,863,295

Filed July 19, 1955

2 Sheets-Sheet 1

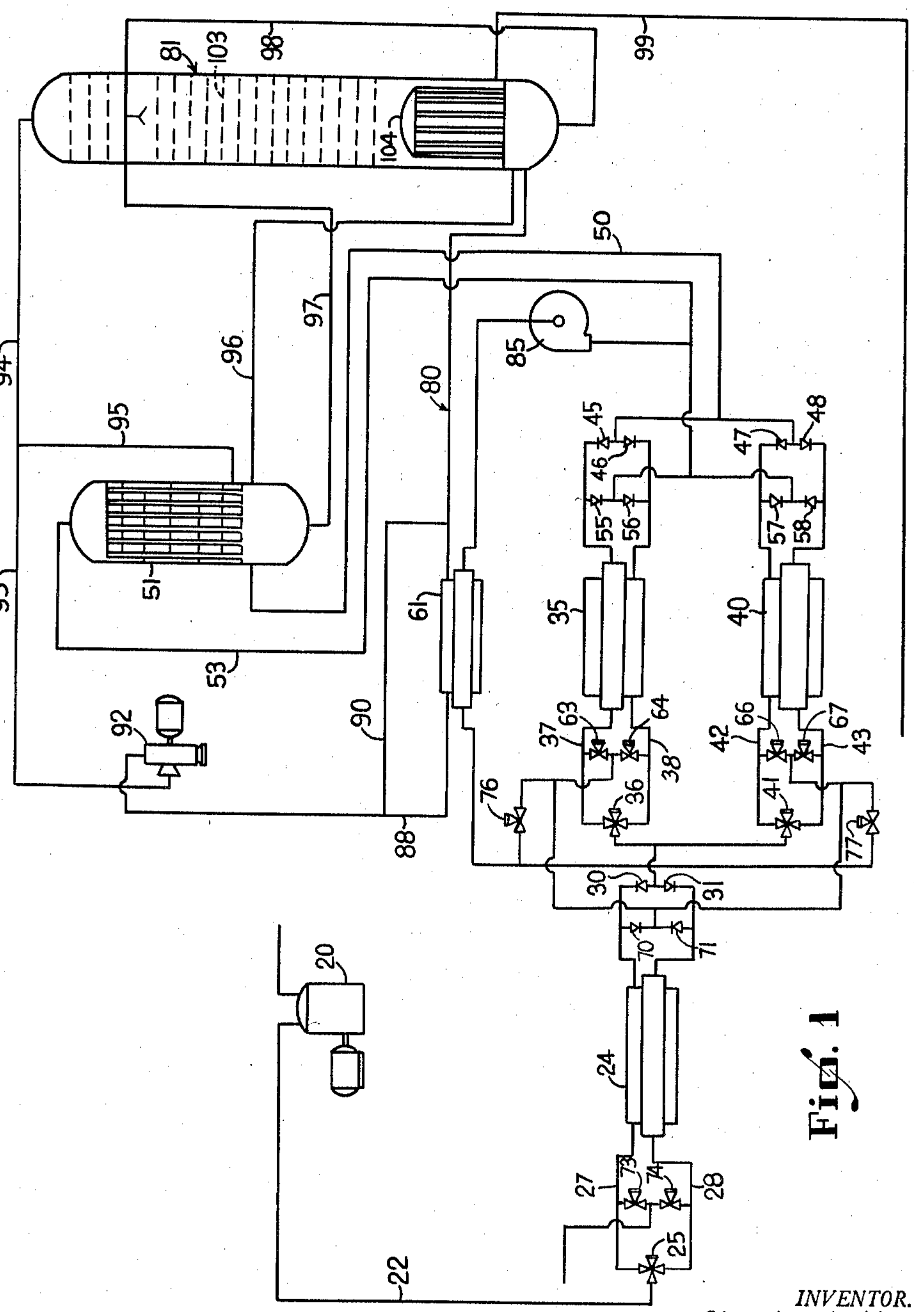

Fig. 1

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O-OPEN
C-CLOSED

Time Minutes	VALVE NUMBER																			
	73	74	70	71	30	31	63	64	66	67	76	77	55	56	57	58	45	46	47	48
0-5	O	C	O	C	C	O	O	C	O	C	O	C	O	C	O	C	C	O	C	O
5-10	O	C	O	C	C	O	C	O	O	C	C	O	C	O	O	C	O	C	C	O
10-15	C	O	C	O	O	C	C	O	C	O	O	C	C	O	C	O	O	C	O	C
15-20	C	O	C	O	O	C	O	C	C	O	C	O	O	C	C	O	C	O	O	C
20-25	O	C	O	C	C	O	O	C	O	C	O	C	O	C	O	C	C	O	C	O

Fig. 2

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PROCESS FOR SEPARATING A GAS MIXTURE INTO ITS COMPONENTS

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9 Claims. (Cl. 62—13)

This invention relates to the separation of gas mixtures and more particularly to a process for continuously effecting such separation and for more efficiently eliminating higher boiling point impurities from the gas mixtures.

In the separation of gas mixtures, such as air, reversing heat exchangers have been employed to remove the undesirable higher boiling impurities such as water vapor and carbon dioxide. A major problem resulting from the use of such reversing exchangers results from the fact that such higher boiling impurities precipitate from the gas mixture, accumulate in the paths of the exchangers, and must therefore be efficiently removed from the paths in order to provide an efficient system. The deposits of higher boiling impurities are removed from the paths of the exchanger by periodically alternating the flow of warm incoming gas mixture and backward returning cold product in any given path through the exchanger. The reversal of flow is effected before the accumulation of higher boiling impurities has become great enough to plug the paths of the exchanger.

In order to achieve efficient operation of systems of this type it is necessary to effectively maintain conditions influencing complete re-evaporation of the higher boiling impurities from the path in which such impurities are deposited. Hence it is necessary to provide sufficient volume of backward returning cold product into which the deposited impurities can be evaporated. Moreover, it is necessary to maintain sufficient vapor pressure at the deposited impurities to effect an efficient evaporation rate of the impurities.

It will be understood that the vapor pressure of the deposited impurities is a function of the temperature and that such impurities are usually removed at a temperature lower than the temperature at which they are deposited. Hence the smaller the temperature difference, the smaller will be the difference between the existing vapor presure of the impurities at the time of their precipitation and re-evaporation.

It is an object of the present invention to provide a novel method which effects such small temperature differences, between the incoming gas mixture and the backward returning cold product, at any region along the paths of the reversing heat-exchanger means utilized in the process.

It is another object of the present invention to provide the above described results in a novel manner whereby a plurality of portions of the stream of backward returning cold product are cyclicaly diverted from the warm ends of a plurality of parallel reversing heat-exchangers. Such diverted portions are then passed through an auxiliary heat-exchanger for effecting temperature control of the diverted portions after which the diverted portions are cyclically united with the backwardly returning cold product entering the cold ends of said plurality of parallel heat-exchangers.

It is another object of the present invention to provide a novel process of the type described adapted to continuously warm cold substantially saturated gas passing

from a fractionating zone subsequent to its passage through the reversing heat-exchangers, and prior to the introduction of such gas to an expansion means whereby said gas is conditioned for isentropic expansion.

Further objects and advantages of the present invention will be apparent from the following description, reference being had to the accompanying drawings wherein a preferred form of embodiment of the invention is clearly shown.

In the drawings:

Figure 1 is a diagram schematically illustrating apparatus for continuously separating a gas mixture into its components, with such apparatus being constructed according to the present invention; and

Figure 2 consists of a table which illustrates the sequence of valve actuation for the valves utilized in the apparatus of Figure 1.

Referring to Figure 1 of the drawing, the incoming atmospheric air first passes through a compressor **20** and then through line **22** to a warm reversing heat-exchanger **24**. A valve **25** serves to alternately connect line **22** with two paths **27** and **28** through the exchanger **24**. The air leaves the warm exchanger **24** through one of the air check valves **30** and **31** after being cooled and partially dried.

The air flow is then split into two portions. One portion of the air flow passes to a cold reversing heat-exchanger **35**, with a reversing valve **36** serving to alternately direct the flow through the paths **37** and **38** of the exchanger **35**. The other portion of the air flow from warm exchanger **24** passes to a second cold reversing heat-exchanger **40**, with a reversing valve **41** serving to alternately direct the flow through the paths **42** and **43** of the exchanger **40**. The cold reversing heat-exchangers **35** and **40** serve to further dry the air and free it of its carbon dioxide content. The dried and carbon dioxide free air leaves the cold exchangers through the air check valves **45** or **46** and **47** or **48** and then passes through the line **50** to a liquifier **51**.

A stream of cold nitrogen waste product leaves the liquifier **51** through a line **53**. The nitrogen stream is divided into two portions, with one portion entering the cold end of the cold reversing exchanger **35** and the other portion entering the cold end of cold reversing exchanger **40**. Nitrogen check valves **55** and **56** serve to alternately direct a portion of the nitrogen flow through paths **37** and **38** of exchanger **35**, and nitrogen check valves **57** and **58** serve to alternately direct a portion of the nitrogen flow through paths **42** and **43** of exchanger **40**.

In passing through the cold exchangers **35** and **40**, the nitrogen is passed in counter current heat-exchange with the air whereby the nitrogen is warmed in giving up its refrigeration to the air passing through the adjacent parallel path in the same exchanger. The nitrogen also serves to pick up the water and carbon dioxide from the path through which it is flowing, with such water and carbon dioxide having been deposited in the path by incoming air prior to reversal of exchanger paths.

The nitrogen then leaves the warm ends of the cold reversing exchangers **35** and **40**, and a portion of the nitrogen is passed directly to the cold end of the warm reversing exchanger **24**. A separate portion of the nitrogen leaving the warm ends of the cold reversing exchangers is diverted through a reheat exchanger **61**.

Two-way reversing valves **63** and **64** serve to alternately connect the paths **37** and **38** of the cold reversing exchanger **35** with the warm exchanger **24** whereby the backward flowing nitrogen passes to the exchanger **24**. In a like manner, two-way reversing valves **66** and **67** serve to alternately connect the paths **42** and **43** of exchanger **40** with the warm exchanger **24**.

At this point it should be pointed out that a portion of the nitrogen flow is continuously diverted through the reheat exchanger **61**, but such diverted portion is cyclically taken from the exchanger paths **37**, **42**, **38** and **43** in the mentioned sequence. Moreover, nitrogen is not diverted from any path of flow until the second half of the period of nitrogen flow. For example, with the nitrogen flowing in path **37** for a period of ten minutes, for the first five minutes of nitrogen flow through path **37**, all of the nitrogen from path **37** will pass through valve **63** and one of check valves, **70** or **71**, and thence through the warm exchanger **24** and out through one of the reversing valves **73** and **74**. After five minutes of nitrogen flow has occurred in path **37**, a two-way reversing valve **76** is opened whereby a portion of the nitrogen flow through path **37** is diverted through the reheat exchanger **61**.

In reheat exchanger **61** the nitrogen portion is passed in counter current heat-exchange with substantially saturated gas from a line **80** which leads from the base of a fractionating column **81**. Line **80** serves to continuously supply substantially saturated gas from the high pressure side of fractionating column **81** to the reheat exchanger **61** wherein the nitrogen stream is cooled and gives up part of its heat content to the saturated gas from the column.

Continuing with the description of the cyclical operation of the cold reheat exchangers **35** and **40**, after nitrogen has flowed through path **37** for an entire ten minute period, check valve **55** is closed and check valve **56** is opened whereby nitrogen flow ceases in path **37** and commences in path **38**. In addition, valve **76** is closed whereby no nitrogen is diverted from path **38** during the first five minutes of nitrogen flow through that path.

At the time valve **76** is closed, valve **77** is opened whereby a portion of the nitrogen flow from path **42** of exchanger **40** is diverted and passed through reheat exchanger **61**. It will be understood that due to the valve timing, at the time valve **77** is opened, nitrogen will have been flowing for five minutes in path **42** whereby the path, and nitrogen flow therethrough, will be clear of the water and carbon dioxide previously deposited by the incoming air.

After the ten minute nitrogen flow terminates for path **42**, portions of the nitrogen flow will next be diverted from paths **38** and **43** in that sequence to complete the cyclical diverting of nitrogen from all four paths in sequence. The cycle is then repeated beginning with the diverting of a portion of the nitrogen flowing through path **37**.

Reference is next made to Figure 2 which consists of a chart setting forth the sequence of valve operation for the various time phases of the cycle operation. It will be noted that the cycle is completed in twenty minutes of operation after which the valving sequence is repeated.

Upon leaving the reheat exchanger **61**, the above described diverted portion of the nitrogen, having been cooled, passes to a centrifugal blower **85** wherein the nitrogen is slightly compressed to make up for the pressure drop occurring during the recirculating cycle of the nitrogen. The recirculated nitrogen from blower **85** is then introduced into line **53** to combine with the nitrogen stream from the liquifier **51**. The combined nitrogen stream then passes through the two cold reversing heat exchangers **35** and **40**.

Referring again to the reheat exchanger **61**, the reheated air stream leaving the reheat exchanger through line **88** is united with the portion of the air which bypasses the reheat exchanger by means of a by-pass line **90**. In passing through the reheat exchanger **61**, the heat content of the air from the base of the fractionating column **81** is increased whereby such air is conditioned for isentropic expansion prior to passing the air through an expansion engine **92**. Hence it is seen that reheat exchanger **61** serves the dual purpose of cooling the recirculated portion of the nitrogen and reheating the substantially saturated cold air stream from the base of the fractionating column.

Line **96** leading from liquefier or condenser **51** to the high pressure side of the column **81** carries gaseous air to provide high pressure condensate for fractionation in the low pressure side of the column **81**.

Line **97** carries the high pressure air condensate from condenser **51** into the low pressure side of the column where it is used as reflux.

Line **98** from the high pressure side of column **81** carries high pressure air condensate into the low pressure side of the column for reflux.

Line **80** carries substantially saturated gas from above the high pressure condensate in the high pressure side of column **81** to reheat exchanger **61** for the purpose previously described.

Line **94** carries low pressure nitrogen-rich gas from the low pressure side of column **81** to condenser **51**.

Line **99** carries the liquid oxygen product from the low pressure side of column **81** to storage means or to a point of use for such product.

As seen in Figure 1, column **81** includes a plurality of fractionating trays **103** and a reboiler condenser unit **104**. The reflux liquid entering the column **81** through lines **97** and **98** passes downwardly across each tray **103** to the low pressure side of reboiler condenser **104**. The liquid is vaporized by heat transfer with the condensing gas on the high pressure side of reboiler condenser **104**. The boil-off gas from reboiler condenser **104** flows upwardly through each of the trays **103** whereby fractionation is accomplished and the rising gas becomes rich in nitrogen. The gas then leaves column **81** through line **94** and enters line **95** along with a low pressure gaseous stream from expansion engine **92** via line **93**. The combined gas in line **95** enters the low pressure side of condenser unit **51**. The high pressure gas from reversing exchangers **35** and **40** flows to the high pressure side of the condenser unit **51** via line **50** where part of such high pressure air is condensed by means of heat transfer with the above mentioned combined gases. The condensed liquid then leaves condenser unit **51** via line **97** and the high pressure gases leave the condenser unit **51** via line **96** as previously described. The low pressure combined gases leave the condenser unit **51**, after being warmed, via line **53** and enter the reversing heat exchangers **35** and **40**.

While the form of embodiment of the present invention as herein disclosed constitutes a preferred form, it is to be understood that other forms might be adopted, all coming within the scope of the claims which follow:

I claim:

1. A process for the separation of a gas mixture into its components comprising passing a first gaseous stream of said mixture through a reversing heat-exchange zone along a cooled path therein progressively decreasing in temperature from a warm end to a cold end; passing a second gaseous stream of cold separation product through said path in the opposite direction after said first stream has ceased to flow for a first time interval sufficient for said second gaseous stream to clear said path of the higher boiling point components deposited in said path by said first gaseous stream; diverting a portion of the second mentioned stream from the warm end of said heat-exchange zone subsequent to said first time interval while continuing to pass said second gaseous stream through said path; passing said diverted portion through another heat exchange zone for a second time interval to effect cooling thereof and combining said diverted portion during said second time interval with the cold separation product entering the cold end of said first heat exchange zone, to effect cooling of said cleared path during said second time interval.

2. A process for the separation of a gas mixture into its components comprising passing a first gaseous stream of said mixture through a reversing heat-exchange zone along a cooled path therein progressively decreasing in

temperature from a warm end to a cold end passing a second gaseous stream of cold separation product through said path in the opposite direction after said first stream has ceased to flow, for a first time interval sufficient for said second gaseous stream to clear said path of the higher boiling point components deposited in said path by said first gaseous stream; diverting a portion of the second mentioned stream from the warm end of said heat-exchange zone subsequent to said first time interval while continuing to pass said second gaseous stream through said path; passing said diverted portion through another heat-exchange zone in counter current heat exchange with a third gaseous stream of cold separation product for a second time interval; and uniting said diverted portion during said second time interval with cold separation product entering the cold end of said first heat exchange zone to effect cooling of said cleared path during said second time interval.

3. A process for the separation of a gas mixture into its components comprising passing a first gaseous stream of said mixture through a reversing heat-exchange zone along a cooled path therein progressively decreasing in temperature from a warm end to a cold end; passing a second gaseous stream of cold separation product through said path in the opposite direction after said first path has ceased to flow for a first time interval sufficient for said second gaseous stream to clear said path of the higher boiling point components deposited in said path by said first gaseous stream; diverting a portion of the second mentioned stream from the warm end of said heat-exchange zone subsequent to said first time interval while continuing to pass said second gaseous stream through said path; passing said diverted portion through another heat-exchange zone for a second time interval to effect cooling thereof; passing said diverted portion through a compressing zone during said second time interval to effect compression thereof; and uniting said diverted portion during said second time interval with said second stream entering the cold end of said first heat-exchange zone to effect cooling of said cleared path during said second time interval.

4. In a process for the separation of a gas mixture into its components, the steps which comprise passing a first gaseous stream of said mixture in one direction of flow through a first reversing heat-exchange zone alternately along two cooled paths therein to effect cooling of the first stream; passing a second gaseous stream of said mixture in one direction of flow through a second reversing heat-exchange zone alternately along two cooled paths therein to effect cooling of the second stream; passing a third gaseous stream, free of the higher boiling point components of said mixture, alternately through said two paths in said first heat-exchange zone in the opposite direction of flow in heat exchange relationship with said first stream, passing a fourth gaseous stream, free of the higher boiling point components of said mixture, alternately through said two paths in said second heat-exchange zone in the opposite direction of flow in heat exchange relationship with said second stream; cyclically diverting a portion of said third stream and a portion of said fourth stream from said paths at the warm ends of said heat-exchange zones; passing said cyclically diverted portions through a third heat-exchange zone to effect cooling of said portions; and uniting said cyclically diverted portions with said third and fourth streams entering the cold ends of said heat-exchange zones to effect cooling of said third and fourth streams.

5. In a process for the separation of a gas mixture into its components, the steps which comprise passing a first gaseous stream of said mixture in one direction of flow through a first reversing heat-exchange zone alternately along two cooled paths therein to effect cooling of the first stream; passing a second gaseous stream of said mixture in one direction of flow through a second reversing heat-exchange zone alternately along two cooled paths therein to effect cooling of the second stream; passing a third gaseous stream, free of the higher boiling point components of said mixture, alternately through said two paths in said first heat-exchange zone in the opposite direction of flow in heat-exchange relationship with said first stream; passing a fourth gaseous stream, free of the higher boiling point components of said mixture, alternately through said two paths in said second heat exchange zone in the opposite direction of flow in heat exchange relationship with said second stream; cyclically diverting a portion of said third stream and a portion of said fourth stream from said paths at the warm ends of said heat-exchange zones, each portion being diverted after its respective path has been freed of substantially all of said higher boiling point components; passing said cyclically diverted portions through a third heat-exchange zone to effect cooling of said portions; and uniting said cyclically diverted portions with said third and fourth streams entering the cold ends of said heat-exchange zones to effect cooling of said third and fourth streams.

6. In a process for the separation of a gas mixture into its components, the steps which comprise passing a first gaseous stream of said mixture in one direction of flow through a first reversing heat-exchange zone alternately along two cooled paths therein to effect cooling of the first stream; passing a second gaseous stream of said mixture in one direction of flow through a second reversing heat-exchange zone alternately along two cooled paths therein to effect cooling of the second stream; passing a third gaseous stream, free of the higher boiling point components of said mixture, alternately through said two paths in said first heat-exchange zone in the opposite direction of flow in heat exchange relationship with said first stream; passing a fourth gaseous stream, free of the higher boiling point components of said mixture, alternately through said two paths in said second heat-exchange zone in the opposite direction of flow in heat-exchange relationship with said second stream; cyclically diverting a portion of said third stream and a portion of said fourth stream from said paths at the warm ends of said heat-exchange zones; cyclically passing each of said diverted portions in heat-exchange relationship with a third gaseous stream of cold output components obtained from at least one of said first and second streams after passage of said first and second streams along said cooled paths to effect cooling of said diverted portions and warming of said third gaseous stream; and uniting said cyclically diverted portions with said third and fourth streams entering the cold ends of said heat-exchange zones to effect cooling of said third and fourth streams.

7. The method of freeing higher boiling point components from a gaseous mixture in a parallel combination of first and second reversing heat exchangers having first and second paths and third and fourth paths, respectively, comprising: routing in one direction portions of a stream of said gaseous mixture in repetitive timed sequence through the second and fourth paths, then the fourth and first paths, then through the first and third paths, and finally through the third and second paths, so that the flow of one portion of said mixture in each path occurs for a predetermined period but the flow of another portion of said mixture begins in another path midway during said period, routing in the opposite direction portions of a cold separation product stream in like repetitive timed sequence through the first and third paths, then through the third and second paths, then through the second and fourth paths, and finally through the fourth and first paths, so that the flow of one portion of said cold separation product in each path occurs for a predetermined period but the flow of another portion of said product begins in a different path midway during such period, diverting separation product successively from the first, third, second and fourth paths after

scavenging, cooling the diverted cold separation product, and finally uniting the diverted cold separation product with said cold separation product stream entering the cold ends of such exchangers.

8. The method of freeing higher boiling point components from a gaseous mixture in a parallel combination of a plurality of reversing heat exchangers each having two paths comprising: routing portions of a stream of said gaseous mixture in one direction in repetitive timed overlapping sequence first through a first series of paths in said heat exchangers and then through a second series of paths in said heat exchangers, so that the flow of one portion of said mixture in any path occurs for a predetermined period and the flows of said mixture in the next succeeding paths begin at intervals equal to a fraction of said period, said fraction having two as its numerator and the number of paths as its denominator, routing portions of a cold separation product stream in the opposite direction in like repetitive timed overlapping sequence first through said second series of paths and then through said first series of paths so that the flow of one portion of said separation product in any path occurs for a predetermined period and the flows of said product in other paths begin at intervals equal to the same fraction of said period, diverting separation product from at least one of said paths after scavenging, cooling the diverted cold separation product, and uniting the diverted cold separation product with said cold separation product stream entering the cold ends of such exchangers.

9. The method of freeing higher boiling point components from a gaseous mixture in a parallel combination of a plurality of reversing heat exchangers each having two paths comprising: routing portions of a stream of said gaseous mixture in one direction in repetitive timed overlapping sequence first through a first series of paths in said heat exchangers and then through a second series of paths in said heat exchangers, so that the flow of one portion of said mixture in any path occurs for a predetermined period and the flows of said mixture in the next succeeding paths begin at intervals equal to a fraction of said period, said fraction having two as its numerator and the number of paths as its denominator, and routing portions of a cold separation product stream in the opposite direction in like repetitive timed overlapping sequence first through said second series of paths and then through said first series of paths so that the flow of one portion of said separation product in any path occurs for a predetermined period and the flows of said product in other paths begin at intervals equal to the same fraction of said period.

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