April 28, 1964 C. J. SCHILLING 3,131,045

METHOD AND APPARATUS FOR FRACTIONATING GASEOUS MIXTURES

Filed May 19, 1958 3 Sheets-Sheet 1

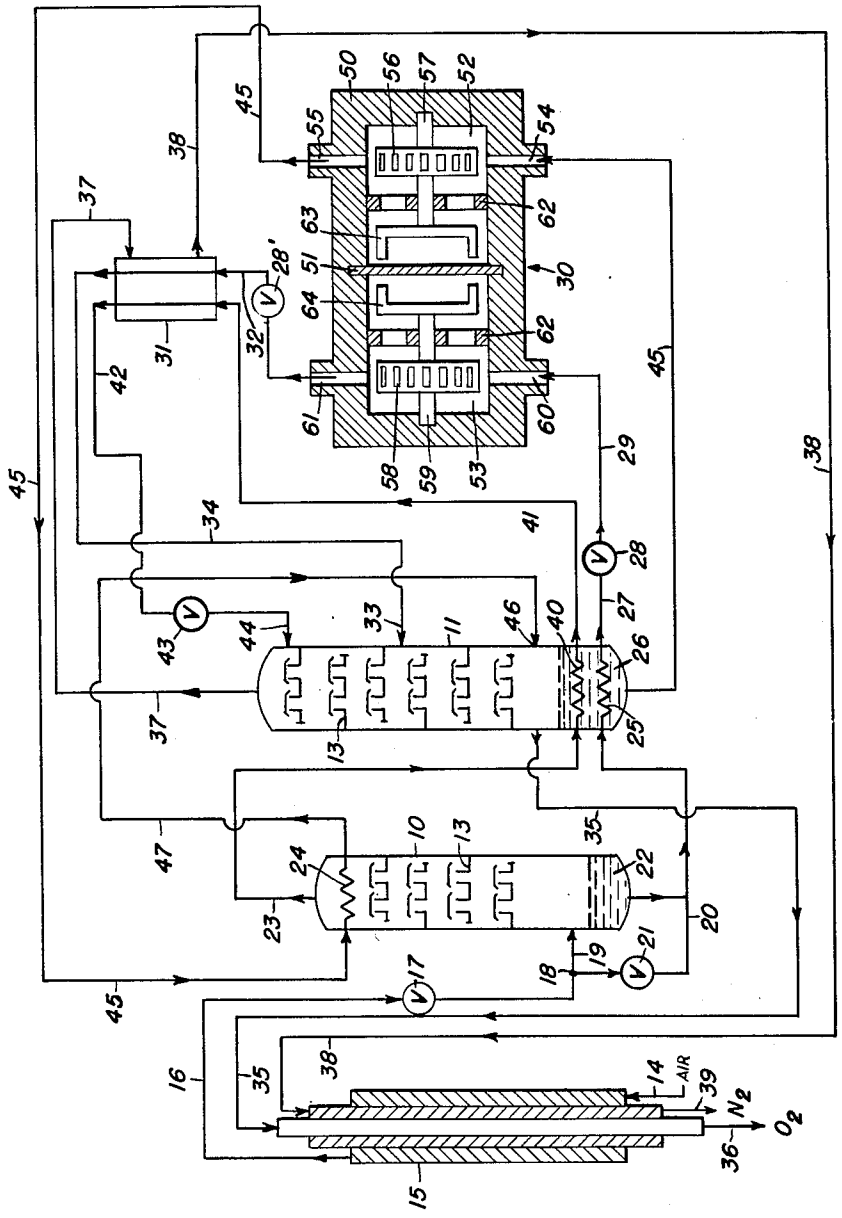


FIG. 1

INVENTOR
CLARENCE J. SCHILLING
BY James J. Shanley
SHANLEY & O'NEIL
ATTORNEYS

April 28, 1964 C. J. SCHILLING 3,131,045

METHOD AND APPARATUS FOR FRACTIONATING GASEOUS MIXTURES

Filed May 19, 1958 3 Sheets-Sheet 2

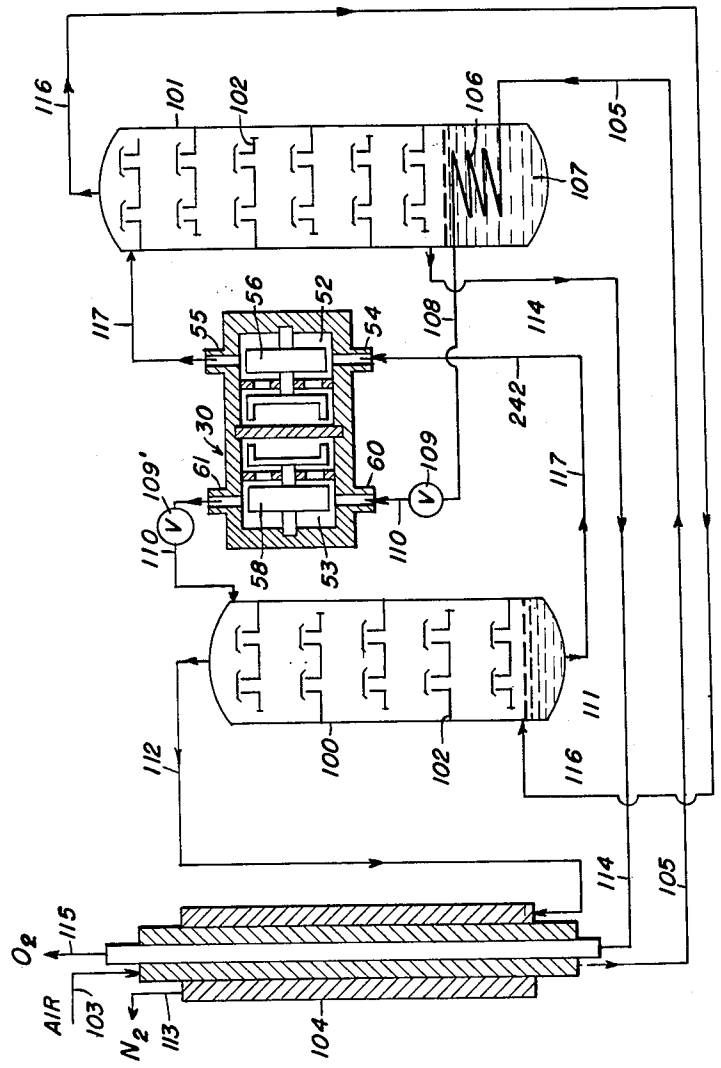


FIG. 3

INVENTOR
CLARENCE J. SCHILLING
BY James J. Shanley
SHANLEY & O'NEIL
ATTORNEYS.

April 28, 1964 C. J. SCHILLING 3,131,045

METHOD AND APPARATUS FOR FRACTIONATING GASEOUS MIXTURES

Filed May 19, 1958 3 Sheets-Sheet 3

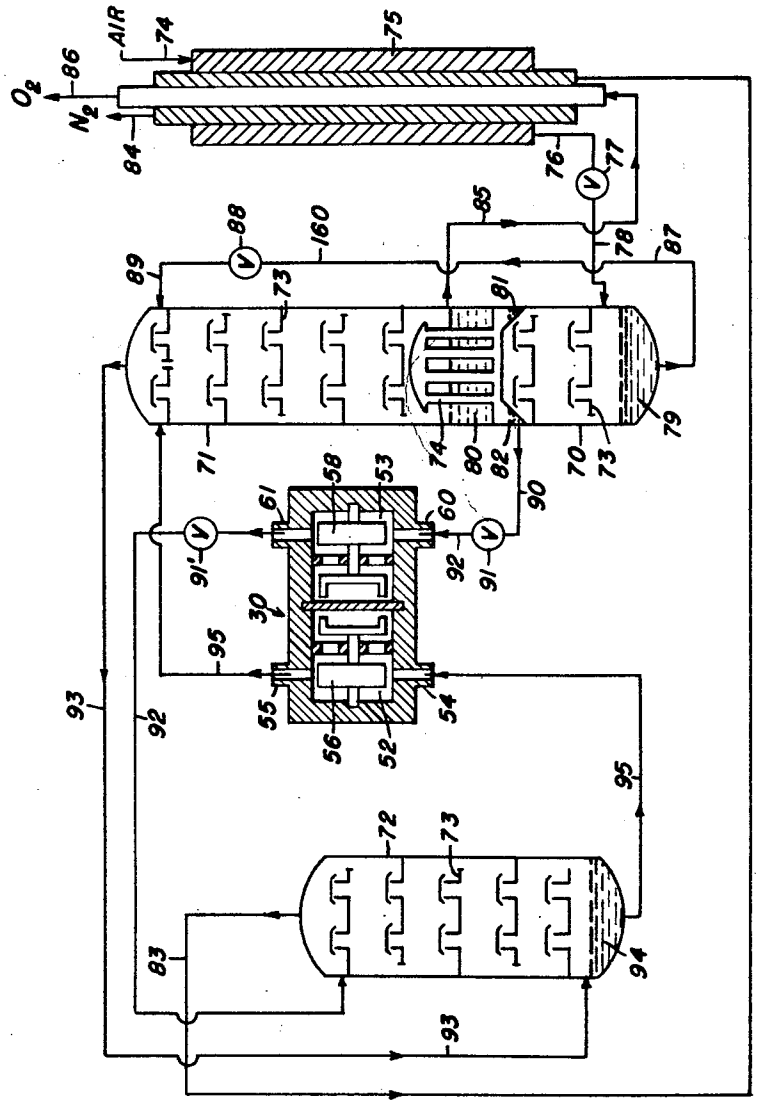


FIG. 2

INVENTOR
CLARENCE J. SCHILLING
BY James J. Shanley
SHANLEY & O'NEIL
ATTORNEYS

United States Patent Office 3,131,045 Patented Apr. 28, 1964

3,131,045
METHOD AND APPARATUS FOR FRACTIONATING GASEOUS MIXTURES

Clarence J. Schilling, Allentown, Pa., assignor, by mesne assignments, to Air Products and Chemicals, Inc., Trexlertown, Pa., a corporation of Delaware

Filed May 19, 1958, Ser. No. 736,374
4 Claims. (Cl. 62—30)

This invention relates to the fractionation of gaseous mixtures and more particularly to methods of and apparatus for fractionating gaseous mixtures by which the overall height of the fractionating equipment is materially reduced and a more compact arrangement is provided without substantially affecting fractionating efficiency.

The separation of many gaseous mixtures, such as the separation of oxygen and nitrogen from air, is ordinarily accomplished in a fractionating zone in the form of a liquid-vapor contact column in which downwardly flowing liquid and upwardly flowing vapor pass in intimate countercurrent relation. The liquid-vapor contact columns are necessarily of substantial height to provide the liquid-vapor contact required for complete fractionating of gaseous mixtures, and the height of the liquid-vapor columns is relatively high as compared to the other components, such as heat exchangers required for the fractionating operation.

Copending application Serial No. 428,079, filed May 6, 1954, now Patent No. 2,913,882, November 24, 1959, which is a continuation-in-part of copending application Serial No. 262,235, filed December 18, 1951, and now abandoned, provides novel methods of and apparatus for fractionating gaseous mixtures, which allows the fractionating column to be divided into separate sections and thus reduce the overall height of the equipment. The copending application discloses fractionating cycles of the single-stage and multiple-stage type in which the fractionating zone or zones of the cycles is divided into sub-fractionating zones by means of separate liquid-vapor contact sections which may be mounted at a common elevation to reduce the overall height of the equipment. Each contact section is provided with a liquid collecting region at its base and means are provided for elevating the liquid material collecting in the base of one contact section to the elevation of the top of another contact section so that the fractionating cycle operates as if the contact sections were mounted one above the other according to conventional practice. The copending application discloses a heat pump for elevating the liquid material, the heat required for this operation being provided by a stream of relatively warmer fluid of the fractionating operation.

The present invention is in the nature of an improvement in the fractionating cycles disclosed in the copending application and provides a novel arrangement for utilizing the kinetic energy of a fluid stream of the fractionating cycle for pumping liquid material from the base of one contact section to the elevation of another contact section of a fractionating cycle which may be of single or multiple-stages.

In general, the present invention provides novel methods of and apparatus for fractionating gaseous mixtures in single-stage or multiple-stage fractionating cycles in which the fractionating zone of the cycles is divided into a plurality of separate fractionating zones or sub-fractionating zones by means of separate liquid-vapor contact sections or columns adapted to be mounted at a common elevation to reduce the overall height of the apparatus. A pump of relatively simple construction designed for handling liquid material is provided for elevating liquid material collecting in the base of one contact section to the elevation of another contact section so that

the liquid material flows through the various contact sections as if the contact sections were mounted one on top of the other according to conventional practice. The liquid material pump is driven by kinetic energy derived from another stream of the cycle, such as a fluid stream under relatively high pressure which is expanded to a lower pressure and introduced into one of the contact sections. The present invention provides a novel liquid transfer means for pumping one stream of liquid material responsively to the kinetic energy of another fluid stream in which the pump and the kinetic energy driving means therefor are mechanically isolated. The transfer means is particularly designed for operation in connection with a low temperature fractionating system, but may be utilized in other fractionating cycles.

The specific features and objects of the invention will appear more fully below from the following detailed description considered with the accompanying drawings which disclose several embodiments of the invention. It is to be expressly understood however that the drawings are designed for purposes of illustration only and not as a definition of the limits of the invention, reference for the latter purpose being had to the appended claims.

In the drawings, in which similar references denote similar elements throughout the several views:

FIG. 1 is a diagrammatic view of a two-stage fractionating cycle incorporating the principles of the present invention;

FIG. 2 is a diagrammatic view of another form of two-stage fractionating cycle constructed in accordance with the principles of the present invention, and

FIG. 3 is a diagrammatic view of a single-stage fractionating cycle incorporating the principles of the present invention.

A two-stage air fractionating cycle embodying the principles of the present invention is disclosed in FIG. 1 comprising a fractionating zone including a high pressure column or section **10** and a low pressure column or section **11**, each having a plurality of bubble plates **13** or other conventional liquid-vapor contact means. The sections **10** and **11** comprise fractionating zones and in a sense they may be considered as sub-fractionating zones. The sections **10** and **11**, being structurally independent may be mounted on a common base to reduce the overall height of the apparatus.

A stream of compressed and cooled gaseous mixture, such as air substantially free from water and carbon dioxide, enters the system by a conduit **14** and is conducted through one passageway of a primary interchanger **15** in which it is cooled by heat interchange with fractionation products. The cooled air then passes through conduit **16** to an expansion valve **17** by which its pressure is reduced with a concomitant drop in its temperature. The expanded air stream then passes through a point of division **18**, and a major portion of the stream flows through a conduit **19** into the base of the high pressure section **10**. The remaining portion of the air stream passes through a conduit **20** to the low pressure section **11** through various steps of heat interchange described hereinafter. A valve **21** is included in conduit **20** to control the percentage of the air stream flowing therethrough.

In the high pressure section **10** that portion of the total air feed introduced by the conduit **19** is separated by fractionation into an oxygen-rich liquid fraction (crude oxygen) which collects in a pool **22** in the base of the section, and a vapor fraction consisting substantially of nitrogen which passes to the top of the section and is delivered therefrom by way of a conduit **23**. A coil **24**, through which a stream of liquid oxygen product from the low pressure section **11** is continually circulated as described below, is located in the top of the high pressure section **10** for condensing a portion of the vapor

fraction to provide wash liquid or liquid reflux for the high pressure fractionating zone.

The oxygen-rich liquid collecting in the base of the high pressure section is mixed with the other portion of the air stream flowing in the conduit 20, and the stream of mixture is cooled and at least partly liquefied upon passing through a coil 25 immersed in a pool 26 of boiling liquid product, i.e., liquid oxygen is the case of fractionation of air, in the base of the low pressure section 11. The stream of air and crude oxygen mixture is then conducted by a conduit 27 to an expansion valve 28, and then by way of a conduit 29 to the input of a liquid transfer device 30, described below. From the output of the transfer device the stream is passed through an expansion valve 28' and then to a passageway of a secondary heat exchanger 31 by way of a conduit 32. The expansion valves 28 and 28' may be employed together to reduce the pressure of the stream substantially to the pressure of the low pressure section 11, however, either of the expansion valves may be used alone with the other expansion valve wide open to provide the desired expansion. The expanded stream of mixture is further cooled to a state of liquefaction in the secondary heat exchanger by heat interchange with a stream of gaseous product from the low pressure section 11 as described below, and the stream of mixture is then introduced in liquid phase into the low pressure section at an intermediate level 33 thereof by way of a conduit 34.

Fractionation of the stream of air and crude oxygen mixture is completed in the low pressure section 11 producing gaseous product nitrogen collecting in the dome of the section and liquid oxygen product collecting in the pool 26 in the base of the section. The product oxygen may be removed in liquid phase in any conventional manner, or in gaseous phase by means of a conduit 35 connected to the section at a point above the level of the pool 26. The conduit 35 conducts a stream of gaseous oxygen product through a passageway of the primary interchanger 15 in heat interchange with the incoming stream of gaseous mixture and leaves the cycle through a conduit 36. A stream of gaseous nitrogen product is conducted from the collecting space or dome of the section 11 to the shell of the secondary interchanger 31 by way of a conduit 37 for heat interchange with the stream of air and crude oxygen mixture as described above, and thence through a conduit 38 to another passageway of the primary interchanger 15 in heat interchange with the incoming gaseous mixture and leaves the heat exchange through a conduit 39 at substantially atmospheric temperature and pressure.

The stream of gaseous nitrogen fraction withdrawn from the high pressure section 10 by the conduit 23 is liquefied upon passing through a coil 40 immersed in the boiling liquid oxygen pool 26. The stream of liquefied nitrogen fraction is conducted by way of a conduit 41 to to passageway of the secondary interchanger 31 for heat interchange with the stream of gaseous product nitrogen. The cooled stream of liquefied nitrogen fraction is then fed through a conduit 42 to an expansion valve 43 where the stream is expanded to the pressure of the low pressure section 11 with a concomitant temperature reduction. The expanded stream is then fed through a conduit 44 into the top of the low pressure section 11 as liquid reflux therefor.

As mentioned above, a stream of liquid oxygen product from the pool 26 is continuously passed through the coil 24 to liquefy a portion of the gaseous nitrogen fraction collecting in the dome of the high pressure section 10 to provide liquid reflux for this section. This is accomplished by withdrawing a stream of liquid product from the pool 26 through a conduit 45 and then elevating the withdrawn stream to the level of the coil 24 and conducting the elevated stream through the coil, the stream of oxygen product following the heat exchange being returned to the low pressure section at a point 46 above the level of the pool 26 by way of a conduit 47. According to the present invention the withdrawn stream of liquid product is elevated to the level of the coil 24 by the liquid transfer device 30.

According to the principles of the present invention the liquid transfer device 30 functions to elevate the stream of liquid product to the level of the coil 24 by means of kinetic energy of another fluid stream of the fractionating operation. As shown, the transfer device includes a cylindrical casing 50 provided with a transverse partition wall 51 of nonmagnetic material dividing the casing into chambers 52 and 53. The chamber 52 is provided with an input port 54 and an output port 55 connected in series with the conduit 45 conducting the stream of liquid product, and houses a liquid pumping unit or impeller 56 supported therein by a rotatably mounted shaft 57. The other chamber 53 houses a turbine wheel or impeller 58 carried by a shaft 59 rotatably mounted in the casing. The chamber 53 is provided with an input port 60 and an output port 61. The shafts 57 and 59 are rotatably supported in recesses in the end walls of the casing and by bearings carried by spiders 62, 62. The inboard ends of the shafts carry cooperating magnetic coupling members 63 and 64 located on opposite sides of the partition member 51. With this arrangement rotation of the turbine impeller 58 produces rotation of the pump impeller 56 through the magnetic attraction of the coupling members 63 and 64 and there exists no mechanical connection through the partition member 51 presenting sealing problems. In the embodiment shown in FIG. 1, the input port 60 is fed with a stream of expanded air and crude oxygen mixture from the conduit 29, and the output port 61 is connected to the conduit 32 leading to the secondary heat exchanger 31. The input port 60 is located relative to the turbine impeller 58 so that kinetic energy of the stream of mixture flowing through the chamber 53 rotates the turbine impeller and imparts rotation to the pump impeller 56 through the magnetic coupling members 63 and 64. Rotation of the pump impeller 56 elevates the stream of liquid product to the level of the coil 24.

In the embodiment of the invention shown in FIG. 2 the high pressure fractionating zone is formed by high pressure column or section 70 and the low pressure fractionating zone is formed by liquid-vapor contact columns or sections 71 and 72. The high pressure section and the low pressure sections are each provided with liquid-vapor contact means such as bubble plates 73. The high pressure section 70 and the low pressure section 71 may be structurally joined together in a conventional manner and separated by a downwardly draining reflux condenser 74, while the low pressure sections 71 and 72 are structurally independent, with the section 72 being adapted to be mounted on a common base with the high pressure section 70, as shown. The sections 70, 71 and 72 each comprise a fractionating zone and the total fractionating zone of the fractionating operation is the combined fractionating zones of the sections. Thus, with respect to the fractionating operation, the section may also be considered as sub-fractionating zones. Moreover, the sections 71 and 72 provide the total low pressure fractionating zone of the fractionating operation, and with respect to the latter zone they may be considered as sub-fractionating zones.

A stream of compressed and cooled gaseous mixture, such as air substantially free from water and carbon dioxide, enters the system by a conduit 74 and is conducted through a passageway of a heat interchanger 75 where it is cooled by heat interchange with relatively cold products of the fractionating operation. The stream of cooled air from the heat interchanger then passes through a conduit 76 to an expansion valve 77 by which its temperature and pressure are further reduced. The expanded air stream then passes by way of conduit 78 into the base of the high pressure section 70. The air feed mixture undergoes a preliminary fractionation in the high pressure section 70, producing a crude oxygen liquid fraction col-

lecting in a pool 79 in the base of the section, and a gaseous nitrogen fraction which flows upwardly and into the passageways of the refluxing condenser 74 and is liquefied by heat exchange with liquid oxygen product collecting in a pool 80 in the base of the low pressure section 71 and surrounding the refluxing condenser. A portion of the liquefied nitrogen fraction flows downwardly into the high pressure section as liquid reflux therefor, while another portion collects in a pool 81 formed by a trough 82.

The fractionation is completed in the low pressure fractionating zone comprising the fractionating zones provided by the sections 71 and 72, producing a liquid oxygen product collecting in the pool 80 at the base of the section 71 and a gaseous nitrogen product collecting in the dome of the section 72. A stream of gaseous nitrogen product is withdrawn from the section 72 through a conduit 83 and conducted through a passageway of the heat exchanger 75 in heat interchange with the incoming gaseous mixture, the nitrogen product leaving the heat exchanger at substantially atmospheric temperature and pressure through a conduit 84. The oxygen product may be withdrawn in liquid phase or in gaseous phase by a conduit 85 communicating with the section 71 at a level above the pool 80 and leading to a passageway of the heat exchanger 75 for heat interchange with the incoming gaseous mixture. The oxygen product leaves the heat exchanger through a conduit 86 as substantially atmospheric temperature and pressure.

A stream of liquid crude oxygen is withdrawn from the base of the high pressure section by way of a conduit 87, expanded to the pressure of the section 71 by an expansion valve 88 and then introduced by way of a conduit 89 into the top of the section 71 as feed for the low pressure fractionating zone. Liquid reflux or wash liquid for the low pressure fractionating zone is obtained by withdrawing through a conduit 90 a stream of liquefied high pressure nitrogen from the pool 82, expanding the withdrawn stream to the pressure of the low pressure zone in expansion valves 91 and 91', which may be employed in combination or alone similarly to the expansion valves 28 and 28' of FIG. 1, and conducting through a conduit 92 the expanded stream of liquefied nitrogen to the top of the section 72. In order to operate the sections 71 and 72 as the low pressure fractionating zone a stream of gaseous mixture collecting in the dome of the section 71 is conducted through a conduit 93 to the bottom of the section 72, and a stream of liquid mixture collecting in a pool 94 in the base of the section 72 is conducted by way of a conduit 95 to the top of the section 71 to provide liquid reflux for the sub-fractionating zone presented by the latter section. A liquid transfer device 30 which may be similar to the liquid transfer device of FIG. 1 is employed for pumping the stream of liquid mixture from the pool 94 to the top of the section 71. The input and output ports 54 and 55 of the pumping chamber 52 are connected in series with the conduit 95 and the kinetic energy of a stream of high pressure fraction from the high pressure zone is employed for operating the pump. The stream of liquid crude oxygen withdrawn from the pool 79 or the stream of liquefied nitrogen withdrawn from the pool 81 may be employed for this purpose. In FIG. 2, the stream of liquefied nitrogen fraction flowing from the pool 81 of the high pressure section to the top of the section 72 is utilized. For this purpose the input port 60 and the output port 61 of the turbine chamber 53 are connected in series with the conduit 92.

It will be understood that the provision of the transfer device 30 and the conduits connecting the sections 71 and 72 allows the sections to operate in the manner similar to operation of conventional multiple-stage column structure in which the section 72 would be mounted above the section 71 in a continuous column construction.

The embodiment of the invention shown in FIG. 3 incorporates the principles of the invention in a single-stage fractionating cycle. As shown, the single-stage cycle includes a fractionating zone comprising liquid-vapor contact columns or sections 100 and 101, each including conventional liquid-vapor contact means such as spaced bubble plates 102. The sections 100 and 101 each comprises a fractionating zone and combine to provide the total fractionating zone of the apparatus and therefore may be considered as sub-fractionating zones. The sections 100 and 101 are structurally independent and may be mounted at a common base elevation to materially reduce the overall height of the apparatus. As will appear more fully from the following description, the column sections are equivalent with respect to fractionating characteristics to a conventional single-stage fractionating cycle of similar capacity in which the fractionating zone comprises a continuous liquid-vapor contact section.

A stream of cooled and compressed gaseous mixture, such as air substantially free from water and carbon dioxide, enters the cycle by a conduit 103 and is conducted through passageway of a heat interchanger 104 in which the air stream is cooled by heat exchange with relatively cold fractionating products. The cooled air then passes through a conduit 105 to a boiling coil 106 immersed in a pool 107 of liquid oxygen product collected in the base of the section 101. The air stream is liquefied and cooled to a lower temperature upon passing through the coil 106 and is then conducted by a conduit 108 to an expansion valve 109 and through conduit 110 to an expansion valve 109'. As in the previous embodiments the expansion valves may be operated together or alone to provide the desired expansion. The expanded liquefied air stream is then introduced through the conduit 110 into the upper end of the section 100. The liquid air flows downwardly in the column section 100, over the bubble plates 102, in intimate contact with upwardly flowing vapor and collects in a pool 111 of oxygen-rich liquid air, or partially fractionated liquid air. The section 100 corresponds to an upper portion of a column section in a conventional single-stage fractionating cycle, and the upwardly flowing vapor collecting in the dome of the section 100 comprises a nitrogen product of the fractionating cycle. A stream of gaseous nitrogen product is withdrawn from the section 100 through a conduit 112 and is conducted through a passageway of the heat exchanger 104 in heat interchange with the incoming stream of gaseous mixture and emerges therefrom by way of conduit 113 at substantially atmospheric temperature and pressure. The section 102 comprises the lower section of a conventional single-stage fractionating cycle and in this section the liquid oxygen product collects in the pool 107 in the base of the section. The oxygen product may be withdrawn in liquid phase, or in gaseous phase by means of a conduit 114 leading from within the section at a level above the pool 107. The conduit 114 conducts a stream of gaseous oxygen product through a passageway of the heat exchanger 104 for heat interchange with the incoming stream of gaseous mixture, the oxygen product stream leaving the heat exchanger through a conduit 115.

In order for the sections 100 and 101 to operate as a conventional single-stage section of continuous construction, a stream of gaseous mixture collecting in the dome of the section 101 is withdrawn and conducted by a conduit 116 to the base of the section 100, while a stream of liquid mixture withdrawn from the pool 111 collecting in the base of the section 100 is conducted through a conduit 117 to the top of the section 101. The latter stream of liquid mixture is pumped to the elevation of the top of the section 101 by a liquid transfer device 30 which may be similar to the liquid transfer devices described above. In this embodiment the input and output ports 54 and 55 of the pumping chamber 52 are connected in series with the conduit 117, and kinetic energy of the stream of liquefied air feed in conduit 110 is employed to provide the pumping force. In particular, the portion of the conduit 110, which is downstream of the expansion valve 109 and upstream of the expansion valve 109', is

connected in series with the input port 60 and the output port 61 of the turbine chamber 53.

There is thus provided by the present invention novel methods of and apparatus for fractionation of gaseous mixtures, including different boiling point components, such as the fractionation of air into oxygen and nitrogen, in which the fractionation is accomplished by compact equipment of materially reduced height without significant sacrifice of fractionating efficiency. The foregoing is accomplished according to the present invention by providing a total fractionating zone made up of a plurality of structurally independent liquid-vapor contact sections or zones. These zones are adapted to be mounted side-by-side at a common base elevation and connected together in such a manner as to provide the liquid-vapor contact for effecting complete fractionation of the gaseous mixture in a manner similar to operation of conventional multiple-stage or single-stage fractionating cycles. The present invention accomplishes the foregoing by utilizing kinetic energy of a fluid stream of the fractionating operation for operating a pumping arrangement to elevate a stream of liquid from the base of one section to the top of another section and thereby provide wash liquid or reflux liquid for the latter section, without materially affecting efficiency of the fractionating operation.

Although several embodiments of the invention have been disclosed and described herein it is to be expressly understood that various changes and substitutions may be made therein without departing from the spirit of the invention as well understood by those skilled in the art. For example, other forms of liquid transfer devices operative responsively to the kinetic energy of a fluid stream of a fractionating cycle may be employed. Reference therefore will be had to the appended claims for a definition of the limits of the invention.

What is claimed is:

1. The method of separating gaseous mixtures by fractionation in a plurality of fractionating zones, comprising continuously withdrawing liquid from one said zone, passing liquefied gas from a region of relatively high pressure to said one zone at a lower pressure while causing the liquefied gas to act on a displaceable member to move the member, expanding the liquefied gas through an expansion device in series with the member, continuously utilizing kinetic energy expended in moving the member to elevate the withdrawn liquid above the point of withdrawal, and utilizing the elevated liquid to provide wash liquid in a said zone other than said one zone.

2. The method of separating gaseous mixtures by fractionation in a plurality of fractionating zones, comprising continuously withdrawing liquid from one said zone, passing liquefied gas from a region of relatively high pressure to said one zone at a lower pressure while causing the liquefied gas to act on a displaceable member to move the member, expanding the liquefied gas through an adiabatic expansion device downstream of the member, continuously utilizing kinetic energy expended in moving the member to elevate the withdrawn liquid above the point of withdrawal, and utilizing the elevated liquid to provide wash liquid in a said zone other than said one zone.

3. Apparatus for separating gaseous mixtures by fractionation in a plurality of fractionating zones, comprising means for continuously withdrawing liquid from one said zone, means for passing liquefied gas from a region of relatively high pressure to said one zone at lower pressure while causing the liquefied gas to act on a displaceable member to move the member, an expansion device in series with the member for expanding the liquefied gas, pump means for continuously utilizing kinetic energy expended in moving the member to elevate the withdrawn liquid above the point of withdrawal, and means for utilizing the elevated liquid to provide wash liquid in a said zone other than said one zone.

4. Apparatus for separating gaseous mixtures by fractionation in a plurality of fractionating zones, comprising means for continuously withdrawing liquid from one said zone, means for passing liquefied gas from a region of relatively high pressure to said one zone at lower pressure while causing the liquefied gas to act on a displaceable member to move the member, an adiabatic expansion device downstream from said member, pump means for continuously utilizing kinetic energy expended in moving the member to elevate the withdrawn liquid above the point of withdrawal, and means for utilizing the elevated liquid to provide wash liquid in a said zone other than said one zone.

References Cited in the file of this patent

UNITED STATES PATENTS

1,063,168	Whitney	May 27, 1913
2,252,775	Lichtenstein	Aug. 19, 1941
2,552,451	Patterson	May 8, 1951
2,595,284	Mullins	May 6, 1952
2,608,070	Kapitza	Aug. 26, 1952
2,650,482	Lobo	Sept. 1, 1953
2,667,044	Collins	Jan. 26, 1954
2,675,884	Deanesly	Apr. 20, 1954
2,700,282	Roberts	Jan. 25, 1955
2,728,205	Becker	Dec. 27, 1955
2,861,432	Haselden	Nov. 25, 1958
2,913,882	Schilling	Nov. 24, 1959
2,975,606	Karwat	Mar. 21, 1961

FOREIGN PATENTS

849,850	Germany	Sept. 18, 1952
64,004	France	May 11, 1955

(Third addition to No. 1,024,585.)