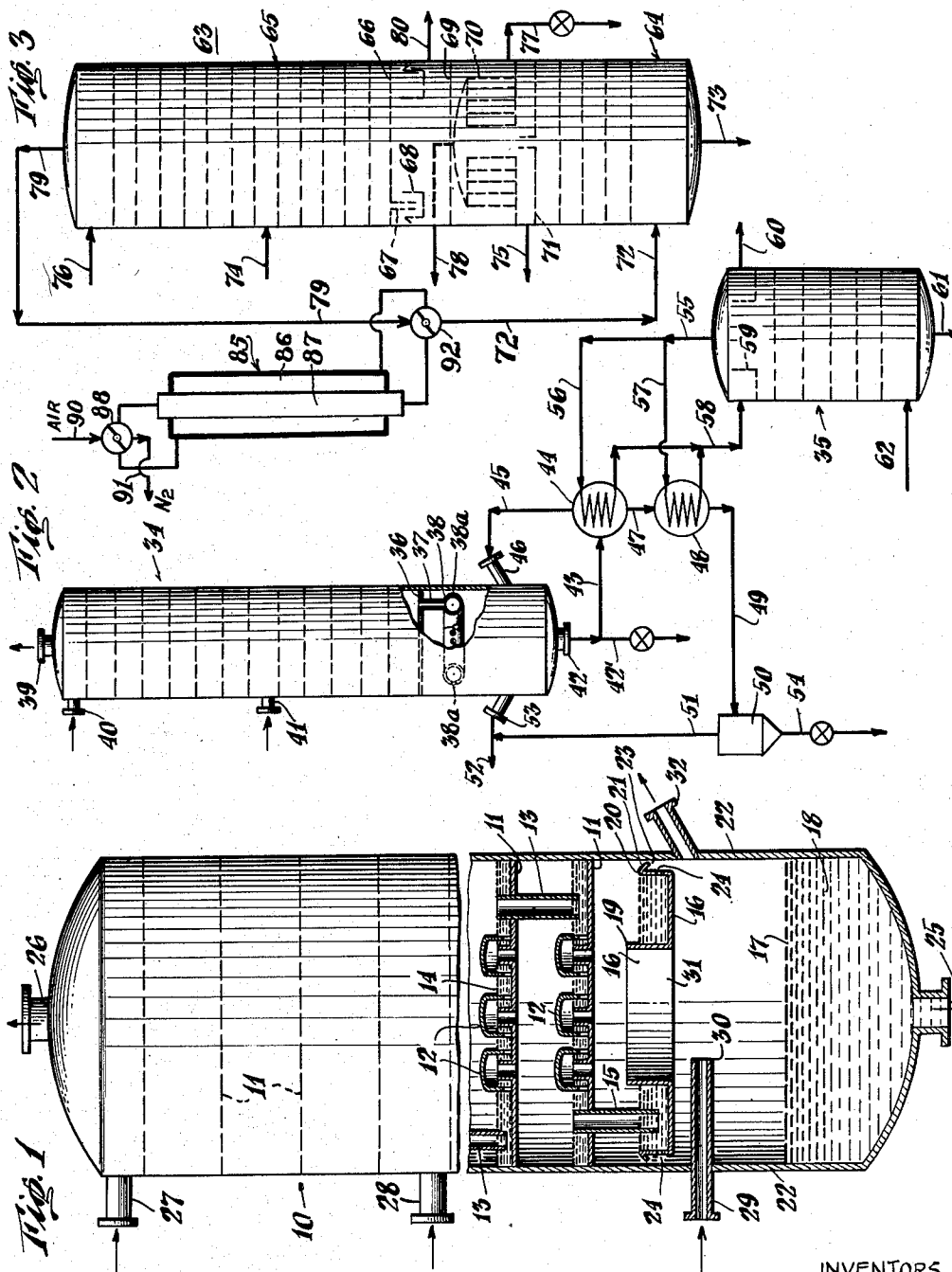


May 1, 1951

E. G. ENGEL ET AL
AIR FRACTIONATING COLUMN AND PROCESS OF
OPERATING SAME
Filed Dec. 29, 1947

2,550,679



INVENTORS
Edward G. Engel
John A. Hufnagel
BY
Benjamin Sweedler
ATTORNEY

UNITED STATES PATENT OFFICE

2,550,679

AIR FRACTIONATING COLUMN AND
PROCESS OF OPERATING SAME

Edward G. Engel, Roselle, N. J., and John A. Hufnagel, Mineola, N. Y., assignors to Hydrocarbon Research, Inc., New York, N. Y., a corporation of New Jersey

Application December 29, 1947, Serial No. 794,260

10 Claims. (Cl. 62-122)

1

2

This invention relates to the production of oxygen by the liquefaction and rectification of air, and more particularly to the construction and operation of the rectification column or system.

In the liquefaction and rectification of air it is common to employ a rectification column in which a body of liquid oxygen is boiled, the vapors thus produced rising up through the column countercurrent to a descending stream of reflux liquid, the rising vapors removing from the reflux liquid nitrogen and other relatively low boiling constituents contained therein and the oxygen vapors being condensed resulting in the production of a product oxygen of desired purity which is withdrawn as product from the column. Air thus subjected to liquefaction and rectification invariably contains acetylene, usually in minute amounts of the order of a few parts per million. It has been found, however, that while the acetylene content of the air is so small as to have heretofore been considered insignificant, in the continued operation of the rectification system the acetylene tends to accumulate and build up in the body of liquid oxygen reboiled to produce the oxygen vapors which are rectified by flow in contact with the liquid reflux. To avoid the acetylene concentration reaching a point where an explosive mixture would result, it has been suggested to withdraw continuously or periodically from the base of the body of liquid oxygen subjected to reboiling a purge stream of this oxygen in order to prevent the accumulation of acetylene in the body of liquid oxygen to a dangerous concentration. One proposal calls for passing this purge oxygen stream downwardly through a heat exchanger wherein the oxygen along with the acetylene contained therein is vaporized, allegedly without danger of an explosion. Observation of this precaution of withdrawing a purge stream of oxygen, however, does not completely eliminate the danger of violent explosions taking place.

It is an object of this invention to provide a process of operating a rectification column which entirely eliminates danger of explosions taking place due to accumulation of acetylene in the rectification system.

Another object is to provide an improved and safer design of rectification column.

Other objects and advantages of this invention will be apparent from the following detailed description thereof.

We have found that in the operation of the rectification system surges of the level of the

body of liquid oxygen maintained in the rectification column inevitably take place. Thus, for example, small surges take place whenever the reversing valves are operated, usually every three minutes, to cause reversal of flow of the rectification product or products and air through their respective flow paths in the heat exchangers, as is customary practice. In those systems in which the refrigeration necessary to compensate for enthalpy losses and heat leaks into the system is provided by expanding a portion of the relatively high pressure air and introducing the expanded air into the rectification column, such surges, which at times are quite extensive, occur due to fluctuations in the operation of the expander. Such surges of liquid level may occur for other reasons, such as fluctuations in the volume of air supplied to the system, etc. Such surges, we have found, cause precipitation of acetylene on the walls of the column, which precipitate tends to build up until it reaches an amount such that an explosion may occur due to the reaction between the oxygen and the acetylene deposited on the walls.

In accordance with this invention the side walls of the rectification column just above the portion in which the body of liquid oxygen is maintained are continuously wetted or washed with reflux oxygen to dissolve or prevent the accumulation of acetylene on these walls.

In accordance with a preferred embodiment of this invention, in addition to the above step, a stream of oxygen is continuously withdrawn from the base of the body of liquid oxygen subjected to reboiling. Preferably the major portion, say about 90%, of this stream is vaporized and the vapors returned to the column where they flow countercurrent to the descending reflux liquid. The remaining minor portion of the stream is passed through a second reboiler where all but a small proportion, say about 1% of the original stream, is vaporized, the vapors thus produced being returned to the column or admixed with the product oxygen stream withdrawn from the column. The residual unvaporized oxygen contains the acetylene and is suitably disposed of, for example, it may be wasted as by introduction into the effluent rectification product stream such as the nitrogen stream withdrawn from the system, or may be introduced into the product oxygen stream passed in heat exchange relation with the incoming air, so that the acetylene is vaporized. By purging from the system a small amount of liquid oxygen continuously or periodically withdrawn from the base of the body of

3

liquid oxygen subjected to reboiling, accumulation of acetylene within this body of oxygen such as to form an explosive mixture is prevented. Also, as noted above, by continuously wetting or washing with liquid oxygen the walls above the body of liquid oxygen subjected to reboiling, accumulation of solidified acetylene on these walls such as to constitute a serious hazard of explosion is prevented.

In the accompanying drawing forming a part of this specification and showing, for purposes of exemplification, preferred forms of this invention without limiting the claimed invention to such illustrative instances:

Figure 1 is a composite fragmentary view, the upper portion being a side elevation and the lower portion a vertical section of a fractionating column embodying this invention;

Figure 2 is a diagrammatic view of a rectification system comprising two columns, one a high pressure and the other a low pressure column, embodying our invention; and

Figure 3 is a diagrammatic elevational view of a rectification system comprising a two-stage rectification column embodying our invention in association with a reversing heat exchanger.

In Figure 1, 10 indicates a rectification column provided as usual with rectification plates 11 of the bubble cap or other desired type. Plates 11 shown in the drawing are provided with bubble caps 12 and downcomers 13 for maintaining a body 14 of liquid reflux at the desired level on each of the plates 11. A downcomer 15 leading from the lowermost plate 11 in the column 10, in accordance with this invention, leads into an annular trough or other container 16 suitably disposed in the base portion of the column above the level 17 of the body of liquid oxygen 18 subjected to reboiling. Trough 16 is suitably mounted in column 10 in the relative position shown in Figure 1 above the body of liquid oxygen 18 and just below lowermost plate 11. It comprises an inner wall 19 of a height greater than that of outer wall 20 insuring that liquid reflux overflows the depending lip 21 spaced from inner walls 22 of the column 10 to provide annular clearance 23 through which clearance liquid from trough 16 flows along the side walls 22 on to the body of liquid 18. If desired, side wall 20 is provided with a series of closely spaced openings or ports 24 spaced at suitable intervals circumferentially of this side wall through which liquid within the annular trough 16 is projected on to the side walls 22. The liquid flowing over the lip 21 through the clearance 23 and through ports 24 continually wets and washes the side walls 22 throughout the full circumferential area thereof, this liquid flowing on to the body of liquid oxygen 18.

Column 10 is provided with a base outlet port 25 through which liquid oxygen from the body 18 flows continuously to a heat exchanger as hereinafter more fully described. This column is provided, as customary, with a top nitrogen exit port 26, an inlet 27 for addition of liquid nitrogen reflux at the top of the column and an inlet 28 disposed at an intermediate point in the column for the introduction of liquid crude oxygen (containing approximately 40% oxygen) which is fractionated as it flows down through the column from plate to plate. A port 29 having its inlet terminal 30 extending well beyond the inner wall 22 is positioned below trough 16, the end 30 desirably being disposed so that it occurs beneath cylindrical opening 31 in the an-

4

nular trough 16. Thus, oxygen vapors introduced through port 29 rise through opening 31 and are introduced into the bottom of the column 10 at a point spaced remotely from the inner walls 22 having the film of liquid oxygen flowing thereover so that the rising vapor stream entering through port 29 does not entrain liquid flowing from trough 16 along the side wall 22 into the body of liquid oxygen 18.

An exit port 32 extends in an upward direction and leads from side wall 22 just below annular trough 16. Product gaseous oxygen may be withdrawn through this port. By having this port inclined in an upward direction, any liquid oxygen entrained in the gas stream flowing through port 32 separates out in this port, flows downwardly along the walls of the port and is thus returned to the column 10. It will be understood that the construction of ports 29 and 32 may be varied; thus, instead of an inclined exit port 32, an exit port similar to port 29 may be used in which the inlet to this port may be disposed spaced remotely from the side walls 22, or instead of a horizontally extending inlet port 29, an upwardly inclined inlet port corresponding to the exit port 32 may be used. This construction is shown in Figure 2.

The rectification system of Figure 2 comprises two columns 34, 35. Column 35 desirably is operated at a pressure of from about 60 to about 100 pounds per square inch gauge, and column 34 at a pressure of from about 5 to about 12 pounds per square inch gauge; column 35 is therefore the high pressure column and 34 is the low pressure column. Both columns, as customary, are provided with rectification plates of the bubble cap or other desired type and, in accordance with this invention, column 34 has leading from lowermost plate 36 a downcomer 37 extending into an annular tubular ring 38 provided with ports 38a or perforations. Hence the reflux liquid oxygen flows from the lowermost plate 36 through the downcomer 37 into the annular ring 38, thence through the ports or perforations in this ring onto the inner side walls of column 34 so as to continuously wet the entire area of the inner side walls below ring 38, the liquid oxygen from the side walls flowing into the body of liquid oxygen maintained at the base of column 34.

As customary, column 34 has a top nitrogen outlet 39, liquid nitrogen reflux inlet 40 and a crude oxygen reflux inlet 41. The base of the column is provided with an outlet 42 through which a portion of the liquid oxygen from the body thereof maintained in the column flows continuously through a line 43 into a heat exchanger 44. Outlet 42 communicates with a valve controlled line 42'; by opening the valve in line 42' the body of liquid oxygen normally maintained at the base of column 34 may be removed therefrom. Vapors generated in heat exchanger 44 flow through line 45 into inlet port 46 leading into the base portion of column 34 just above the level of the body of liquid oxygen maintained therein. From heat exchanger 44 a line 47 leads to a second heat exchanger 48 which is provided with a line 49 leading to a centrifugal or other separator 50. A vapor line 51 leads from this separator 50 to product gaseous oxygen line 52 leading from exit port 53 disposed at the base of column 34 just above the level of the body of liquid oxygen therein. A valve controlled liquid discharge line 54 leads from the separator 50.

A nitrogen line 55 leads from the top of column

5

35. This line is provided with two branches 56 and 57. Branch 56 passes through heat exchanger 44 and leads into nitrogen return line 58 leading into top tray 59 of the column 35. Branch 57 passes through exchanger 48 and leads into the nitrogen return line 58. Thus the exchangers 44 and 48 function as a pair of reboilers for the liquid oxygen continuously withdrawn through the base port 42.

Column 35, as customary, is provided with a nitrogen line 60 leading from the top tray 59; this line passes through a suitable exchanger (not shown), has an expansion valve therein and communicates with inlet 40 of column 34. The base of column 35 is provided with a crude oxygen exit line 61 which passes through a suitable exchanger (not shown), is provided with an expansion valve and communicates with the crude oxygen reflux inlet 41. An air inlet line 62 leads into the base portion of column 35.

In operation the major portion, say about 75%, of the oxygen flowing through port 42 is vaporized by the nitrogen flowing in indirect heat exchange relation through exchanger 44, the vapors of oxygen thus generated flowing through line 45, port 46 into and up through column 34. The residual liquid oxygen, say about 25%, flows through line 47 into exchanger 48 where substantially all, say 24% of the oxygen originally withdrawn through port 42, is vaporized by the nitrogen flowing in indirect heat exchange relation through exchanger 48. The mixture of oxygen vapor and liquid thus produced flows into separator 50 through line 49, the vapors leaving separator 50 through line 51 and entering the product oxygen stream 52 leading from column 34. The liquid oxygen entering the separator 50 which contains the acetylene in solution and in suspension is withdrawn through line 54. By continuously removing a stream of liquid oxygen, as hereinabove described, and purging a small proportion of this stream, say 1%, in unvaporized form, the concentration of acetylene within the body of liquid oxygen in column 34 is maintained below that at which there is any danger of a violent explosion taking place. By continuously washing the side walls of column 34 below ring 38 and above the body of liquid oxygen, accumulation of acetylene on these side walls is prevented.

The rectification system of Figure 3 comprises a two-stage column 63, lower section 64 of which is operated at a pressure of about 60 pounds to about 100 pounds gauge, and the upper section 65 of which is operated at a pressure of from about 5 pounds to about 12 pounds gauge. This column, as customary, is provided with rectification plates of the bubble cap or other desired type. In accordance with this invention, lowermost plate 66 of section 65 is provided with a downcomer 67 leading into an annular trough 68 of the same type as trough 16 hereinabove described.

A body of liquid oxygen is maintained in section 65 at a height indicated by line 69 above condenser-reboiler 70 which has a liquid collecting shelf 71 disposed immediately therebelow for collecting liquid nitrogen. As customary, section 64 is provided with an air inlet line 72, crude oxygen withdrawal line 73 which has an expansion valve therein and communicates through a heat exchanger (not shown) with oxygen reflux line 74 and a nitrogen withdrawal line 75 leading from collecting shelf 71 through a heat exchanger (not shown), provided with a reducing valve and lead-

6

ing into the low pressure section 65 at 76. A valve controlled oxygen withdrawal line 77 leads from the base of section 65. A vent line 78 leads from the condenser 70 through which line a minor portion of the nitrogen may be vented to remove incondensable gases. A nitrogen line 79 leads from the top of low pressure section 65 to reversing heat exchanger 85. Line 80 serves for the withdrawal of product gaseous oxygen from above the oxygen liquid level 69. Reversing heat exchanger 85 is provided with flow paths 86 and 87 through which alternately streams of air and nitrogen flow. Reversing valve 88 has an air supply line 90 and a nitrogen vent line 91 and reversing valve 92 at the opposite end of exchanger 85 is connected to line 72 which feeds the chilled air to section 64 of column 63 and is connected to line 79 to receive nitrogen from section 65 of column 63. For simplicity, other conventional heat exchangers usually interposed in lines 72 and 79 between the reversing exchanger 85 and column 63 have been omitted.

In the operation of Figure 3, condenser 70 operates as a reboiler to effect vaporization of a portion of the liquid oxygen in section 65, which vapors rise upwardly through section 65 where they are fractionated by flowing countercurrent to the descending liquid reflux and through the bodies of liquid reflux on each plate. Liquid oxygen reflux flows from plate to plate to the lowermost plate 66, then through downcomer 67 to the annular trough 68. From this trough liquid oxygen overflows continuously along the side walls of section 65 wetting these walls and preventing accumulation of acetylene thereon due to surges of level 69 of the body of oxygen or for other reasons. Oxygen is continuously or periodically withdrawn from the body of liquid oxygen in which condenser 70 is immersed through line 77 and may be passed through the usual heat exchangers in indirect heat exchange relation with the incoming air, whereby the oxygen and the acetylene contained therein vaporized. Thus, an accumulation of acetylene on the side walls above the body of liquid oxygen subjected to reboiling and within this body of oxygen to a concentration which would create an explosion hazard is prevented.

It will be noted this invention provides a process of operating a rectification column which process entirely eliminates danger of explosions taking place due to accumulation of acetylene in the rectification system, and therefore provides an improved and safer design of rectification column.

Since certain changes in carrying out the above process and certain modifications in the rectification column which embody the invention may be made without departing from the scope of this invention, it is intended that all matter contained in the above description or shown in the accompanying drawing shall be interpreted as illustrative and not in a limiting sense. Thus, for example, the invention comprehends a rectification column corresponding to the high pressure unit 35 of Figure 2 in which a trough, closed ring provided with ports or other container is provided for producing a continuously flowing stream of liquid reflux rectification product over the inner walls of this column above the body of crude liquid oxygen therein to prevent accumulation of acetylene on these inner walls.

The term "oxygen" is used in the claims in a comprehensive sense and is intended to include relatively pure oxygen, as well as oxygen con-

taining impurities usually associated therewith, such as the crude oxygen (containing up to about 40% oxygen) produced in the high pressure section or column of the modifications of Figures 2 and 3 or produced by processes involving a single rectification step or column.

What is claimed is:

1. In a process of producing oxygen by the liquefaction and rectification of air involving the maintenance in a vessel of a body of liquid oxygen containing acetylene and the boiling of said body to produce vapors passed in countercurrent flow to a descending stream of reflux liquid oxygen, the improvement which comprises continuously wetting with said reflux liquid all of the walls of the vessel in the region where the surface of said body of liquid oxygen containing acetylene contacts said walls to prevent accumulation of acetylene on said walls.

2. In a process of producing oxygen by the liquefaction and rectification of air involving the maintenance in a vessel of a body of liquid oxygen containing acetylene and the boiling of said body to produce vapors passed in countercurrent flow to a descending stream of reflux liquid oxygen, the improvement which comprises continuously wetting with said reflux liquid all of the walls of the vessel in the region where the surface of said body of liquid oxygen containing acetylene contacts said walls to prevent accumulation of acetylene on said walls and removing liquid oxygen containing acetylene from said body of oxygen to prevent the concentration of acetylene in said body of oxygen reaching the point such as to form an explosive mixture.

3. In a process of producing oxygen by the liquefaction and rectification of air involving the maintenance of a body of liquid oxygen in a rectification column at a pressure of about 5 to about 12 pounds per square inch gauge, subjecting a portion of said body of liquid oxygen to vaporization and passing the resultant oxygen vapors in countercurrent flow to descending reflux liquid oxygen, the improvement which comprises collecting the reflux liquid oxygen at a point above said body of oxygen, and causing the collected oxygen to flow on to said body by flowing in contact with the walls of said column above the said body of liquid oxygen and below said point of collection so as to completely wet the entire area of said walls above said body and below said point of collection.

4. The process defined in claim 3 in which a stream of liquid oxygen is continuously removed from the said body of liquid oxygen, a major portion of said stream is vaporized and the vapors thus produced introduced into the column to provide said vapors flowing in countercurrent flow to the descending reflux liquid, substantially all of the remainder of said oxygen stream is continuously vaporized except for a small portion containing acetylene, and the said small portion containing acetylene is continuously removed.

5. A fractionating column having at its base a chamber adapted to contain a body of liquid, a series of fractionating plates disposed in spaced relationship above said chamber, and an annular collecting container for receiving reflux liquid from said lowermost plate positioned at the top

of said chamber and below said lowermost plate, said container being provided on its outer wall with a plurality of closely spaced openings whereby liquid flowing through said openings passes into contact with the inner walls of said column and flows downwardly therealong into said chamber.

6. A fractionating column as defined in claim 5 in which the container is a closed annular ring.

7. A fractionating column as defined in claim 5 in which the container is a trough open at its top and having said closely spaced openings in a side wall thereof opposite the inner wall of said column.

8. In a process of producing oxygen by the liquefaction and rectification of air involving the periodic reversal of flow of air and a rectification product through their respective flow paths in a heat exchange zone so that the air recovers the cold content of the rectification product, the maintenance in a rectification column of a body of liquid containing oxygen, vapors from which body of liquid are passed in countercurrent flow to descending reflux liquid containing oxygen and in which column surges of the level of the body of liquid containing oxygen take place due in part to said reversal of flow, which surges cause precipitation of acetylene on the walls of said rectification column just above said body of liquid containing oxygen to take place, the improvement which comprises continuously wetting the said walls of the rectification column with said reflux liquid to prevent accumulation of acetylene on said walls.

9. A fractionating column comprising a chamber at the base thereof adapted to contain a body of liquid, a series of fractionating plates disposed in spaced relationship above said chamber, a container for receiving liquid from said plates and openings associated with said container for feeding said liquid on to all of the inner walls of said chamber in the region where the surface of said body of liquid contacts said inner walls to wash down said inner walls with said liquid throughout the operation of said column.

10. In a process of fractionating a gaseous mixture by the liquefaction and rectification of said gaseous mixture involving the maintenance in a vessel of a body of liquefied gas containing a solidifiable impurity in solution and the boiling of said body to produce vapors passed in countercurrent flow to a descending stream of reflux liquefied gas, the improvement which comprises continuously wetting with said reflux all of the walls of the vessel in the region where the surface of said body of liquefied gas containing said impurity in solution contacts said walls to prevent accumulation of said impurity in solidified form on said walls.

EDWARD G. ENGEL.
JOHN A. HUFNAGEL.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,501,415	Lafferty	July 15, 1924