

March 16, 1954

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2,672,031

FRACTIONATION OF GAS MIXTURES

Original Filed June 27, 1945

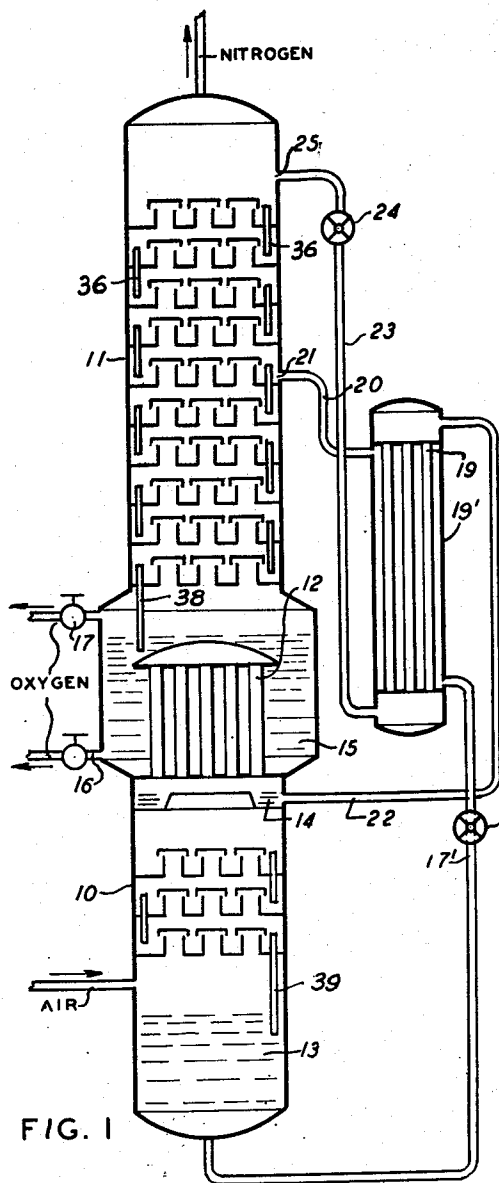


FIG. 1

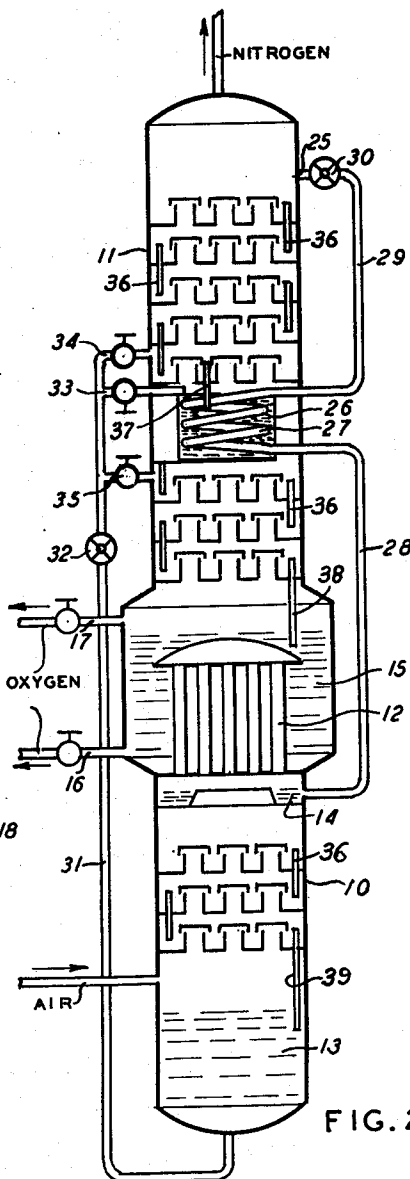


FIG. 2

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FRACTIONATION OF GAS MIXTURES

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Continuation of application Serial No. 601,769,
June 27, 1945. This application October 10,
1950, Serial No. 189,357

9 Claims. (Cl. 62—122)

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This invention relates to the fractionation of gas mixtures, as for example (but without limitation) the separation of air for the production of oxygen and nitrogen.

This application is a continuation of my application Serial No. 601,769, filed June 27, 1945, now abandoned.

The invention is applicable to any two-stage fractionating operation which is conducted at a low subatmospheric temperature and in which the two stages are maintained at materially different pressures.

The invention will be described in connection with the operation of the double or two-stage column commonly used in air fractionation and illustrated in the attached drawings, in which

Figure 1 illustrates a double column in vertical section, the column being provided with an external heat interchanger for a purpose to be described, and

Figure 2 illustrates a modified form of the apparatus having the interchanging elements located inside the low pressure section of the column.

The functioning of the two-stage column is so well known that only the briefest description is needed. Referring to Figure 1, the column itself consists of two stacks of fractionating plates, as at 10 and 11, separated by a partition including a downwardly draining condenser 12.

Air under a relatively high pressure, as for example 75 pounds gauge, and previously cooled to the point of incipient or partial liquefaction, is admitted into the lower compartment 10. In this section gaseous nitrogen is separated, in a variable degree of purity which depends on the number and effectiveness of the plates in this section, and a crude oxygen (for example, of 35% concentration) collects in a pool 13 in the base of the column.

The gaseous nitrogen passes into condenser 12, in which it is liquefied, a part of the liquid returning to reflux the plates of the lower section while a part is collected on an annular tray 14. From this tray the liquid nitrogen passes to the top of the upper section of the column, in which it functions as reflux liquid for the upper stack of plates, this section being maintained at a materially lower pressure, as for example 5 pounds gauge. In passing from the higher to the lower pressure a portion of the liquid nitrogen is evaporated, leaving the column as a saturated vapor.

The crude oxygen collecting in pool 13 is similarly expanded into the upper section of the column, but at a medial height. The plates in

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this section separate the crude oxygen into two fractions: gaseous nitrogen, which passes out of the upper end of the tower, and liquid oxygen, which collects in a pool 15 surrounding condenser 12. Each of these products may and ordinarily will be somewhat impure, depending on the overall efficiency of the column, but the purity of the nitrogen and the quantity of oxygen recoverable at any given degree of purity will be enhanced by the use of the step hereinafter described.

By reason of the difference in the boiling points of oxygen and nitrogen, the pressure of the first or higher pressure stage rises until it becomes stable at a point where the nitrogen, in condensing in condenser 12, is at a temperature slightly higher than that of the oxygen in pool 15 of the second or lower pressure stage, and is thus enabled to impart heat to the oxygen, constantly boiling it.

The separated oxygen collecting in pool 15, which is one of the final products of the column, may be withdrawn in liquid form from below the surface of the pool, as at 16. If preferred, the oxygen may be withdrawn as a vapor from the gaseous body above the surface of the pool, as at 17.

It will be understood that, in this description and also in the appended claims, the terms "gaseous nitrogen" and "liquid nitrogen" are used for identification only and do not imply that the nitrogen is in a state of purity. The nitrogen vapor condensing in condenser 12 is in equilibrium with a liquid containing oxygen and therefore cannot be wholly oxygen-free, and the nitrogen vapor passing out of the upper end of the column also may contain an appreciable proportion of oxygen. While the operation of a double column is improved by reducing the oxygen content of the refluxing nitrogen to the lowest feasible point, the invention herein described is functional so long as some fractionation takes place in the high-pressure section of the column, so that a boiling point difference is established. Thus the term "liquid nitrogen" should be understood to refer to the liquid collecting in the upper end of the high-pressure section, this liquid containing more nitrogen and less oxygen than the air feed. The term "crude oxygen" should be understood to refer to the liquid collecting in the base of the column, this liquid containing less nitrogen and more oxygen than the air feed.

I have discovered that the functioning of a two-stage fractionating system, as exemplified in

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the operation of the double column above described, may be improved materially by abstracting heat from the liquid nitrogen in its passage from the high-pressure to the low-pressure stage. Two methods of effecting this heat abstraction, by interchange against colder fluids, are illustrated in the two figures of the attached drawings.

Referring first to Fig. 1, the crude oxygen flows from pool 13 through conduit 17 and an expansion valve 18 into one side of an indirect heat interchanger 19. In this unit a portion of the crude oxygen vaporizes, conduit 20 conveying the vapor together with the residual liquid to a medial point in the upper column section, as at 21. The high-pressure nitrogen passes from the pool in tray 14 through conduit 22 to the opposite side of the interchanger, in which it preferably counterflows the crude oxygen, flowing thence through conduit 23 and an expansion valve 24 to the upper end of the column as at 25.

In the modification illustrated in Fig. 2 a similar interchange is performed within the low-pressure section of the column. A deep tray 26 is placed within the column at a medial height and a suitable transfer surface, such as a coil 27, is fitted within it. Conduit 28 conducts high-pressure liquid nitrogen to the coil, which discharges through conduit 29 and expansion valve 30 into the upper portion of the column at 25. The stream of crude oxygen flows through conduit 31 and an expansion valve 32 to a point of entry into the low-pressure section, which may be directed into the tray as at 33, or onto a plate above the tray as at 34, or onto a plate below the tray as at 35. In the drawing a single expansion valve is shown and the valves in the branch connections are used as diversion valves only, but if preferred these may be individual expansion valves and any one or two of them may be omitted if the desired point of entry has been predetermined.

The immediate effect of this abstraction of heat from the liquid nitrogen is to reduce, or even to eliminate, the vaporization of the liquid nitrogen stream as it passes the nitrogen expansion valve into a zone of reduced pressure.

The liquid nitrogen leaves the lower section of the column at its boiling point under the pressure existing in this (high pressure) section. In passing to the lower pressure existing in the upper section, without the use of my invention, it must vaporize to such extent as to bring it to its boiling point at the lower pressure. This vaporization has at least three undesirable effects, all of which are reduced or even eliminated by the use of the steps herein described. These undesirable effects are:

First, the excessive quantity of vapor accompanying the liquid nitrogen entering the low-pressure section results in the entrainment of liquid mist in the nitrogen vapor leaving the column and also requires special means to prevent turbulent conditions on the topmost plate.

Second, and this becomes more important as the oxygen content of the liquid nitrogen increases, the vaporization of liquid results in the loss of some oxygen which would otherwise be fractionated from the mixture and added to the oxygen production.

Third, the vaporization of liquid reduces the quantity of reflux available for those plates of the upper section which are located above the crude oxygen inlet. This reduction in reflux necessitates either an increase in the number of plates, the acceptance of a reduced oxygen pro-

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duction or purity, or any two or even all three of these disadvantages.

The abstraction of heat from the liquid nitrogen, prior to its expansion, reduces all three of these undesirable effects and, if performed to a sufficient extent, results in their complete elimination.

While the invention is described only in connection with the fractionation of air, it is equally adapted to and useful in the separation of any binary gas mixture. More broadly, the invention is useful in any fractionation in which a low-boiling reflux liquid is condensed at a relatively high pressure by the evaporation of the higher-boiling component of the feed mixture at a relatively low pressure. Thus the utility of the invention is not confined to column arrangements in which the low-pressure section is superimposed on the high-pressure, but extends to pairs of columns arranged in parallel.

I claim:

1. In a liquefied gas fractionating operation conducted in two zones maintained at a higher and at a lower pressure respectively, with upflowing vapors in repeated direct contacts with downflowing liquids in each zone; the sequence of steps consisting in withdrawing a stream of a relatively high-boiling liquid and a stream of a relatively low-boiling liquid from the higher pressure zone; cooling the first mentioned stream solely by expanding it to the lower pressure; bringing the lower-boiling liquid without change of phase into heat interchange relation with said expanded stream and thereby cooling said lower-boiling stream; expanding said cooled lower-boiling stream to the lower pressure, and introducing both said expanded streams into the lower-pressure zone.

2. In a liquefied gas fractionating operation conducted in two zones maintained at a higher and at a lower pressure respectively, with upflowing vapors in repeated direct contacts with downflowing liquids in each zone; the sequence of steps consisting in withdrawing liquid streams of a relatively high-boiling and a relatively low-boiling component from the higher-pressure zone; cooling the higher-boiling stream solely by expanding it to the lower pressure; and cooling the liquid lower-boiling stream under the higher pressure by heat interchange with the higher-boiling stream under the lower pressure.

3. In a liquefied gas fractionating operation conducted in two zones maintained at a higher and at a lower pressure respectively: the sequence of steps consisting in withdrawing a relatively low-boiling liquid stream from the upper end of the higher-pressure zone; expanding said liquid stream to the lower pressure and introducing the expanded stream into the lower-pressure zone, and cooling said liquid stream prior to said expansion by heat interchange with a mixed liquid drawn in part from the lower end of the higher-pressure zone and in part from the upper portion of the lower-pressure zone, said mixed liquid boiling at substantially the lower pressure during said interchange.

4. In a liquefied gas fractionating operation conducted in two zones maintained at a higher and at a lower pressure respectively, with upflowing vapors in repeated direct contacts with downflowing liquids in each zone, the combination of steps comprising: withdrawing a liquid stream rich in the lower-boiling components of said gas from the higher-pressure zone; expanding said liquid stream to the lower pressure and introduc-

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ing said expanded stream into the lower-pressure zone, and cooling said liquid stream prior to said expansion by heat interchange with a liquid derived from at least one of said zones, said liquid being initially at the temperature at which it existed in the zone from which it was derived and boiling at substantially the lower pressure during said interchange.

5. In a liquefied gas fractionating operation conducted in two zones maintained at a higher and at a lower pressure respectively: the sequence of steps consisting in withdrawing a liquid stream rich in the lower-boiling components of said gas from the upper end of the higher-pressure zone; expanding said liquid stream to the lower pressure and introducing the expanded stream into the lower-pressure zone, and cooling said liquid stream prior to said expansion by heat interchange with a liquid refluxing from the upper portion of the lower-pressure zone and boiling therein at the lower pressure.

6. In combination with a fractionating column for liquefied gaseous mixtures having sections adapted to be maintained at different pressures, at least the higher pressure section having means for producing repeated direct contacts between liquids and vapors counterflowing therein and having means in its upper end for collecting a liquid rich in the lower-boiling component of said gaseous mixture: a heat interchanger having separate flow paths for two fluids; conduit means connecting the respective ends of one of said flow paths with said collecting means and with the upper portion of the lower-pressure section; conduit means connecting the respective ends of the other of said flow paths directly with the lower end of the higher-pressure section and with a medial point in the height of the lower-pressure section; an expansion valve in first said conduit means downstream from said interchanger and an expansion valve in last said conduit means upstream from said interchanger.

7. In combination with a fractionating column for liquefied gaseous mixtures having sections adapted to be maintained at different pressures, at least the higher pressure section having means for producing repeated direct contacts between liquids and vapors counterflowing therein and having means in its upper end for collecting a liquid rich in the lower-boiling component of said gaseous mixture: a heat interchanger comprising a tubular element and a surrounding vessel, the upper end of said vessel communicating with the interior of the lower-pressure section; a conduit connecting said collecting means with one end of said tubular element, and a conduit connecting the other end of said tubular element with the upper portion of said lower-pressure section; a conduit directly connecting the lower portion of the higher-pressure section with the interior of the vessel; an expansion valve in the second said conduit between said tubular element and said lower-pressure section, and an expansion valve in the last said conduit between said higher pressure section and said vessel.

8. In combination with a fractionating column

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for liquefied gaseous mixtures having sections adapted to be maintained at different pressures, each section being provided with means for producing repeated direct contacts between liquids and vapors counterflowing therein and the higher pressure section having means in its upper end for collecting a liquid enriched in the lower-boiling component of said gaseous mixture: a tubular element located at a medial height within the lower-pressure section and means for retaining a pool of liquid surrounding said element; a conduit connecting said collecting means with one end of said tubular element and a conduit connecting the other end of said tubular element with the upper portion of the lower-pressure section; an expansion valve in last said conduit downstream from said element; a conduit arranged to convey liquid from the lower portion of the higher-pressure section into said liquid pool and an expansion valve in last said conduit, and means for directing liquids condensing in the upper portion of the lower pressure section into said liquid pool.

9. In combination with a fractionating column for liquefied gaseous mixtures having sections adapted to be maintained at different pressures, each section having means for producing repeated direct contacts between liquids and vapors counterflowing therein and the higher-pressure section having means in its upper end for collecting a liquid rich in the lower-boiling component of said gaseous mixture: a tubular element located at a medial height within the lower-pressure section and means for retaining a pool of liquid surrounding said element; a conduit connecting said collecting means with said tubular element and a conduit connecting said tubular element with the upper portion of the lower-pressure section; an expansion valve in last said conduit; a conduit connecting the lower portion of the higher-pressure section with a point in the lower-pressure section below said liquid pool and an expansion valve in said conduit, and means for directing liquids condensing in the upper portion of the lower-pressure section into said pool and from said pool into the lower portion of the lower-pressure section.

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