Aug. 26, 1952 P. L. KAPITZA 2,608,070

METHOD AND MEANS FOR DISTILLATION OF LOW BOILING POINT LIQUIDS

Filed Feb. 20, 1946 2 SHEETS—SHEET 1

Fig. 1.

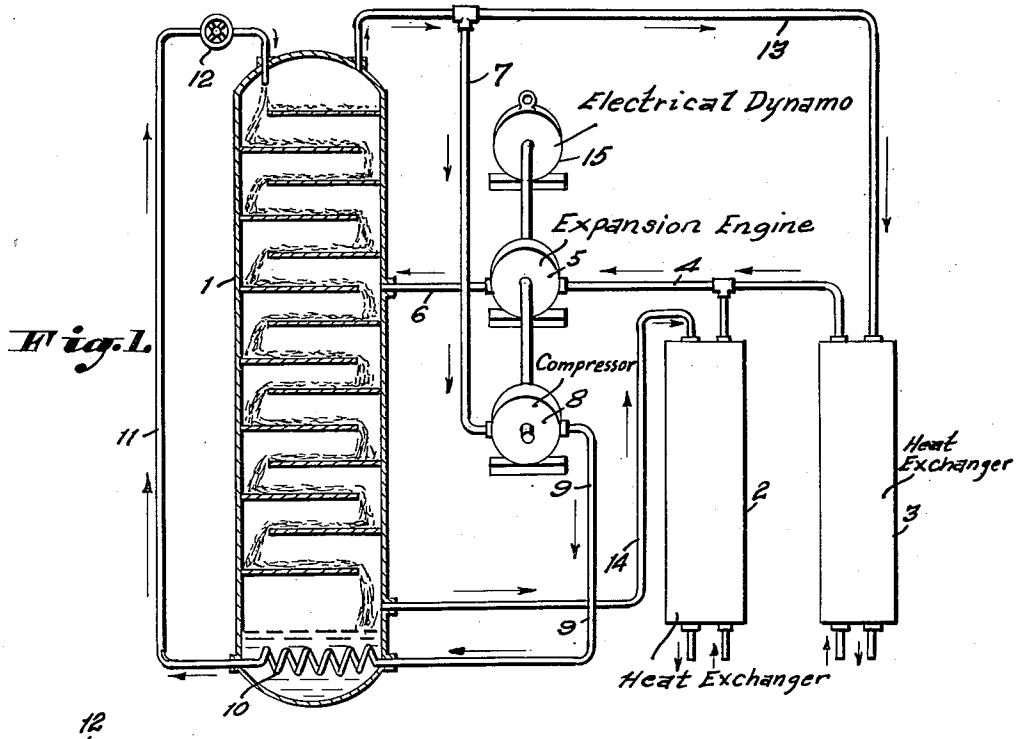

Fig. 2.

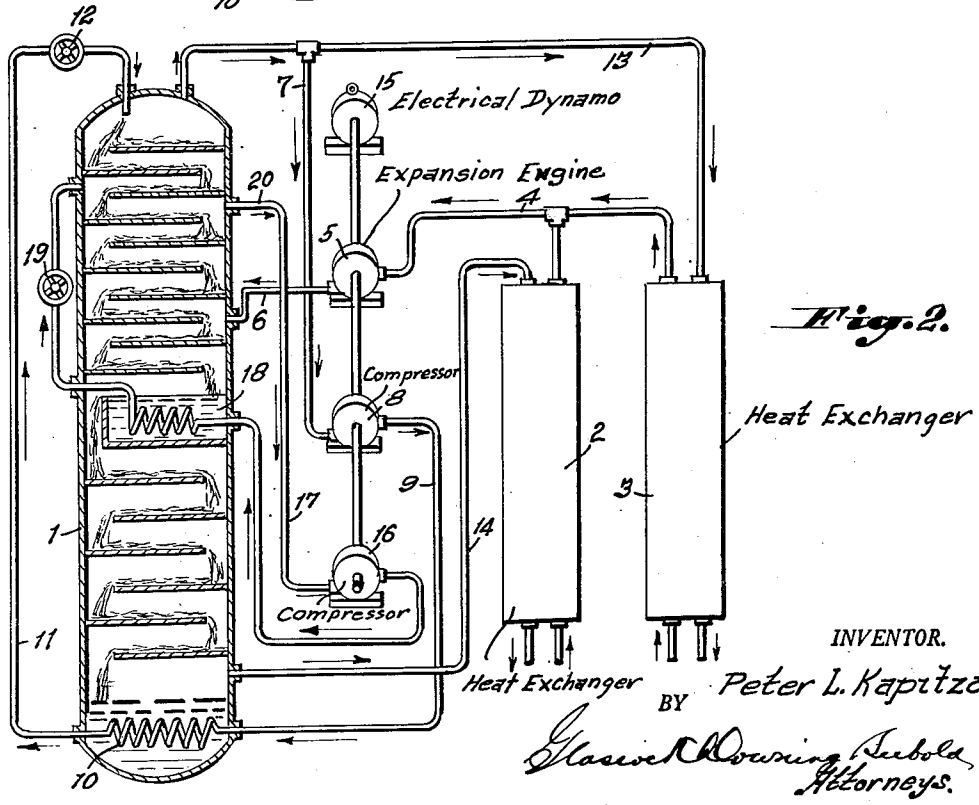

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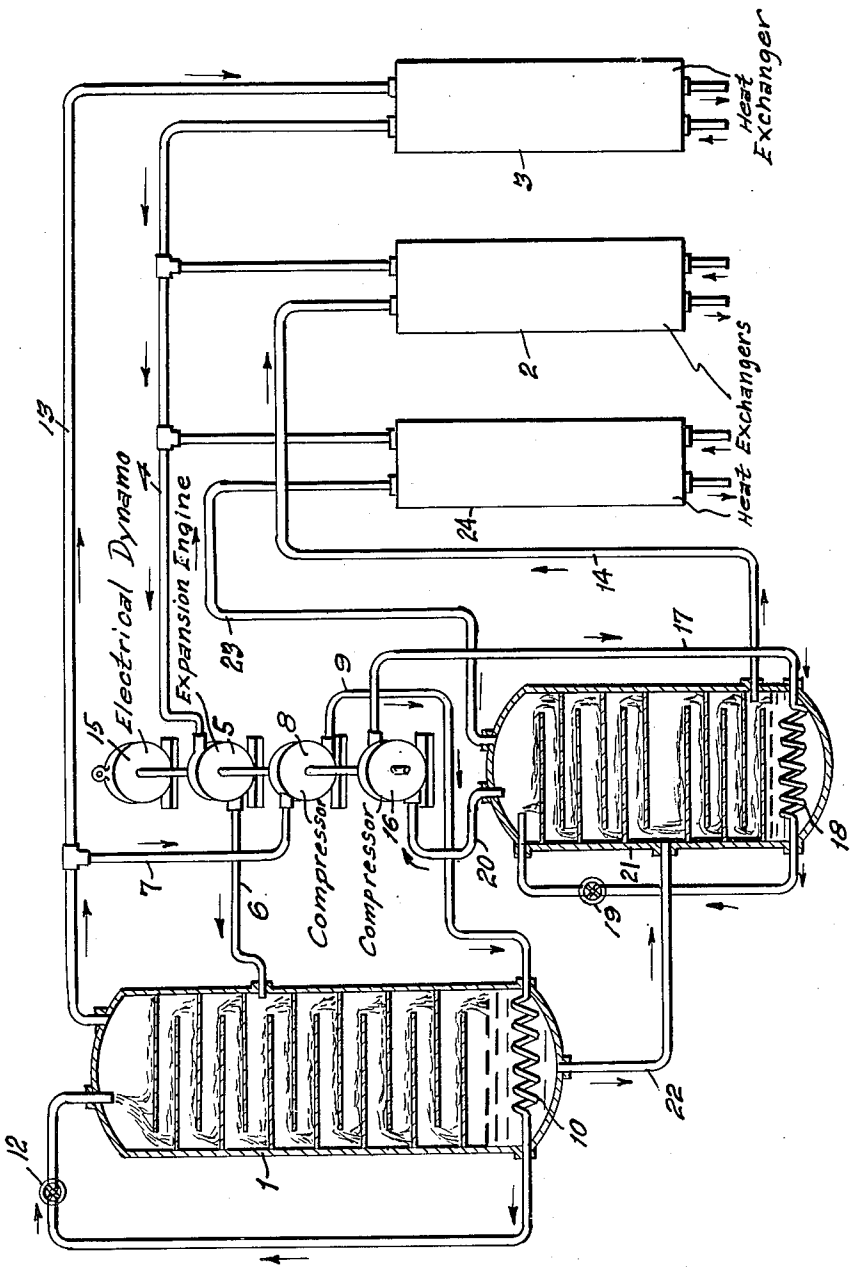
Fig. 3.



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METHOD AND MEANS FOR DISTILLATION OF LOW BOILING POINT LIQUIDS

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In the Union of Soviet Socialist Republics February 11, 1944

6 Claims. (Cl. 62—123)

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The present invention relates to method and means for rectification and distillation of liquid with low temperature boiling points.

The main difficulty encountered in the fractional distillation or rectification of liquids boiling at low temperatures such as occur for example in the distillation of air, water gas, or coal gas from the liquid phase consists in liquifying the more volatile fraction.

In particular, when producing oxygen from air the greatest difficulty is encountered in obtaining a reflux liquid of liquid nitrogen. Hitherto these difficulties were overcome by the use of double columns (for example, the double columns used in the Linde and Claude methods for the fractional distillation of liquid air), in which one column serves to obtain a reflux of the most volatile component, while the other serves for the actual distillation. This method requires that the air be supplied at high pressures while the design of the apparatus necessitates the use of additional condensers and evaporators. This reduces the efficiency of the whole system considerably and requires a complicated construction of large dimensions.

Proposals have been made in the art to obtain a reflux by compressing the most volatile components in a compressor, the compression being performed at a temperature higher than the temperature at which said component leaves the column and to afford this said component is led through a series of heat exchangers. In this connection I cite United States Patent 2,095,809 disclosing ample evidence of the use of the above-mentioned principle.

The present invention is characterised by the direct liquefaction of the most volatile components at the cold end of the cycle by a compressor.

The appended schematic diagrams illustrate: in Figure 1, the apparatus for liquifying the most volatile component in the case of a binary mixture; in Fig. 2, a variant of the system, having a second compressor coupled in parallel to increase the efficiency of the rectifying column, and in Fig. 3, a system for separating mixtures containing three or more components.

For the sake of simplicity and ease in describing the systems illustrated, all mechanisms and apparatus common to all three diagrams are given the same designations.

The system shown in Fig. 1 is the simplest and may be used, for example, to fractionate air composed of oxygen and nitrogen. The compressed gas is passed through the heat exchangers 2 and

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3, and by means of the pipe 4 passes into the expansion engine 5 which may be of the turbine type or any machine wherein the gas is expanded, this expansion being accompanied by the production of mechanical energy delivered for adequate use after which the cooled and partly liquefied gas is conducted through pipe 6 to the column 1. The most volatile component concentrates in the upper part of the column, and is removed by pipe 7 and passed to the compressor 8 in which it is compressed to a pressure, at which liquefaction of the reflux occurs. Through pipe 9 the compressed gas flows into the evaporator 10 situated at the lower end of the column in which the component of higher boiling point collects. The pressure at the exhaust of the compressor 8 is made such that the most volatile component is condensed in the evaporator 10. This liquified component is directed through pipe 11 having a valve 12 to the top of column 1 and flows to the bottom of the column 1 passing successively over the transverse perforated plates inside the column. The separated fractions are removed by pipes 13 and 14 passing through heat exchangers 2 and 3. The efficiency of the fractionating column can be increased very simply by the addition of other compressors working in parallel with the first. The conditions for condensing and evaporating the reflux at various levels in the fractionator are in accordance with the conditions requisite for improving the efficiency of the system. Fig. 2 shows the same arrangement as in Fig. 1 with one additional compressor.

According to the arrangement of the apparatus shown in Fig. 2, the gas of which the reflux is to be made, is removed from the fractionating column at two points, one at the top, through pipe 7 and one somewhat lower through pipe 20. The part of the gas removed through pipe 7 passes through the cycle described above while that portion which is removed by pipe 20 is directed through a second compressor 16 out of which it is led through pipe 17 to the evaporator 18 arranged in the upper half of the fractionating column 1. The liquified gas is led into the column through the pipe and valve 19, the inlet being somewhat higher in the column 1 than the exhaust pipe 20. Following the above principle a third, fourth, and further compressors may be added, and the more of such compressors there are in operation, the closer will be the mode of operation of the column approach to that of a reversible one, and the higher will be its efficiency.

The same principle may be applied to the separation of three or more components from a gaseous mixture such as the argon, nitrogen and oxygen in air. A system arranged for this purpose is shown in Fig. 3, in which there are two fractionating columns of the type shown in Fig. 1.

The diagram shown in Fig. 3 illustrates the use of one compressor per fractionating column. It is clear, however, that the efficiency of the system may be improved by the addition of further compressors according to the system illustrated in Fig. 2.

In the case of a trinary mixture, the gas is passed through the expansion machine **5** to column **1** the two less volatile components collecting in the evaporator **10** from which they flow through pipe **22** to the central part of the fractionating column **21**. In this column, the reflux is produced by means of the compressor **16** to which the gas is led through pipe **20**. The compressed gas passes through pipe **17** to the evaporator **18** in which it is condensed, the resulting reflux liquid being directed through the pipe and valve **19** to the upper end of the fractionating column. The component of intermediate boiling point, in this case—argon, is led through the heat exchanger **24** through pipe **23**, the component of high boiling point—oxygen—leaving the system through the pipe **14** and heat exchanger **2**.

It is clear from the principle illustrated in Fig. 3 that each additional component requires an additional fractionating column and heat exchanger for its separation from the original mixture. The fractionators may be supplied with one or more compressors.

Obviously, the principle described above may be applied to all existing types of fractionating columns such as those having perforated plates, rotating discs etc. Similarly, the heat exchangers **2**, **3** and **24** may be of any existing types.

One of the advantages of the system according to the present invention lies in the possibility of driving the compressors from the shaft of the expansion engine. In this case, the work expended on compressing the most volatile component and consequently raising its temperature is compensated by the temperature drop in the expansion engine and in this manner, the temperature balance in the fractionating column is not violated.

Another feature of the proposed system lies in the fact that the reflux is usually composed of only a part of the total quantity of the gas flowing through the expander and consequently the pressure of the gas leaving the compressor may be made higher than that of the gas entering the expander. Furthermore when the efficiencies of the compressor and expansion engine are sufficiently high, the pressure of the most volatile component (nitrogen, in the case when air is being separated into its two main components—oxygen and nitrogen) can be made higher at the evaporation point of the cycle than that of the air entering the system. This assists in the formation of the reflux used in the fractionating column. This is a marked advantage over the system proposed by Linde and Frenkel.

The greater density of gases at low temperatures enables greater pressure changes to be obtained in compressors having few compression stages (such as turbine compressors) and in expansion engines of the turbine type and consequently increases their efficiency as a result of which, the use of this type of compressor and expansion engine is to be recommended in installation of the proposed type.

A further increase in the efficiency of the system may be obtained by adding an auxiliary load **15** to the axle of the expansion machine as shown in Figures 1, 2 and 3. This may be a dynamo or any other power absorbing machine.

The method advanced in the present invention for producing a reflux liquid of the most volatile components by means of a compressor at the cold end of a cycle can be applied in practice in various ways as is obvious from the discussion of the examples given above. Furthermore it is clear that the application of the method considerably simplifies the construction of fractional stills, affording new possibilities to the art of low temperature distillation.

Since details of method and means for the rectification and distillation of liquid with low temperature boiling points may be modified, the scope of the invention is defined by the claims as hereunto appended.

I claim:

1. A method of rectification and fractional distillation of liquids boiling at low temperatures, comprising removing at least one of the most volatile fractions of the composition undergoing fractionation from a first point at the top portion of a rectification zone, compressing at least a part of the fraction removed at a temperature level corresponding substantially to the temperature at the top of said rectification zone and returning the compressed material as a reflux to the top portion of the rectification zone, feeding compressed gases to the rectification zone between the top and bottom thereof, expanding the gases so fed, and utilizing the mechanical energy released by said expansion for compressing said part of the fraction removed from the rectification zone at the first point.

2. In the rectification and fractional distillation of liquids boiling at the low temperatures, the process which comprises liquefying gaseous components of normally gaseous material, expanding the liquefied gas and introducing the same into a rectification zone between the top and bottom thereof, removing at least one of the most volatile fractions from the upper portion of said zone, compressing at least a part of said fraction at a temperature level corresponding substantially to the temperature at the top of said rectification zone, passing the compressed material through a lower portion of the rectification zone and utilizing the same for heating that portion of the zone, subsequently passing the compressed material to the top portion of the zone and utilizing it as a reflux therein, and utilizing mechanical energy released by the expansion of the liquefied gas to compress the part of the fraction removed from the top portion of the rectification zone.

3. Means for effecting rectification and fractional distillation of liquids boiling at low temperatures, comprising a rectification column, an expander, a heat exchanger, means for passing a mixed component fluid to be rectified through the heat exchanger and expander into the rectification column between the top and bottom thereof, a compressor driven by the expander, an evaporator arranged in the bottom portion of the rectification column, conduit means for leading a fraction of said fluid from the top of the column directly through the compressor and through the evaporator, and means for leading

said fraction from the evaporator and for discharging it into the top of the column.

4. Means for effecting rectification and fractional distillation of liquids boiling at low temperatures, comprising a rectification column having two evaporators arranged therein at different levels, means for leading a normally gaseous fluid and for discharging it into the column above said evaporators, an expander interposed in said means for utilizing the expansion of said fluid to create mechanical energy, first and second compressors driven by said expander, first and second conduits leading from spaced points of the rectification column above the point where the normally gaseous fluid is introduced directly to the compressors respectively, a passageway connecting one of said compressors to one of the evaporators, another passageway connecting the other compressor to the other one of said evaporators, a third passageway connecting the evaporator at the lower level to the top of the column, and a fourth passageway connecting the other evaporator to the column at a point above that where the normally gaseous fluid is introduced.

5. Means for effecting rectification and fractional distillation of liquids boiling at low temperatures, comprising rectification column means having a plurality of evaporators arranged therein at different levels, a plurality of heat exchangers, an expansion engine, conduit means for passing a normally gaseous fluid through said heat exchangers and the expansion engine and for discharging the same into the rectification column between the top and bottom thereof and at a point above said evaporators, a plurality of compressors corresponding in number to the number of the evaporators and driven by said expansion engine, passageways placing points in the rectification means above the evaporators in direct communication respectively with the respective compressors, other passageways placing the compressors in communication with the respective evaporators, other passageways placing each evaporator in communication with the rectification column means at a point above that where the evaporator is located, and conduit means for leading fractions from the rectification column means through the heat exchangers.

6. Means for effecting rectification and fractional distillation of liquids boiling at low temperatures, and containing at least three components, comprising series connected rectification columns each having at least one evaporator arranged therein, the number of said rectification columns being one less than the number of components subjected to distillation, at least one compressor for each rectification column, communicating with the corresponding evaporator in said columns, an expander, heat exchangers, means for passing a normally gaseous fluid through said heat exchangers and said expander and for discharging the same into at least one of said rectification columns between the top and bottom thereof, means for leading gaseous fractions from the tops of the columns directly to the compressors, means for utilizing the mechanical energy of expanding gases in the expander to drive said compressors, means for leading fluid through each evaporator to the rectification column in which the evaporator is located and for discharging the same into that column at a higher elevation than the evaporator, and means for passing fluid from each rectification column through at least one of the heat exchangers.

PETER LEONIDOVITCH KAPITZA.

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