

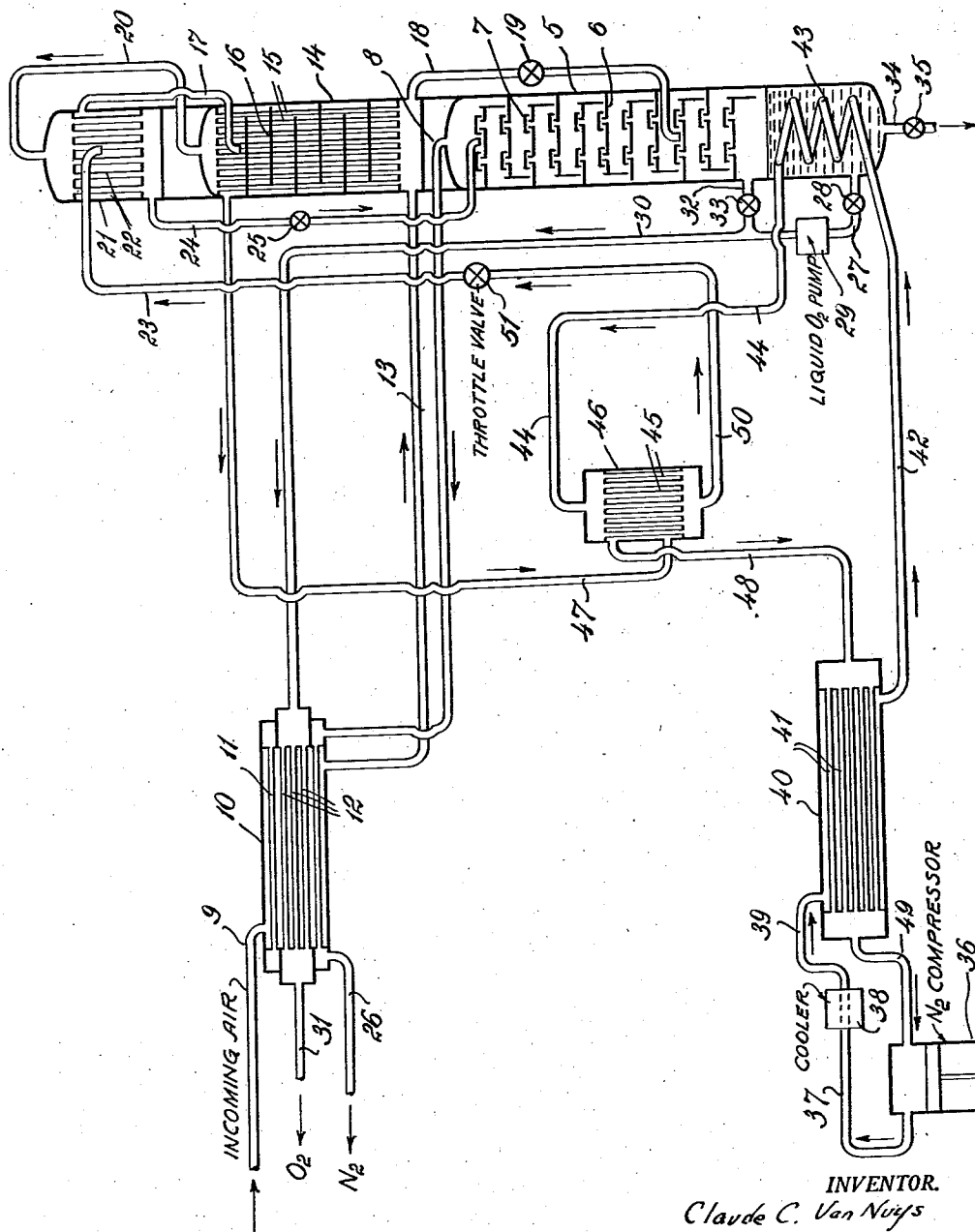
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PROCESS FOR SEPARATING THE CONSTITUENTS OF AIR

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PROCESS FOR SEPARATING THE CONSTITUENTS OF AIR

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This invention relates to the separation of atmospheric air into its constituents by liquefaction and rectification, and particularly to improvements in the method and apparatus therefor.

In commercial liquefaction methods of separating oxygen from the atmosphere as heretofore practised, the necessary refrigeration to attain low temperatures is obtained by compression and subsequent expansion of the air treated. This necessitates initial compression of the air to relatively high pressure of the order of 15-20 atmospheres. Such compression can be attained only in reciprocating compressors which require oil lubrication. The oxygen of the air under such pressures and at the temperatures resulting from compression causes decomposition of the lubricating oil and the introduction to the air stream of substantial proportions of hydrocarbons in addition to any which may be present. The use of special lubricating oils does not avoid this source of contamination.

Since the hydrocarbons are not removed from the air stream by the usual treatment intended to separate moisture and carbon dioxide, they tend to accumulate and eventually find their way to the compartment of the apparatus where liquid oxygen accumulates. The presence of such material in contact with pure oxygen, whether liquid or gaseous, is undesirable. Hence very elaborate attempts have been made heretofore to eliminate the hydrocarbons at some point intermediate compression of the air and the formation of liquid or vapor rich in oxygen in which the hydrocarbons might accumulate. Such attempts involve operations which interfere with the efficiency of the cycle and the use of necessarily complicated equipment. Furthermore, these attempts have failed substantially to achieve the desired object. It is still possible for hydrocarbons derived from the lubricating oil to accumulate at points in the apparatus where gases or liquids rich in oxygen are present.

It is the object of the present invention to provide a simple and effective method and apparatus whereby the presence of hydrocarbons resulting from decomposition of oil in the compression of the air can be eliminated and further improvements in the separation of oxygen from the atmosphere can be obtained.

Other objects and advantages of the invention will be apparent as it is better understood by reference to the following specification and the accompanying drawing, which illustrates diagram-

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matically an apparatus suitable for the practice of the invention.

In accordance with the present invention, the air to be separated is not utilized to produce any refrigeration in the cycle. I employ instead a separate nitrogen cycle which acts as a heat transfer agent everywhere in the cycle. The air need be compressed in accordance with my invention only to pressures not substantially higher than three atmospheres absolute. Usually a pressure of two atmospheres, or sufficient pressure only to ensure movement of the gas through the apparatus is needed to accomplish the purpose of the invention. Such pressures are easily attainable in various types of blowers which require no internal lubrication. Hence no lubricating oil and decomposition products thereof are mingled with the air which is eventually separated to recover oxygen therefrom. The procedure as hereinafter described effectively increases the proportion of oxygen which can be recovered from the atmosphere and the purity of the product.

The nitrogen which affords the refrigerative effect is compressed, circulated and expanded in such a way as to provide sufficient cold so that the air can be liquefied at low pressure. The nitrogen of the refrigeration cycle does not mingle at any point with the air undergoing separation. Hence the presence of hydrocarbons in the nitrogen cycle is not inimical to the method and affords no possibility of accumulation of hydrocarbons in the presence of oxygen or products rich in oxygen. The nitrogen can be compressed to the necessary pressure, for example 2500 pounds per square inch, in starting the apparatus, and from 900 to 1,000 pounds per square inch thereafter, in any available form of compressor. Oil lubrication may be used. The quantity of nitrogen necessary for the refrigeration cycle will depend upon the size of the apparatus but will not in any case exceed the quantity of air contained in the air separation cycle.

The procedure will be readily understood by reference to the drawing, in which 5 indicates a rectification column having the usual trays 6 and bubble caps 7. The column is provided at its top with an outlet 8 through which the effluent rich in nitrogen escapes. Liquid oxygen accumulates in the bottom of the column, and the vapors rise therefrom through the trays 6 and bubble caps 7 in contact with liquid flowing downwardly over the trays.

To provide this liquid, air, previously compressed to the initial pressure, preferably not greater than three atmospheres absolute in any

suitable apparatus such as a blower (not shown) is introduced through a pipe 9 into an exchanger 10 and travels about pipes 11 and 12 therein in heat exchange relation with cold products of the separation. The air thus cooled is delivered through a pipe 13 to the bottom of a backward return condenser 14 having tubes 15 and baffles 16 therein. The air flows upwardly through the tubes, being subjected therein to backward return condensation by heat exchange with nitrogen liquid and vapors supplied through a pipe 17. The liquid from the condenser 14, which is enriched in oxygen, is delivered by a pipe 18 controlled by a valve 19 to an intermediate level of the column 5.

The gaseous residue from the tubes 15 consisting principally of nitrogen is delivered by a pipe 20 to a condenser 21 containing tubes 22 through which the residue flows in heat exchange relation with liquid nitrogen supplied through a pipe 23. The surplus liquid from the condenser 21 overflows through the pipe 17. The residue is condensed by heat exchange with the liquid nitrogen and is delivered by a pipe 24 controlled by a valve 25 to the top of the column 5 and affords the reflux nitrogen liquid required to effectively separate substantially all of the oxygen from the air treated.

The effluent nitrogen from the column is delivered by the pipe 8 to the tubes 11 of the exchanger 10 and is withdrawn through a pipe 26 to any suitable receptacle or to the atmosphere. The oxygen product may be withdrawn as a liquid through a pipe 27 controlled by a valve 28 and delivered by a liquid pump 29 to a pipe 30 which delivers it to the pipes 12 of the exchanger 10 wherein the liquid is vaporized. The oxygen product is withdrawn through a pipe 31 and delivered to any suitable storage receptacle. Alternatively, oxygen in the form of vapor may be withdrawn through a pipe 32 controlled by a valve 33 and delivered to the pipe 30. Liquid oxygen may be withdrawn through a pipe 34 controlled by a valve 35 to purge the column of any impurities which may accumulate therein.

To afford the necessary refrigeration for the system, nitrogen is compressed in a compressor 36 to a suitable pressure as hereinbefore described and delivered by a pipe 37 to a cooler 38. Thence the nitrogen is delivered by a pipe 39 to an exchanger 40 and travels about tubes 41 therein where it is further cooled by heat exchange with cold nitrogen vapor returning in the cycle. The cold nitrogen is delivered by a pipe 42 to a coil 43 which is immersed in the liquid oxygen accumulating in the bottom of the column 5. The resulting heat exchange cools the nitrogen and causes vaporization of a portion of the liquid to maintain the vapor which rises through the column. The sub-cooled nitrogen is then delivered through a pipe 44 to the tubes 45 of an exchanger 46 where it is liquefied by heat exchange with cold nitrogen vapors entering the liquefier through a pipe 47. These vapors escape through a pipe 48 to the exchanger 40 and after passing through the tubes 41 of the exchanger, are delivered by a pipe 49 to the compressor 36.

The liquid nitrogen from the liquefier 46 is delivered through a pipe 50 and throttle valve 51 to the pipe 23 and thus to the condenser 21, affording the liquid nitrogen necessary to liquefy the nitrogen reflux for the column 5 and also to effect the initial backward return condensation of the air entering the system. The nitrogen

vapor from the condenser 14 escapes through the pipe 47 to the liquefier 46. Hence the nitrogen circulates continuously, affording all of the necessary refrigeration for the system. The nitrogen in the refrigeration cycle does not at any point mingle with the air undergoing separation or the products thereof. Nitrogen separated from the air is withdrawn, as is also the oxygen product, independently of the refrigeration cycle. Since the nitrogen employed for refrigeration travels in an independent cycle, it is immaterial whether or not it may carry hydrocarbons. These hydrocarbons, if present, cannot mingle with the air undergoing separation and thus arrive at a point in the system where oxygen or products rich in oxygen are present. Thus the liquid oxygen which accumulates in the bottom of the column 5 is free from any contaminating hydrocarbons which might have been introduced owing to the necessity of employing oil in the initial compression of the air. The use of the blower to compress the air obviates that possibility.

The invention as described affords a practical and simple solution of a problem which has existed for many years in the commercial production of oxygen by liquefaction and rectification. It also permits substantially total recovery of the oxygen content of the air, since the nitrogen reflux which is supplied at the top of the column prevents the escape of oxygen in the effluent.

Various changes may be made in the details of procedure and in the apparatus as described without departing from the invention or sacrificing the advantages thereof.

I claim:

1. The method of separating the constituents of atmosphere air in a liquefaction apparatus by liquefaction and rectification which comprises compressing nitrogen to a relatively high pressure, liquefying the nitrogen, compressing the air to a pressure not materially in excess of that required to overcome the resistance to travel of the air through the apparatus without abrupt change in pressure, subjecting the air to indirect heat exchange with the liquid nitrogen to separate liquid fractions enriched respectively in oxygen and nitrogen, and rectifying the two fractions to recover substantially pure liquid oxygen.

2. The method of separating the constituents of atmospheric air in a liquefaction apparatus by liquefaction and rectification which comprises compressing nitrogen to a relatively high pressure, liquefying the nitrogen, compressing the air to a pressure not materially in excess of that required to overcome the resistance to travel of the air through the apparatus without abrupt change in pressure, subjecting the air to backward return condensation with partial condensation thereof, condensing the gaseous residue from the backward return condensation by indirect heat exchange with the liquid nitrogen to separate liquid fractions enriched respectively in oxygen and nitrogen, and rectifying the two fractions to recover substantially pure liquid oxygen.

3. The method of separating the constituents of atmospheric air by liquefaction and rectification which comprises compressing the air to a relatively low pressure such that expansion from that pressure will not produce refrigeration sufficient to effect liquefaction thereof, subjecting the compressed air to cooling and liquefaction in two fractions enriched respectively in oxygen and nitrogen by indirect heat exchange with the liquid nitrogen, rectifying the two fractions to recover substantially pure liquid oxygen and main-

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taining the refrigeration necessary for the operation by compressing nitrogen to relatively high pressure and liquefying the nitrogen to supply continuously the liquid required to effect liquefaction of the air.

4. The method of separating the constituents of atmospheric air by liquefaction and rectification which comprises compressing the air to a relatively low pressure such that expansion from that pressure will not produce refrigeration sufficient to effect liquefaction thereof, subjecting the compressed air to cooling and liquefaction in two fractions enriched respectively in oxygen and nitrogen by indirect heat exchange with the liquid nitrogen, rectifying the two fractions to recover substantially pure liquid oxygen and maintaining the refrigeration necessary for the operation by compressing nitrogen to relatively high pressure and liquefying the nitrogen by heat exchange successively with the liquid oxygen and cold nitrogen vapors to supply continuously the liquid required to effect liquefaction of the air.

5. The method of separating the constituents of atmospheric air which comprises compressing the air to a relatively low pressure without contaminating the air with lubricating oil, maintaining a separate nitrogen cycle including compression of the nitrogen to a relatively high pressure and liquefaction of the nitrogen, subjecting the air at low pressure to indirect heat exchange with the liquid nitrogen to separate two liquid fractions respectively enriched in oxygen and nitrogen, and rectifying the two fractions to recover substantially pure liquid oxygen.

6. The method of separating the constituents of atmospheric air which comprises compressing the air to a relatively low pressure without contaminating the air with lubricating oil, main-

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taining a separate nitrogen cycle including compression of the nitrogen to a relatively high pressure and liquefaction of the nitrogen by heat exchange with cold nitrogen vapors, subjecting the air at low pressure to indirect heat exchange with the liquid nitrogen to separate two liquid fractions respectively enriched in oxygen and nitrogen and rectifying the two fractions to recover substantially pure liquid oxygen.

7. The method of separating the constituents of atmospheric air which comprises compressing nitrogen to a relatively high pressure sufficient to afford the necessary refrigeration to liquefy the nitrogen, liquefying the compressed nitrogen, compressing the air to a relatively low pressure, vaporizing the liquid nitrogen by backward return condensation of the air with partial condensation thereof, liquefying the gaseous residue from such backward return condensation to provide two liquid fractions enriched respectively in oxygen and nitrogen, and rectifying the two fractions to recover substantially pure liquid oxygen.

8. The method of separating the constituents of atmospheric air which comprises compressing nitrogen to a relatively high pressure sufficient to afford the necessary refrigeration to liquefy the nitrogen, liquefying the compressed nitrogen, compressing the air to a relatively low pressure, vaporizing the liquid nitrogen by backward return condensation of the air with partial condensation thereof, liquefying the gaseous residue from such backward return condensation to provide two liquid fractions enriched respectively in oxygen and nitrogen, rectifying the two fractions to recover substantially pure liquid oxygen, and recompressing the vapor produced by vaporizing the liquid nitrogen to maintain the cycle.

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