

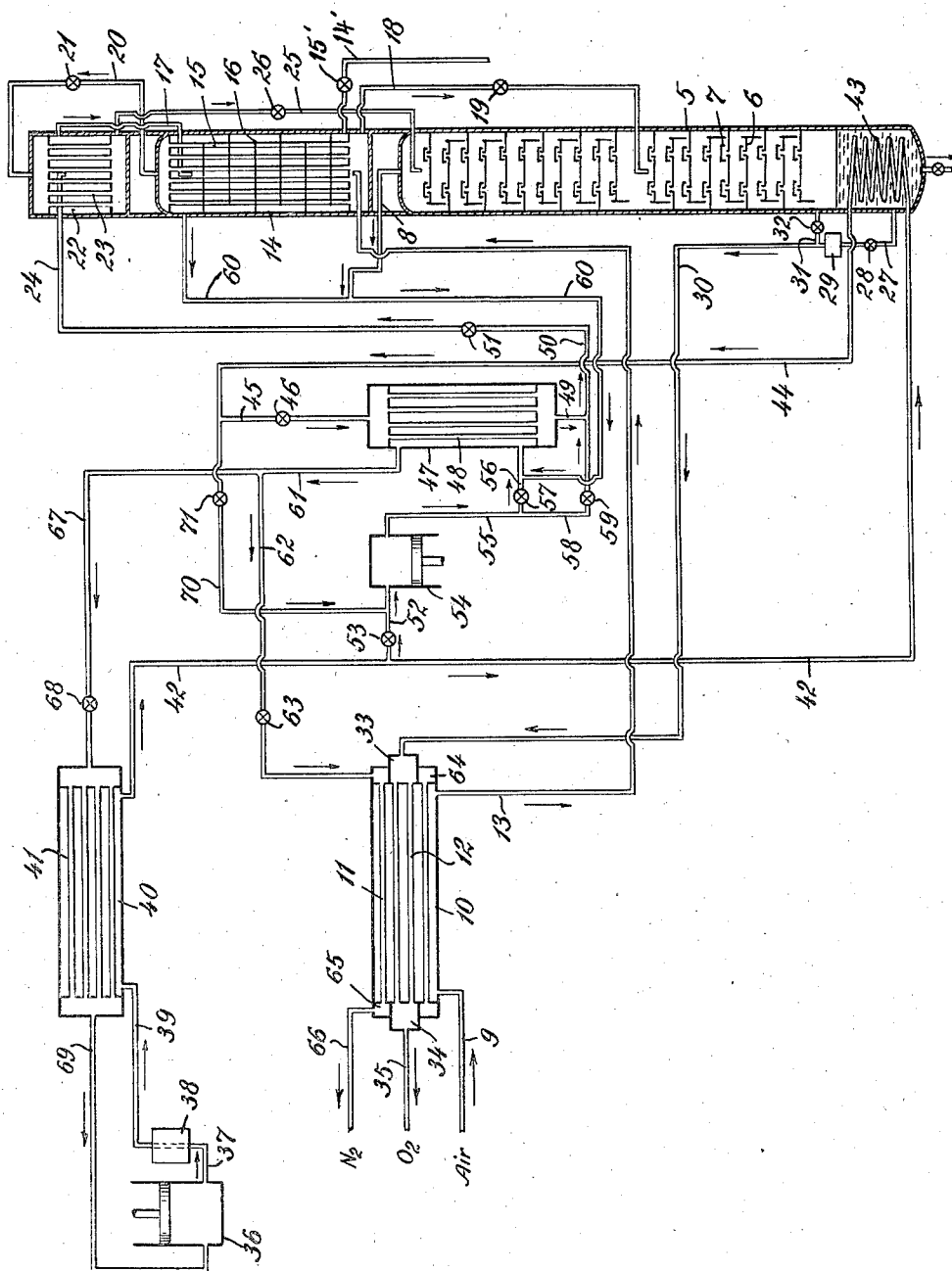
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SEPARATION OF THE CONSTITUENTS OF AIR

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SEPARATION OF THE CONSTITUENTS
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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 cause 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.

Another object of the invention is the provision of a method and apparatus whereby the power required to maintain refrigeration necessary for the liquefaction of air and the separation of the constituents thereof is materially reduced, thus affording a more economical procedure for the

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recovery of oxygen and nitrogen from the atmosphere.

A further object of the invention is the provision of a method and apparatus affording more economical utilization of the available refrigeration, avoiding premature liquefaction of the air introduced for separation.

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 diagrammatically 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 sufficient refrigeration to maintain the cycle. I employ instead a nitrogen cycle which acts as a heat transfer agent to effect the liquefaction of the entering air. The air need be compressed in accordance with the 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 refrigeration to liquefy the air 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.

However, in accordance with the present invention, hydrocarbons may be eliminated also from the nitrogen cycle since it is unnecessary to compress the nitrogen to pressures higher than those attainable in available types of blowers. The necessary refrigerative effect can be provided readily by compression of the nitrogen to relatively low pressures, generally not exceeding 5 atmospheres absolute, and the subsequent expansion of a portion of the nitrogen with external work in a suitable engine or turbine. Higher

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pressures may be used, but it is usually inadvisable for economic reasons to compress the nitrogen to pressures higher than 8 atmospheres absolute, since the additional work expended affords no advantage in the system. While a reciprocating compressor can be employed to compress the nitrogen to the relatively low pressures necessary, a suitable blower is ordinarily satisfactory for the purpose.

The amount of refrigeration which is readily obtainable in the system as hereinafter described is in fact such that some of the refrigeration available must be transferred from the products of rectification to avoid excessive cooling of the entering air, and this may be accomplished by heat exchange between the liquid oxygen product and the nitrogen circulating in the separate nitrogen cycle, for example by the provision of a coil at the bottom of the rectifier through which the nitrogen flows. The effluent nitrogen which leaves the top of the rectifier is still very cold, and if all of this nitrogen is delivered to an exchanger through which the entering air passes, some of the air, even at the low pressure employed, may be prematurely liquefied. Accordingly, I utilize the effluent nitrogen as a cooling agent for nitrogen flowing in the nitrogen cycle, diverting only a portion thereof for heat exchange with the entering air while the remainder is mingled with the circulating nitrogen of the nitrogen cycle. The proportional distribution of the cold effluent nitrogen is such that the entering air is not prematurely liquefied and the remainder of the available refrigeration is conserved, thus reducing the work necessary to maintain refrigeration through the operation of the separate nitrogen cycle.

That considerable economy is possible in the operation of the procedure as described is readily evident. In commercial systems for the liquefaction and separation of the constituents of air such as the well known Claude system in which a part of the air is expanded with external work, it is necessary usually to compress the air initially to a pressure approximating 20 atmospheres absolute. In the engine a portion of the air is expanded to approximately 5 atmospheres absolute or in a ratio of 4:1. The refrigerative effect obtained is a function of the ratio of expansion. Hence initial compression of the gas which is employed to attain refrigerative effect to a pressure of 4 atmospheres absolute and expansion of a portion thereof to a pressure of 1 atmosphere absolute will afford the same amount of refrigerative effect in respect to a given volume of the gas. It is not possible to take advantage of this condition where the gas to be separated, such as air, is utilized also in maintaining refrigeration. The advantage can be secured, however, by utilizing nitrogen in a separate refrigeration cycle in accordance with the invention as herein described.

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 the liquid flowing downwardly over the trays.

To provide this liquid, air previously compressed to the initial pressure, preferably not greater than 3 atmospheres absolute, in any suitable ap-

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paratus such as a blower (not shown) is introduced through a pipe 9 to an exchanger 10 and travels about pipes 11 and 12 therein in a heat exchange 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 liquid nitrogen 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 of the air from the tubes 15, consisting principally of nitrogen, is delivered by a pipe 20 controlled by a valve 21 to a condenser 22 containing tubes 23 through which the residue flows in contact with liquid nitrogen supplied through a pipe 24. The gaseous residue is condensed by heat exchange with the liquid nitrogen and is delivered by a pipe 25 controlled by a valve 26 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 surplus liquid nitrogen from the condenser 22 overflows through the pipe 17 together with the pure nitrogen vapor formed in the condenser and serves as the cooling agent in the condenser 14. A purge line 14' controlled by a valve 15' permits withdrawal of liquid nitrogen from the condenser 14.

The liquid oxygen from the bottom of the column 5 may be withdrawn through a pipe 27 controlled by a valve 28 and may be pumped by a suitable liquid pump 29 through a pipe 30. If desired, oxygen vapor from the bottom of the column 5 may be withdrawn through a pipe 31 controlled by a valve 32 and delivered to the pipe 30. The oxygen liquid or vapor in the pipe 30 is delivered to a compartment 33 at the end of the exchanger 10 and after passing through the tubes 12, enters a compartment 34 whence it is withdrawn through a pipe 35 and delivered to any suitable storage receptacle.

To maintain refrigeration, nitrogen is compressed in a compressor 36 and delivered by a pipe 37 to a cooler 38. Thence it is delivered by a pipe 39 to an exchanger 40 having tubes 41 where the nitrogen is cooled by cold nitrogen returning in the cycle as hereinafter described. The cooled nitrogen is withdrawn through a pipe 42 and delivered to a coil 43 in the bottom of the column 5 which permits further cooling of the nitrogen by heat exchange with the liquid oxygen in the bottom of the column. The nitrogen is then delivered by a pipe 44 and a branch 45 having a valve 46 to a liquefier 47 having tubes 48. Here the nitrogen is completely liquefied by heat exchange with cold nitrogen vapors provided as hereinafter described. The liquid nitrogen is withdrawn through a pipe 49 and delivered to a pipe 50 having a throttle valve 51 which is connected to the pipe 24. The liquid nitrogen is thus supplied to the condenser 22 for utilization in liquefying the air in the condensers 14 and 22.

A portion of the nitrogen from the pipe 42 is diverted through a pipe 52 controlled by a valve 53 to an expansion engine 54 where the nitrogen is expanded with external work and thereby cooled. The expanded nitrogen escapes from the engine through a pipe 55 and is delivered through a pipe 56 controlled by a valve 57 to the liquefier 47 where the refrigeration is utilized to liquefy nitrogen flowing through the tubes 48. A portion

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of the expanded nitrogen may be diverted through a pipe 58 controlled by a valve 59 which delivers it to the pipe 50 in which it is mingled with liquid produced in the liquefier 47.

Nitrogen vapor from the condenser 14 escapes through a pipe 60 and is mingled with the effluent nitrogen from the rectification which escapes through the pipe 8. The total of the nitrogen vapor is delivered by the pipe 60 to the pipe 56 and thus mingles with the expanded nitrogen entering the liquefier 47. After passing about the tubes 48 of the liquefier the nitrogen which has given up some of its refrigeration is delivered through a pipe 61 and a portion is withdrawn through the pipe 62 controlled by a valve 63 to a chamber 64 at one end of the exchanger 10. After passing through the tubes 11 in heat exchange with the entering air, the nitrogen enters a chamber 65 and is withdrawn through a pipe 66. This portion of the nitrogen, having no longer any refrigerating value, may be discarded.

The remainder of the nitrogen from the pipe 61 is delivered by a pipe 67 controlled by a valve 68 to the exchanger 40, and after passing through the tubes 41 therein, it is returned through a pipe 69 to the compressor 36 for recompression and circulation in the cycle.

Under certain circumstances, it may be desirable to divert a portion of the high pressure nitrogen which has been reduced to a low temperature in the coil 43 and to expand such portion with external work. In order to provide for this contingency, a pipe 70 controlled by a valve 71 is connected to the pipe 44 and to the pipe 52. The introduction of the cold high pressure nitrogen to the expansion engine 54 permits further reduction in the temperature of the nitrogen delivered through the pipe 55 and hence maintenance of the required refrigeration in the liquefier 47.

As hereinbefore indicated, it is possible, in accordance with the procedure described, to cool the entering air to the required temperature in order that it may be liquefied in the condensers 14 and 22 without danger of premature liquefaction which would reduce the advantages of selective liquefaction in the tubes 15. The desired result is accomplished by diverting to the exchanger 10 only so much of the cold nitrogen as may be necessary to attain the desired temperature. This procedure has the further advantage that refrigeration is conserved at the point where it is most desirable, that is, the liquefier 47 where the nitrogen liquid necessary for the operation is produced. Any refrigeration available after the nitrogen vapor leaves the liquefier 47 is utilized in the exchanger 40 where the nitrogen is initially cooled. The procedure affords, therefore, the most economical operation.

The nitrogen in the refrigeration cycle does not at any point mingle with the air undergoing separation. The effluent nitrogen from the rectification is, however, mingled with the nitrogen in the independent nitrogen cycle. This does not permit hydrocarbons which may be present in the nitrogen cycle to mingle with the air undergoing separation and thus arrive at a point in the system where oxygen or products rich in oxygen are present. The liquid oxygen which accumulates in the bottom of the column 5 is free from any contaminating hydrocarbons which might have been introduced had it been necessary to employ oil in the initial compression of the air. The use of the blower obviates that possibility.

A further major advantage of the method and

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apparatus as described is that the initial pressure of the nitrogen may be relatively low as compared with pressures normally employed in commercial systems. An initial pressure of 4 atmospheres absolute requires much less power input to maintain the cycle than a pressure of 20 atmospheres absolute. Even pressures as high as 8 atmospheres absolute in the initial compression of the nitrogen afford material savings. Nevertheless it is possible to supply all of the required refrigeration by expansion of a portion of the nitrogen from its initial pressure to a pressure of approximately 1 atmosphere absolute. This insures operation on an economical basis.

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 permits the substantially total recovery of the oxygen content of the air, since the nitrogen reflux which is supplied at the top of the rectification 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 atmospheric air by liquefaction and rectification under conditions affording resistance to travel of the air which comprises compressing air to a relatively low pressure not materially in excess of that required to overcome resistance to travel, subjecting the air to liquefaction by indirect heat exchange with liquid nitrogen and subsequently to rectification to separate liquid oxygen and a gaseous effluent, compressing nitrogen, expanding a portion of the nitrogen to a lower pressure with external work, liquefying the other portion of the nitrogen by indirect heat exchange with the expanded portion and the gaseous effluent from the rectification, and thereby supplying the liquid nitrogen utilized in the liquefaction of the air.

2. The method of separating the constituents of atmospheric air by liquefaction and rectification under conditions affording resistance to travel of the air which comprises compressing air to a relatively low pressure not materially in excess of that required to overcome resistance to travel, subjecting the air to liquefaction by indirect heat exchange with liquid nitrogen and subsequently to rectification to separate liquid oxygen and a gaseous effluent, compressing nitrogen, expanding a portion of the nitrogen to a lower pressure with external work, liquefying the other portion of the nitrogen by indirect heat exchange with the expanded portion, cold nitrogen vapor from preceding liquefied portions of the nitrogen, and the gaseous effluent from the rectification, and thereby supplying the liquid nitrogen utilized in the liquefaction of the air.

3. The method of separating the constituents of atmospheric air by liquefaction and rectification under conditions affording resistance to travel of the air which comprises compressing air to a relatively low pressure not materially in excess of that required to overcome resistance to travel, subjecting the air to liquefaction by indirect heat exchange with liquid nitrogen and subsequently to rectification to separate liquid oxygen and a gaseous effluent, compressing nitrogen to a pressure not materially exceeding 5 atmospheres absolute, expanding a portion of the ni-

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trogen to a lower pressure with external work, liquefying the other portion of the nitrogen by indirect heat exchange with the expanded portion and the gaseous effluent from the rectification, and thereby supplying the liquid nitrogen utilized in the liquefaction of the air.

4. The method of separating the constituents of atmospheric air by liquefaction and rectification under conditions affording resistance to travel of the air which comprises compressing air to a relatively low pressure not materially in excess of that required to overcome resistance to travel, subjecting the air to liquefaction by indirect heat exchange with liquid nitrogen and subsequently to rectification to separate liquid oxygen and a gaseous effluent, compressing nitrogen to a pressure not materially exceeding 8 atmospheres absolute, expanding a portion of the nitrogen to a lower pressure with external work, liquefying the other portion of the nitrogen by indirect heat exchange with the expanded portion and the gaseous effluent from the rectification and thereby supplying the liquid nitrogen utilized in the liquefaction of the air.

5. The method of separating the constituents of atmospheric air by liquefaction and rectification under conditions affording resistance to travel of the air which comprises compressing air to a relatively low pressure not materially in excess of that required to overcome resistance to travel, subjecting the air to liquefaction by indirect heat exchange with liquid nitrogen and subsequently to rectification to separate liquid oxygen and a gaseous effluent, compressing nitrogen to a pressure not materially exceeding 5 atmospheres absolute, expanding a portion of the nitrogen to a lower pressure with external work, liquefying the other portion of the nitrogen by indirect heat exchange with the expanded portion, cold nitrogen vapor from preceding liquefied portions of the nitrogen, and the gaseous effluent from the rectification, and thereby supplying the liquid nitrogen utilized in the liquefaction of the air.

6. The method of separating the constituents of atmospheric air by liquefaction and rectification under conditions affording resistance to travel of the air which comprises compressing air to a relatively low pressure not materially in excess of that required to overcome resistance to travel, subjecting the air to liquefaction by indirect heat exchange with liquid nitrogen and subsequently to rectification to separate liquid oxygen and a gaseous effluent, compressing nitrogen to a pressure not materially exceeding 8 atmospheres absolute, expanding a portion of the nitrogen to a lower pressure with external work, liquefying the other portion of the nitrogen by indirect heat exchange with the expanded portion, cold nitrogen vapor from preceding liquefied portions of the nitrogen and the gaseous effluent from the rectification, and thereby supplying the

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liquid nitrogen utilized in the liquefaction of the air.

7. The method of separating the constituents of atmospheric air by liquefaction and rectification which comprises compressing nitrogen, expanding a portion of the nitrogen to a lower pressure with external work, liquefying the other portion of the nitrogen by indirect heat exchange with the expanded portion and nitrogen separated by the rectification, thereby maintaining a constant supply of liquid nitrogen to afford refrigeration, compressing air, vaporizing the liquid nitrogen by indirect heat exchange of the air therewith and rectifying the liquid obtained containing the oxygen and nitrogen constituents of the air to separate such constituents.

8. The method of separating the constituents of atmospheric air by liquefaction and rectification which comprises compressing nitrogen, expanding a portion of the nitrogen to a lower pressure with external work, liquefying the other portion of the nitrogen by indirect heat exchange with the expanded portion and nitrogen separated by the rectification, thereby maintaining a constant supply of liquid nitrogen to afford refrigeration, compressing air, vaporizing the liquid nitrogen by indirect heat exchange of the air therewith, mixing the nitrogen vapor with the expanded nitrogen and rectifying the liquid obtained containing the oxygen and nitrogen constituents of the air to separate such constituents.

9. The method of separating the constituents of atmospheric air by liquefaction and rectification which comprises compressing nitrogen to a pressure not materially exceeding 5 atmospheres absolute, expanding a portion of the nitrogen to a lower pressure with external work, liquefying the other portion of the nitrogen by indirect heat exchange with the expanded portion and nitrogen separated by the rectification, thereby maintaining a constant supply of liquid nitrogen to afford refrigeration, compressing air, vaporizing the liquid nitrogen by indirect heat exchange of the air therewith and rectifying the liquid obtained containing the oxygen and nitrogen constituents of the air to separate such constituents.

10. The method of separating the constituents of atmospheric air by liquefaction and rectification which comprises compressing nitrogen to a pressure not materially exceeding 8 atmospheres absolute, expanding a portion of the nitrogen to a lower pressure with external work, liquefying the other portion of the nitrogen by indirect heat exchange with the expanded portion and nitrogen separated by the rectification, thereby maintaining a constant supply of liquid nitrogen to afford refrigeration, compressing air, vaporizing the liquid nitrogen by indirect heat exchange of the air therewith and rectifying the liquid obtained containing the oxygen and nitrogen constituents of the air to separate such constituents.

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