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[54] **METHOD OF PRODUCING THE GASEOUS AND LIQUEFIED NITROGEN AND AN APPARATUS USED THEREFOR**

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[58] **Field of Search** 62/24, 27, 28, 29, 62/39, 31, 13, 14

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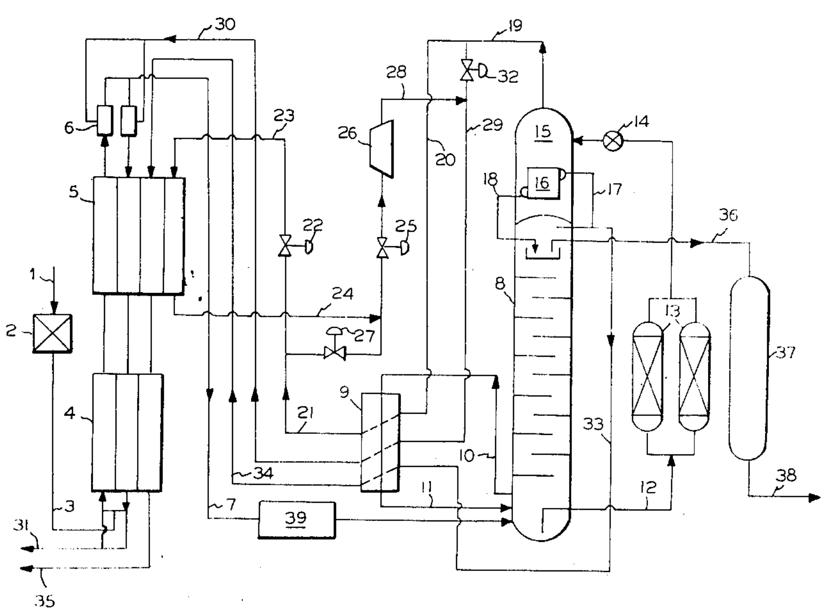
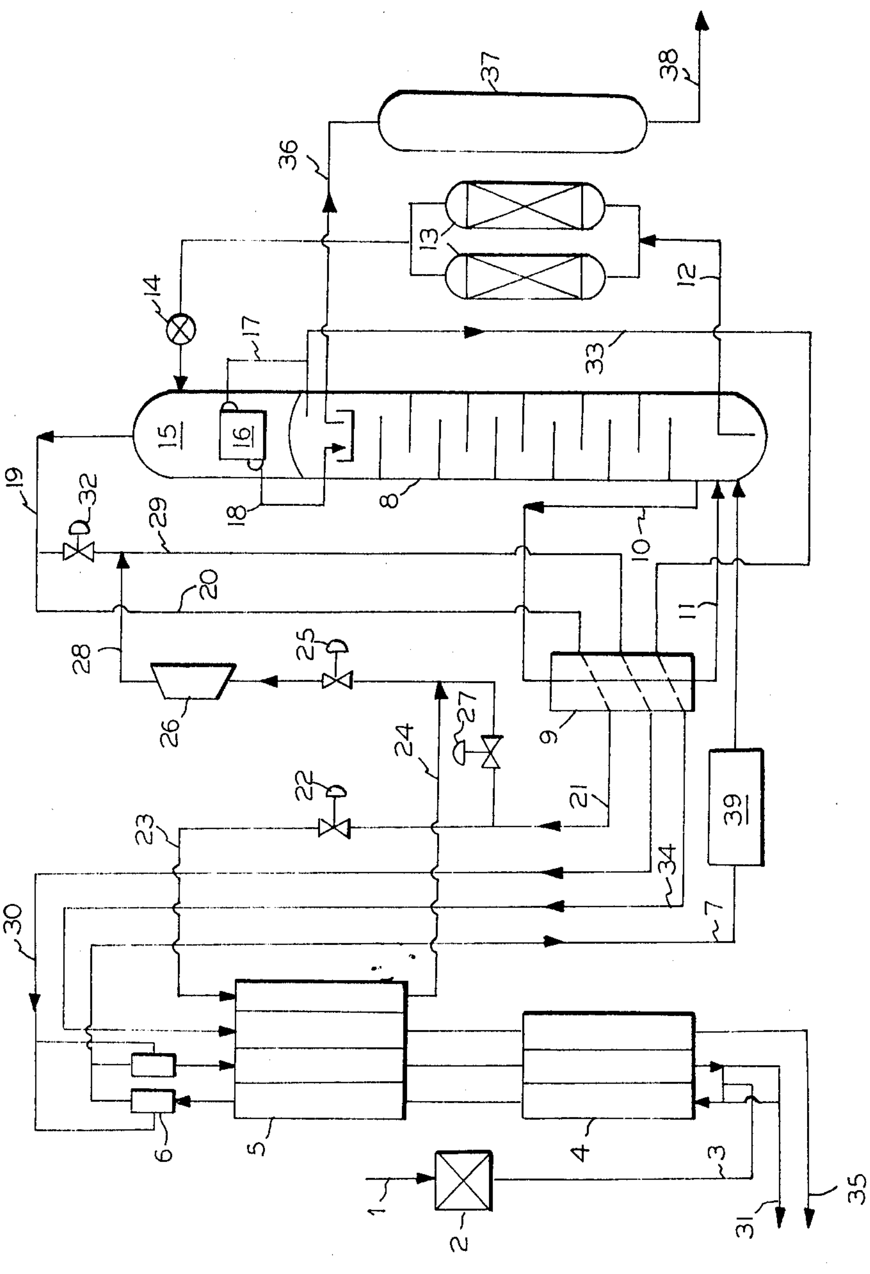
[57] **ABSTRACT**

In a method of producing gaseous and liquefied nitrogen having a pressure higher than atmospheric pressure, by the use of an apparatus comprising a reversible heat exchanger and a single column rectifier, the combination of following steps: the impure gas obtained by heat-exchanging, in a condenser-evaporator, of liquefied air with nitrogen gas, each of which has been separated in the column, is divided into two parts; one of these parts is again divided into two parts; one part of this second division is passed through a control valve and then through the heat exchanger, after which it is united and admixed with the remaining part of the gas resulting from the second division, which has passed through a control valve; this admixture is supplied to an expansion turbine; the expanded impure gas is united and admixed with the remaining part of the gas resulting from the original division, which has passed through a control valve: and this admixture, after having been passed through the air-liquefier and then the heat exchanger, is taken out of the apparatus. Adopting the abovementioned process makes it possible to industrially and advantageously produce gaseous nitrogen and liquefied nitrogen.

4 Claims, 1 Drawing Figure





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METHOD OF PRODUCING THE GASEOUS AND LIQUEFIED NITROGEN AND AN APPARATUS USED THEREFOR

This invention realtes to an improved method of producing gaseous nitrogen or liquefied nitrogen in which a reversible heat exchanger is adopted in place of a main heat exchanger; and inside the single column rectifier, the liquefaction and separation of air are performed, thereby producing the nitrogen gas and liquefied nitrogen each of which products having a high pressure which is slightly lower than the given pressure of the supplied material air.

Generally in the process of producing the gaseous and liquefied nitrogen, it has been the conventional practice that, with a single column rectifier being used therefor, the necessary amount of cold is generated by means of the intermediate pressure expansion turbine for the purpose of liquefying and separating the air, and that the nitrogen gas and liquefied nitrogen having a certain magnitude of pressure respectively are produced through the function of the single column rectifier.

According to the generally known system as above, the elimination of impurities which are contained in the atmospheric air, such as CO_2, moisture and others, has been solely dependent upon such devices as the carbon dioxide gas scrubber which uses soda solution as the cleaning agent, the molecular sieve, or the adsorbing eliminator which uses silica gel or other like adsorbent. However, it has been experienced that those eliminating and cleaning apparatuses are disadvantageous in that the system is complicated, which naturally renders the operation and maintenance work cumbersome and time-consuming; and not only that, the period of continuous operating time of the apparatus is relatively short.

The present invention offers a rationalized method of producing nitrogen of high pressure, entirely eliminating the drawbacks or disadvantages which have hitherto been encountered in practicing those systems of known devices. One feature of this invention is that the reversible heat exchanger is used for the purpose of eliminating the impurities contained in the air supplied as raw material, such as carbon dioxide and moisture; and by adopting this reversible heat exchanger, compactness of the processing system and simplification of operating and maintenance procedures have been effectively accomplished. Another noteworthy feature of this invention is a provision of such mechanism that the intermediate-pressure impure gas which has been heated up to some extent in the air-liquefier, after having left the condenser-evaporator which is attached to the single column rectifier, is introduced into the reversible heat exchanger, which gas operates as a low temperature fluid, an indispensable requisite for thorough elimination of CO_2 and other impurities which are drifting about the lower-temperature portion of the reversible heat exchanger; and therefrom, said impure gas is taken out of the middle portion of said reversible heat exchanger, and is made to mingle with the impure gas which has been divided at the discharge port of the air-liquefier; and further, said admixture of gases is subjected to the expansion turbine, where a certain amount of cold is generated for satisfying the functional need of the whole device. Through such processing mechanism as above, the elimination of CO_2 and other impurities staying around the lower-temperature portion of the reversible heat exchanger can be perfectly performed, by simply adjusting the amount of the impure gas to an optimal quantity; and also, the entire device is enabled to run a continuous operation ranging over a long period of time. Furthermore, according to the method of this invention, not only the equipment expenditures for the entire device can be lowered, but also the operating and overhead costs can be greatly economized.

Still another feature of this invention lies in the provision of three control valves. To explain in more detail, the impure gas which has passed through the air-liquefier, after having left the condenser-evaporator, is divided into two parts, one part of which is introduced into the reversible heat exchanger, and the other part of which is guided directly into the expansion turbine; and for both of those parts, control valves are provided respectively. By regulating those two control valves, the impure gas can be satisfactorily adjusted to an optimal quantity of the gas, as best suited for thorough elimination of CO_2 and other impurities staying around the lower-temperature portion of the reversible heat exchanger. There is a third control valve further provided on the by-pass which has bifurcated from the path of the impure gas which has come out of the condenser-evaporator. By coordinative regulation of the above-mentioned threefold control valves, the operating pressure of the condenser-evaporator can be maintained at an optimal level; and also, the expansion turbine can be adjusted to the condition best suited to the flow rate and the quantity of cold generated.

Hereunder will be given the detailed explanation of one preferred embodiment of this invention, reference being had to the accompanying process-flow sheet.

The accompanying drawing is a process-flow sheet which illustrates the functional phases of one preferred embodiment of the gaseous or liquefied nitrogen producing apparatus to be used for realizing the method of this invention.

As will be understood from the process-flow sheet, the air coming through the suction pipe **1** first goes into the supplied-air compressor **2**, where the air is compressed (in the case when the production of gaseous nitrogen alone is intended, the compression shall extend up to around 6 to 7 kg/cm², whereas in the case where it is intended to produce liquefied nitrogen concurrently with the gaseous nitrogen compression shall be up to around 8 to 9 kg/cm²); and then, the compressed air is directed through the conduit tube **3**, further going on to run through the higher-temperature portion **4** and the lower-temperature portion **5**, of the reversible heat exchanger respectively. Following the above, the air is subjected to heat exchanging with the returning gas, then being cooled down to the proximity of liquefying point; and furtheron, passing through the check valve **6**, conduit tube **7**, and the gas-phase adsorber **39**, the air finally enters into the bottom portion of the single column rectifier **8**. Inside of the gas-phase adsorber **39**, impurities contained in the air such as CO_2, hydrocarbon and the like, are eliminated by way of adsorption; and inside of the single column rectifier, rectification is operated in accordance with the system of the generally known art. The air is thereafter separated into two, i.e., the liquefied air which is abundant in oxygen and which is to be withdrawn from the bottom portion, and the high-purity nitrogen which is to be withdrawn from the top portion. On the single column rectifier 8, there is attached the air-liquefier 9. The gaseous air which has been taken out through the conduit tube 10 is subjected to heat-exchanging with the returning gas inside the air-liquefier 9, where the air is to be liquefied; and this liquefied gas is returned to the bottom portion of the single column rectifier 8, passing through the conduit tube 11.

The liquefied air which is rich in oxygen, and which has been collected in the bottom portion of the single column rectifier, then goes on to pass through the conduit tube 12, and enters the liquid air filter 13, where such impurities as the remainder CO_2, hydrocarbon and others, are adsorbed and eliminated; and then, while passing through the control valve 14, the liquefied air sustains pressure reduction down to around 3 to 4 kg/cm^2, and finally enters the outer shell 15 of the condenser-evaporator. Upon coming into the condenser-evaporator 16, the liquefied air is placed under heat exchanging with the highly pure nitrogen gas which has been guided down there from the top portion of the single column rectifier 8, through the conduit tube 17; and the high-purity nitrogen gas is condensed into liquid form, and is made to pass through the conduit tube 18 to return to the top portion of the single column rectifier 8, thus after all becoming the reflux which is oriented toward the receifier. On the other hand, the liquefied air is vaporized, and is withdrawn as the impure gas, from out of the conduit tube 19.

Part of the impure gas which has been taken out of the conduit tube 19 travels through the conduit tube 20 and the air-liquefier 9. After having run through the conduit tube 21 at the discharge port, part of the gas is again divided into two parts. A part of this second division goes on through the control valve 22 and the conduit tube 23, thus to be introduced, as into the lower-temperature portion 5 of the reversible heat exchanger, where said part of divided impure gas deservedly acts in compensating for the cold of sufficient amount to condense and eliminate the carbon dioxide which is contained in the supplied material air; meanwhile the impure gas itself sustains temperature rise, and is then taken out through the conduit tube 24; and thereafter going on through the inlet port control valve 25 of the expansion turbine, finally enters the expansion turbine 26.

The remaining part of the impure gas which has secondarily been divided as above described goes on through the control valve 27, and unites with part of the second division, and after passing through the control valve 25, enters the expansion turbine 26. The impure gas which has thus entered the expansion turbine 26 is made to expand up to the proximity of atmospheric pressure; and through the thermodynamic external work which itself performs, the impure gas sustains a remarkable temperature drop, thereby generating the cold of such amount as is required for operational function of the entire device. The impure gas still goes on through the conduit tubes 28 and 29, and again enters the air-liquefier 9, and further passes through the conduit tube 30 and the check valve 6, to enter the reversible heat exchangers 5 and 4; and after having entered there, the impure gas is subjected to heat exchanging with the supplied air, thereby being warmed to room temperature, and finally being discharged outside the apparatus, through the conduit tube 31.

The remaining part of the impure gas which has come out of the condenser-evaporator and then been divided, first travels through the by-pass control valve 32, the travelling part unites with the impure gas that has come out of the expansion turbine. This by-pass control valve 32 acts in such way that it co-operates with the control valves 22 and 27, just as described in the foregoing, in taking the role of regulating various factors such as the operating pressure of the condenser-evaporator 16, and the flow rate of the expansion turbine 26 together with the cold-generating amount in said expansion turbine. In other words, by adjusting the opening of the by-pass control valve 32, the flow rate of the impure gas that travels through the conduit tube 20 towards the expansion turbine can be adjusted; and further, by the mutual interaction between the control valve 22 which is provided on the passage which guides the impure gas to the heat exchanger and the control valve 27 which is provided on the passage which guides said impure gas directly to the expansion turbine 26, the flow rate of the passage which is the heating passage for said impure gas and the rate of flow which is directly supplied to the expansion turbine are both to be controlled; and in turn, the inlet-port temperature of the expansion turbine 26 as well as the outlet temperature can be controlled; and at the same time, the cold generating quantity can be controlled. It is further to be noted that, through the accomplishment of the control of pressure inside the outer shell 15 of the condenser-evaporator so achieved by the combined actions of the aforementioned three valves 32, 22 and 27, the temperature of impure gas inside said outer shell 15 can be controlled. Also, it can be concluded that the pressure inside the single column rectifier 8 can be controlled using the aforementioned three valves 32, 22 and 27, because the pressure of the pure nitrogen gas, which is to be liquefied through heat-exchanging by means of the condenser-evaporator 16 with said impure gas, will change depending upon the temperature of the impure gas inside said outer shell 15.

In the next place, the high-purity nitrogen gas product which has been separated at the top portion of the single column rectifier 8 travels through the conduit tube 33; and then, passing through the air-liquefier 9, the conduit tube 34, and the reversible heat exchangers 5 and 4, in succession, the nitrogen product is warmed to room temperature and is further sent out through from the conduit tube 35, then being at a pressure which is slightly lower than the given pressure of the supplied air.

On the other hand, the high-purity liquefied nitrogen product which has been separated at the top portion of the single column rectifier 8 is withdrawn outside after having passed through the conduit tube 36, the liquefied-nitrogen weighing tank 37, and the conduit tube 38, in succession.

In the case where it is intended to produce the gaseous nitrogen only, in accordance with the method of this invention, the apparatus is operated at the level of given pressure of the supplied air, of the order of 6 to 7 kg/cm^2; whereas in such case that simultaneous production of both gaseous nitrogen and liquefied nitrogen is intended, the respective control valves are to work for regulation and adjustments just as described in the foregoing; and under that condition, the flow rate of the expansion turbine is to be raised to a required higher rate, as compared to the case of producing gaseous nitrogen only; and the pressure on the side of the impure gas inside the condenser-evaporator is also to be raised; and in turn, the operating pressure of the single column rectifier and the given pressure of the supplied air are both raised to an appreciably higher level (around 8 to 9 kg/cm^2), for securing the perfect operation of the apparatus.

According to the method of this invention, therefore, the production of gaseous nitrogen only and the simultaneous production of both gaseous and liquefied nitrogen can be optionally and alternately achieved, through a simple operational manipulation; and the desired object of production can be effectively attained by using the simple and compact apparatus which embodies the producing method of this invention.

What is claimed is:

1. A method of producing gaseous and liquefied nitrogen having a pressure higher than atmospheric pressure which comprises cooling compressed air down to approximately its liquefying point by passing the compressed air through a reversible heat exchanger, introducing the air into a single column rectifier, where it is separated into liquefied air rich in oxygen and nitrogen, removing the gaseous air from the column, liquefying the gaseous air in an air-liquefier, returning the liquefied air into the column, withdrawing nitrogen gas from the top portion of the column, introducing at least a part of the withdrawn nitrogen gas into a condenser-evaporator and liquefying it by means of heat-exchange with the liquefied air, which has been withdrawn from the bottom portion of the column and introduced into the outer shell of the condenser-evaporator, returning the liquefied nitrogen into the column, removing part of the liquefied nitrogen from the system, dividing impure gas obtained from the liquefied air as a result of said heat-exchange into two original parts, dividing one of these original parts into two additional parts, passing one of the additional parts through a control valve and then through the reversible heat exchanger, uniting and admixing the resultant material with the remaining additional part of gas, which itself has passed through a control valve, introducing this admixture into an expansion turbine to generate cold of an amount necessary for actuating the system, uniting and admixing the expanded impure gas with the remaining original part of the gas which itself has passed through a control valve, passing this admixture thorugh the air liquefier and then the heat exchanger and removing the resultant material from the system.

2. A method according to claim **1**, wherein prior to dividing one of the original parts of impure gas into the two additional parts of impure gas, said original part is passed through the air-liquefier.

3. A method according to claim **1**, wherein after admixture of the two additional parts of gas, the resultant mixture is passed through a control valve prior to introduction into the expansion turbine.

4. A method according to claim **2**, wherein after admixture of the two additional parts of gas, the resultant mixture is passed through a control valve prior to introduction into the expansion turbine.

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