

[54] **PRODUCTION OF NITROGEN AND ARGON-FREE OXYGEN**
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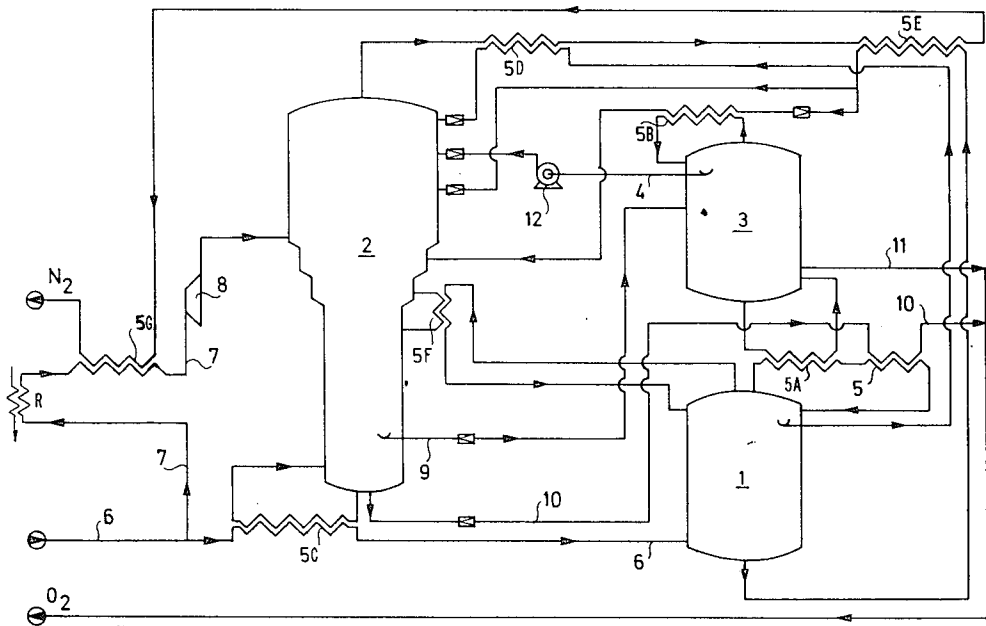
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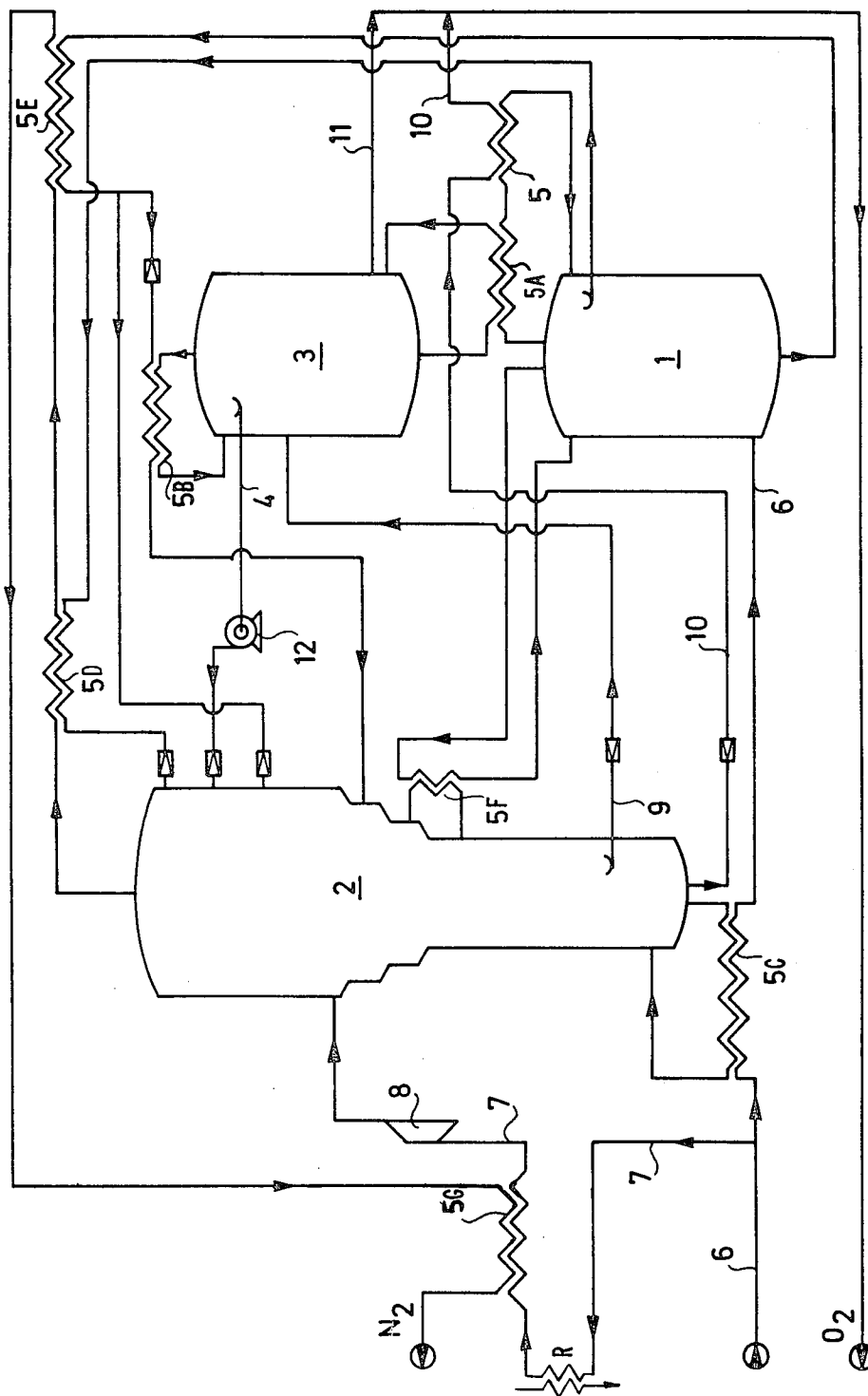
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[58] **Field of Search**.....**62/22, 23, 24, 27, 28, 29, 62/30, 38, 26, 41**

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[57] **ABSTRACT**
Nitrogen- and argon-free oxygen is recovered from air by a three-stage rectification system in which the third stage is maintained at an absolute pressure of 0.8 to 1.1 atmosphere while the pressure of the second stage is higher by only 0.3 to 0.5 atmosphere. A side stream containing argon flows from the second rectifying stage to the third stage which yields a head product enriched in argon. This head product is pumped back into the upper portion of the second stage. High-purity oxygen is recovered from the sumps of the second and third rectifying stages.

5 Claims, 1 Drawing Figure





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PRODUCTION OF NITROGEN AND ARGON-FREE OXYGEN

BACKGROUND OF THE INVENTION

This invention relates to a process for the recovery of nitrogen- and argon-free oxygen by multi-stage rectification of air. In the first rectifying stage, compressed air cooled to the dew point is separated into nitrogen and an oxygen-enriched fraction. The second stage yields a sump product of oxygen which is practically free of argon and nitrogen. A side product of the second rectifying stage, containing argon, is separated in the third stage into an argon-free sump product and an argon-enriched head product. The argon-free oxygen product is withdrawn partly from the sump of the second rectifying stage and partly from the sump of the third rectifying stage.

It is a known practice, in these and other rectifying processes, to apply the principle of the heat pump and thus use the feed air as a working fluid. For the recovery of technical grade oxygen and nitrogen, modern production plants are using two-stage rectifying processes. More than two stages are usually provided if one of the components of the air is to be recovered at particularly high purity, or if one of the rare gas components of air is to be separated as an additional product.

The work expenditure for separating air into oxygen and nitrogen is five to ten times greater than theoretically calculated with a reversible separation process. This is due to losses during compression, heat leak, less than perfect heat exchange, pressure drop across pipes and valves, as well as losses in the rectifying columns. During the last few decades, entropy studies led to numerous improvements in the design of compressors, turbines, heat exchangers, and rectifying columns, as well as process improvements. For instance, Lachmann suggested blowing part of the feed air into the second-stage rectifying column at a pressure slightly above atmospheric, thus reducing the work expenditure. It was also Lachmann's idea, to vent from the second-stage rectifying column an amount of vapor equivalent to 10-15 percent of the feed air volume. This measure is taken not for conservation of energy but for product purity since the venting removes a considerable amount of the undesired argon, however not without loss of product.

Already a few decades ago, the development of air separation for the recovery of nitrogen and oxygen led, after limitation of some of the irreversibilities, to the so-called medium pressure process which operates with two rectifying stages: a medium pressure and a low pressure stage. Since this process is being applied so frequently and the corresponding plants are being built again and again, practically without changes, they have been labelled "classic" in the industry. The fact that these plants continue to be copied signifies that the industry has become resigned to the necessary energy consumption.

To recover part of the argon initially contained in the air, this classic process, as known, was modified to a three-stage rectifying process. In this process, the pressures maintained in the medium and low pressure stages are the same as in the classic process. The pressure in the argon rectifying stage corresponds approximately to the pressure in the low pressure stage. The

argon recovery is about 30 to 60 percent. A mixture of about 45 percent argon, 50 percent oxygen, and 5 percent nitrogen leaves the head of the argon column. The energy consumption for this process is about the same as for the classic process which is considered a special advantage.

SUMMARY OF THE INVENTION

According to this invention, the energy consumption for air separation can be lowered considerably by using, in the third rectifying stage, a rectifying pressure which is lower by 0.3 to 0.5 atm (atmosphere) than the pressure in the second rectifying stage, and by withdrawing the head product of the third rectifying stage and pumping it into the top portion of the second rectifying stage at a point which is already low in oxygen concentration.

Consequently, contrary to the classic two-stage process and the conventional three stage process, the first rectifying stage also can operate at a lower pressure. The compression pressure of the feed air can be 1.5 atm lower than usual. The energy saving, based on gaseous product, is approximately 10 percent and remains significant, even after mathematical conversion to the product data of the classic process. Compared to the conventional three-stage process, oxygen losses are reduced considerably. It is particularly surprising that, in spite of the reduced rectifying pressures, an argon-free oxygen product of 99.5 percent purity is recovered.

Although, in the third rectifying stage, a mixture containing argon is separated into an argon-free and an argon-rich fraction, —which is in itself a known practice —this measure does not serve for the recovery of argon but for facilitating the discharge of argon from the second rectifying stage and is a means of lowering the pressure level.

BRIEF DESCRIPTION OF THE DRAWING

The single FIGURE of the accompanying drawing is a schematic flow diagram illustrating a preferred embodiment of the invention.

DESCRIPTION OF PREFERRED EMBODIMENT

The first rectifying stage is represented by column 1, the second stage by column 2, and the third stage by column 3. The major portion of the compressed, pre-cooled air enters rectifying column 1 through line 6 and is separated into nitrogen and an oxygen-enriched fraction. The sump of the second rectifying column 2 yields a practically argon- and nitrogen-free oxygen product. A side stream 9, containing argon, is withdrawn from rectifying column 2 and separated in rectifying column 3 into an argon-free oxygen sump product and an argon-rich head product. The argon-free oxygen product is withdrawn partly through line 10 from the sump of column 2 being desirably passed in heat exchange with the head product of column 1 in heat exchanger 5, and partly through line 11 from the sump of column 3.

The rectifying pressure in column 3 is lower by 0.3 to 0.5 atm than the pressure in column 2. The head product of column 3 is withdrawn through line 4 and introduced by pump 12 into the top portion of column

2 at an upper level which is already low in oxygen concentration.

The drawing also shows how the heat input and heat withdrawal at column 3, and the heat input to column 2 can be achieved in an advantageous manner. There is indirect heat exchange between the sump liquid of column 3 and the head product from column 1 in heat exchanger 5A, and between the head product of column 3 and the sump product of column 1 in heat exchanger 5B, as well as between the sump liquid of column 2 and the air feed in heat exchanger 5C. It is expedient to operate heat exchanger 5C at the sump of column 2 by means of a control device so that the sump product just remains free of nitrogen. This results in increased argon concentration in side stream 9.

It is furthermore advantageous to operate column 3 at an absolute pressure between 0.8 and 1.1 atm.

ILLUSTRATIVE EXAMPLE

In a system conforming to the drawing and designed to process air fed at the rate of 100 nm³/hr (cubic meters per hour measured at normal conditions), an absolute pressure of 1 atm is maintained in column 3, while the absolute pressure in column 2 is 1.5 atm and 4.2 atm in column 1. The pressure is determined directly above the sump liquid in each column.

Feed air, cooled to a temperature of -176° C and at an absolute pressure of 4.24 atm, is supplied to the process via line 6. A partial air stream of 13 nm³/hr is passed via line 7 through a conventional reheater R and heat exchanger 5G to expansion engine 8 and fed to column 2 at a temperature of -168° C. After passing through heat exchanger 5C at the sump of column 2, the main air stream enters column 1 at -178° C. From the head of column 1, 32.8 nm³/hr of overhead vapor, after heat exchange with the head product of column 2 in heat exchanger 5D, flows into the upper portion of column 2. The inlet temperature is -189° C. From the sump of column 1, liquid at a rate equivalent to 54.2 nm³/hr, after heat exchange with the head product of column 2 in heat exchanger 5E, flows to column 2; a part of this stream equivalent to 32 NM³/hr flows through condenser 5B of column 3 before entering column 2 at approximately the center, whereas the remainder of the stream equivalent to 22.2 nm³/hr enters a column 2 at a higher level. At the head of column 1, a temperature of -181° C is maintained. This is accomplished by heat exchange of the head product with the sump liquid of column 3 in heat exchanger 5A and by heating a partial head product recycle stream of 4.8 nm³/hr with liquid from column 2 in heat exchanger 5F.

From column 2, a side stream of 14.2 nm³/hr, containing argon, is fed via line 9 to column 3. In column 3, a head temperature of -185.5° C and a sump temperature of -183° C are maintained. A head product of 2 nm³/hr from column 3 is pumped into the upper por-

tion of column 2 by way of line 4 and pump 12. Liquid product is withdrawn at a rate equivalent to 7.8 nm³/hr from the sump of column 2 by line 10, and at a rate of 12.2 nm³/hr as 99.5 percent pure oxygen from column 3 by line 11. The argon-contaminated nitrogen leaves overhead of column 2 at a rate of 80 nm³/hr. The oxygen product is recovered by way of lines 10 and 11 at -183° C and atmospheric pressure, and the nitrogen at -178° C and at an absolute pressure of 1.2 atm after leaving heat exchanger 5G.

The pressures of the process may be varied within the given limits. This process however is also suitable for application of the method of balancing refrigeration losses in the classic process by work-performing nitrogen expansion. The recovery of pure nitrogen is not shown in the flow diagram or described in the process example but could be done in the conventional manner without essentially changing the pressure conditions.

What is claimed is:

1. A process for the recovery of substantially nitrogen-and argon-free oxygen by rectification of air in three rectifying stages, which comprises separating air into a nitrogen fraction and an oxygen-enriched fraction in the first rectifying stage, passing entirely said nitrogen fraction to the top portion of the second rectifying stage and said oxygen-enriched fraction to the middle portion of said second stage, passing an argon-containing stream from the bottom portion of said second stage to the middle portion of the third rectifying stage, pumping entirely an argon-rich head product from said third stage to said second stage at a level between the introduction of said nitrogen fraction and said oxygen-enriched fraction, the pressure of said third stage being lower by 0.3 to 0.5 atmosphere than the pressure of said second stage, discharging a nitrogen head product from said second stage, and recovering said oxygen from the bottom portions of said second and third stages.

2. The process of claim 1 wherein there is indirect heat exchange between the nitrogen fraction of the first rectifying stage and the sump liquid of the third rectifying stage and between the oxygen-enriched fraction of said first stage and the argon-rich head product of said third stage.

3. The process of claim 1 wherein the third rectifying stage is maintained at an absolute pressure in the range of about 0.8 to 1.1 atmosphere.

4. The process of claim 1 wherein there is indirect heat exchange between the nitrogen fraction of the first rectifying stage and a recycle stream from the middle portion of the second rectifying stage.

5. The process of claim 4 wherein the third rectifying stage is maintained at an absolute pressure in the range of about 0.8 to 1.1 atmosphere.

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