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Description

Process for producing ultrapure nitrogen

The invention relates to a process for producing ultrapure nitrogen by low-temperature fractionation of air in a rectification system having at least one rectification column, in which process compressed and purified feed air is conducted into a pressure column, an oxygen-free pressurized nitrogen fraction is produced from the pressure column, conducted into a low-pressure column and in the low-pressure column carbon-monoxide-free low-pressure nitrogen is produced as overhead gas.

The German Patent Application having the file no. 198 06 576 which is scheduled for publication discloses a process according to the preamble of Claim 1. An oxygen-free pressurized nitrogen fraction is taken off from a pressure column, the pressure column having at least 160 theoretical plates in order, in addition, to take off the pressurized nitrogen from this pressure column carbon-monoxide-free. However, the energy requirement for this, despite the 160 theoretical plates, is still very high. It is also a disadvantage that a majority of the ultrapure nitrogen is produced in the gaseous state.

It is therefore an object of the invention to avoid this disadvantage and to decrease the energy requirement.

A further object of the invention is to describe a process which is optionally operated with a liquid pressurized nitrogen fraction directly from the pressure column or from a tank containing liquid pressure-column nitrogen.

These objects are achieved according to the invention by a process having the features of Claim 1. Embodiments of the invention are subject-matter of the subclaims.

It is characteristic of the invention that the oxygen-free pressurized nitrogen fraction is either taken off in the liquid state from an upper region of the pressure column or is provided from a liquid tank containing stored oxygen-free nitrogen and that the oxygen-free pressurized nitrogen fraction is expanded into the bottom-heated low-pressure column, ascending vapour being formed in the low-pressure column and being freed from carbon monoxide using a reflux of ultrapure nitrogen applied at the top of the low-pressure column, being taken off at the top of the low-pressure column as carbon-monoxide-free overhead gas and after a pressure increase being partly liquefied and the liquefied part being expanded into a bottom-heated He-Ne-H_2 column from which the ultrapure nitrogen is taken off in the liquid state.

By means of the process according to the invention the ultrapure nitrogen is produced in the liquid state and can in this form be more readily transported and therefore more simply distributed to clients. The process, furthermore, is universally useable. This is because it can be used in connection with an air fractionation plant (with or without liquid nitrogen tank) or only with a liquid nitrogen tank (with spatial separation from the air fractionation plant). At an air fractionation plant having a liquid nitrogen tank, in addition, the ultrapure liquid nitrogen can be produced without the air fractionation plant being in operation.

In an embodiment of the process of the invention, the carbon-monoxide-free overhead gas and an overhead gas

of the He-Ne-H_2 column can be combined to form a carbon-monoxide-free cold gas stream.

Alternatively, the carbon-monoxide-free overhead gas can be partially liquefied in a condenser/evaporator against a liquid refrigerant which is vaporized in the course of this and a gas stream which is produced in the vaporization of the liquid refrigerant and an overhead gas of the He-Ne-H_2 column can be combined to form a carbon-monoxide-free cold gas stream.

Advantageously in both cases, the carbon-monoxide-free cold gas stream is warmed in a heat exchanger, compressed and cooled again in countercurrent in the same heat exchanger and one part is then used in the low-pressure column and another part is used in the He-Ne-H_2 column for heating the bottoms and is liquefied in the course of this, except for residual streams remaining in the gaseous state, and is fed as the liquid refrigerant to the condenser/evaporator.

The streams which are liquefied in the heating of the He-Ne-H_2 column and the low-pressure column can be fed as reflux to the He-Ne-H_2 column.

In the case of the use of a condenser/evaporator which is described above as an alternative, using the streams which are liquefied in the heating of the He-Ne-H_2 column and the low-pressure column, the carbon-monoxide-free overhead gas of the low-pressure column can be liquefied in the evaporator/condensor of the low-pressure column, except for a residual stream, and can be fed at least in part as reflux to the He-Ne-H_2 column. Counteracting the disadvantage of an additional capital cost of the condenser/evaporator is then the advantage, which is not to be underestimated, that in the case of air leaks during the compression of the

carbon-monoxide-free gas stream, no contamination of the ultrapure liquid nitrogen can occur.

Ultrapure nitrogen can be withdrawn in the liquid state from the He-Ne-H_2 column and produced in part as the reflux of the low-pressure column and in part as liquid ultrapure nitrogen product.

The ultrapure liquid nitrogen product can be fed to a product tank.

The ultrapure liquid nitrogen product can be pressurized using a pump, vaporized utilizing the cold content in the production of the oxygen-free pressurized nitrogen fraction, warmed and fed as gaseous pressurized product for use.

In this case, gaseous ultrapure nitrogen product can also be produced by the process for producing ultrapure liquid nitrogen product and in this case the cold of the originally present liquid product can be expediently utilized.

The invention is described in more detail with reference to five embodiments using five figures.

Figure 1 shows a process according to the invention with provision of oxygen-free nitrogen for the process from a liquid nitrogen tank.

Figure 2 shows a process according to the invention with provision of oxygen-free nitrogen for the process from the pressure column of a rectification system.

Figure 3 shows a process as in Figure 1, but having an additional evaporator/condenser.

Figure 4 shows a process as in Figure 2 having an additional evaporator/condenser.

Figure 5 shows a process as in Figure 2 for the case of retrofitting a rectification system with the process according to the invention.

Equivalent process streams and process steps are provided with identical reference numbers in Figures 1 to 5.

Figure 1 diagrammatically shows an embodiment of the process of the invention for producing ultrapure nitrogen. A liquid pressurized nitrogen fraction 1, which is oxygen-free except for a residual content in the range of a few mol ppm is expanded into a heated low-pressure column 2 which is operated at a pressure between 4.5 and 5.5 bar. Vapour ascending in the low-pressure column 2 is freed, by ultrapure nitrogen 3 from an He-Ne-H_2 column 4 which is applied as reflux at the top, from carbon monoxide and thus from impurities boiling higher than carbon monoxide such as argon and the residual content of oxygen, depending on the purity requirement, except for a few mol ppb. The carbon monoxide-free overhead gas 5 and an overhead gas 6 of the He-Ne-H_2 column are combined to form a carbon-monoxide-free cold gas stream 7, warmed in a heat exchanger 8 and, after a compression 9, cooled again in the same heat exchanger 8. One part 11 of the cooled gas stream 10 is used in the low-pressure column 2 and another part 12 is used in the He-Ne-H_2 column 4 for heating 13, 14 the bottoms and is liquefied in the course of this, except for residual streams 15, 16 remaining in the gaseous state. The liquefied streams 17, 18 are fed as reflux to the He-Ne-H_2 column 4. From the He-Ne-H_2 column 4, carbon-monoxide-free ultrapure nitrogen 19 is withdrawn in the liquid state, which nitrogen 19 then also contains the lower-boiling neon

and certainly the still lower-boiling constituents hydrogen and helium, depending on the requirement, in the order of magnitude of a few mol ppb. One part 3 of the liquid ultrapure nitrogen 19 is used as reflux to the low-pressure column 2 and another part is produced as liquid ultrapure liquid nitrogen product 20 and fed to a product tank 21.

In the embodiment of the process according to the invention as in Figure 1, the liquid pressurized nitrogen fraction 1 which is used as feed is taken off from a liquid tank 22 via a pressure boosting pump 23 and conducted via a heat exchanger 24 into the low-pressure column 2. In the heat exchanger 24, the ultrapure liquid nitrogen product 20 is subcooled and then expanded without gaseous expansion losses into the product tank 21. Because of the subcooling of the liquid nitrogen product 20, the product tank 21 can be designed as a liquid tank operated at atmospheric pressure.

Figure 2 shows diagrammatically the production of the ultrapure liquid nitrogen product 20 as in Figure 1. The liquid nitrogen fraction 1 used as feed is, unlike the embodiment according to Figure 1, taken off from a pressure column 25 of a rectification system and conducted via a heat exchanger 24 to the low-pressure column 2. The ultrapure liquid nitrogen product 20 is pressurized by a pump 26 passed in countercurrent to the liquid nitrogen fraction 1 through the heat exchanger 24 and used with utilization of the cold content in a condenser 27 and in a heat exchanger 28 in the production of the oxygen-free pressurized nitrogen fraction, in the course of this being evaporated, warmed and fed as gaseous ultrapure pressurized product 29 for further use.

Figure 3 diagrammatically shows an embodiment of the process according to the invention with use of a condenser/evaporator 30. Unlike the embodiment according to Figure 1, in the embodiment according to Figure 3 the carbon-monoxide-free overhead gas 5 of the low-pressure column 2 is liquefied (against a liquid refrigerant 31 which is formed by combining the liquefied streams 17, 18 and evaporates in the course of this) in the condenser/evaporator 30, except for a residual stream 32 remaining in the gaseous state, and is used as reflux 33 in the He-Ne-H_2 column 4. A gas stream 5' formed in the vaporization of the liquid refrigerant and the overhead gas 6 of the He-Ne-H_2 column 4 are combined to form the cold carbon-monoxide-free gas stream 7.

Although additional capital costs are associated with the condenser/evaporator 30, the columns 2, 4 are decoupled so that even in the case of an air leak occurring during the compression into the carbon-monoxide-free gas 7, 10 the He-Ne-H_2 column 4 and thus the ultrapure nitrogen product 20 are not contaminated.

Figure 4 diagrammatically shows an embodiment of the process according to the invention as in Figure 2, but implemented with the condenser/evaporator 30 as in Figure 3.

Figure 5 diagrammatically shows that the process according to the invention can be used on an existing rectification system. Starting from the embodiment of the process as in Figure 2, the condenser 27 is supplemented on the rectification system and the heat exchanger 8 receives two additional passages for the utilization of the cold of the ultrapure nitrogen 20', which is to be warmed, during the cooling and liquefaction of a partial stream 34 of the air 35 for the rectification system. The ultrapure nitrogen which

is warmed in this case in the heat exchanger 8 is fed as gaseous ultrapure pressurized product 29 for further use.

A shared feature of the embodiment of the process according to the invention according to Figures 1 to 5 is that all residual gas streams arising (15, 16, 36 in Figures 1, 2 and 5; 15, 16, 32 and 36 in Figures 3 and 4) are combined to form a cold residual gas stream 37 in the heat exchanger 8, are warmed and are passed as impure gas 38 to the atmosphere.

Example:

m^3 in this example denotes: m^3 under standard conditions at 0°C and 1.0133 bar; i.e. 1 m^3 is equivalent to 1.25 kg.

Using the process according to the invention in the embodiment of Figure 1, 1500 m^3/h of ultrapure liquid nitrogen containing 20 mol ppb of carbon monoxide are produced from 1750 m^3/h of liquid nitrogen containing 1 mol ppm of carbon monoxide. In this case, 100 m^3/h of losses are produced during compression and 150 m^3/h of residual gas are produced. 12,500 m^3/h of nitrogen are compressed from 6.5 to 7 bar; the pressure boosting pump operates between 1 bar at the inlet and 7 bar at the outlet.

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Patent Claims

1. Process for producing ultrapure nitrogen by low-temperature fractionation of air in a rectification system having at least one rectification column, in which process compressed and purified feed air is conducted into a pressure column, an oxygen-free pressurized nitrogen fraction is produced from the pressure column, conducted into a low-pressure column and in the low-pressure column carbon-monoxide-free low-pressure nitrogen is produced as overhead gas, characterized in that the oxygen-free pressurized nitrogen fraction is either taken off in the liquid state from an upper region of the pressure column or is provided from a liquid tank containing stored oxygen-free nitrogen and in that the oxygen-free pressurized nitrogen fraction is expanded into the bottom-heated low-pressure column, ascending vapour being formed in the low-pressure column and being freed from carbon monoxide using a reflux of ultrapure nitrogen applied at the top of the low-pressure column, being taken off at the top of the low-pressure column as carbon-monoxide-free overhead gas and after a pressure increase being partly liquefied and the liquefied part being expanded into a bottom-heated He-Ne-H_2 column from which the ultrapure nitrogen is taken off in the liquid state.

2. Process according to Claim 1, characterized in that the carbon-monoxide-free overhead gas and an overhead gas of the He-Ne-H_2 column are combined to form a carbon-monoxide-free cold gas stream.

3. Process according to Claim 1, characterized in that the carbon-monoxide-free overhead gas is

partially liquefied in a condenser/evaporator against a liquid refrigerant which is vaporized in the course of this and in that a gas stream which is produced in the vaporization of the liquid refrigerant and an overhead gas of the He-Ne-H_2 column are combined to form a carbon-monoxide-free cold gas stream.

4. Process according to Claim 2 or 3, characterized in that the carbon-monoxide-free cold gas stream is warmed in a heat exchanger, compressed and cooled again in countercurrent in the same heat exchanger and one part is then used in the low-pressure column and another part is used in the He-Ne-H_2 column for heating the bottoms and is liquefied in the course of this, except for residual streams remaining in the gaseous state, and is fed as the liquid refrigerant to the condenser/evaporator.

5. Process according to Claims 2 and 4, characterized in that the streams which are liquefied in the heating of the He-Ne-H_2 column and the low-pressure column are fed as reflux to the He-Ne-H_2 column.

6. Process according to Claims 3 and 4, characterized in that using the streams which are liquefied in the heating of the He-Ne-H_2 column and the low-pressure column, the carbon-monoxide-free overhead gas of the low-pressure column is liquefied in the evaporator/condenser of the low-pressure column, except for a residual stream and is fed at least in part as reflux to the He-Ne-H_2 column.

7. Process according to one of Claims 1 to 6, characterized in that ultrapure nitrogen withdrawn in the liquid state from the He-Ne-H_2 column is

produced in part as the reflux of the low-pressure column and in part as liquid ultrapure nitrogen product.

8. Process according to Claim 7, characterized in that the ultrapure liquid nitrogen product is fed to a product tank.

9. Process according to Claim 7, characterized in that the ultrapure liquid nitrogen product is pressurized using a pump, vaporized utilizing the cold content in the production of the oxygen-free pressurized nitrogen fraction, warmed and fed as gaseous pressurized product for use.