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(54) **Eljárás és berendezés parciális kondenzáció elvégzésére**

Process and devices for partial condensation

The invention relates to a process and also to a device for partial condensation, in which a gas stream that is loaded with a substance that is to be condensed is cooled in a cryocondenser by heat exchange with a cooling medium and thereby the substance present in the gas stream is at least partially condensed.

Impurities can be deposited from gases owing to condensation or freezing. By "gases" are here meant generally exhaust air, gas or process off-gas streams that can consist of vapour, vaporous substances or gases. In particular, they are also taken to mean exhaust air streams relatively highly loaded with solvents which can form, for example, in processes in chemistry, or as off-gases from tank farms. The expression "impurities" is here taken to mean not only harmful materials contaminating the gas and that are to be disposed of, but also solvents or other substances which can be fed as valuable materials to a further use.

For this purpose, heat exchangers are used in the prior art, in which the gas stream that is to be purified is brought into contact with heat exchange surfaces that can be designed in various ways and which are cooled by means of a cooling medium. If in this case the temperature falls below the dew point of the impurities present in the gas stream, at least a part of these substances liquefies or solidifies and can be separated off from the carrier gas stream.

DE 19517273 C1 discloses a process and also a device for off-gas purification with heat exchangers, in which, in a first heat exchanger, the process gas is purified in the above-described manner. In order to prevent the exiting gas being recontaminated with condensate, a second heat exchanger is used, in which the gas stream is conducted in such a manner that it comes into contact with the heat exchange surfaces in an upwardly directed flow. Via this arrangement, the condensate collects in the entrance region of the second heat exchanger and can thus be readily removed.

EP 1 602 401 A1 does not describe heat exchange of the cooling medium fed to the cryocondenser with the cooling medium removed from the cryocondenser and therefore cannot impair the patentability of the claims now presented.

WO 95/24585 describes an arrangement of at least two heat exchangers. In contrast to the invention, therefore, in the document, the warm cooling gas used in the second heat exchanger to vaporize the cooling gas is not recirculated to the first heat exchanger and brought into heat exchange with the product stream in parallel to the primary cooling medium, but in serially arranged heat exchangers.

The fraction of the amount of substance separated off from the carrier gas compared with the total amount of substance present in the gas, is firstly dependent on the degree by which the temperature falls below the dew point. In the cooling process, however, generally oversaturation of the gas stream occurs, and aerosols are formed. Since the aerosols are deposited only to a very small fraction in the heat exchanger, the amount of substance actually separable off from the carrier gas stream is less than the theoretically achievable deposition on the basis of the temperature-dependent vapour pressures of the substances. The aerosols thus form a considerable fraction of the impurities leaving the condenser with the carrier gas stream.

It is therefore an object of the present invention to avoid the formation and propagation of aerosols in the process gas.

This object is achieved in a process of the type mentioned at the outset in that the cooling medium that is to be fed to the cryocondenser is warmed and/or vaporized by heat contact with a heat transfer medium before said cooling medium is fed to the cryocondenser. Before it is fed to the cryocondenser, the cooling medium in this case is vaporized and/or prewarmed to a temperature value which approximates the temperature of the gas stream in the cryocondenser that is loaded and is to be purified.

The concept underlying the invention is that the formation of aerosols is less pronounced the smaller is the difference between the temperature of the cooling medium and the temperature of the gas stream in the cryocondenser. "Cryocondenser" is taken to mean in this case any appliance which permits the transfer of heat energy from a gas stream that is loaded with a substance that is to be condensed to a cold fluid cooling medium. The warming according to the invention of the cooling medium before feed thereof into the cryocondenser is therefore targeted at producing in the cryocondenser only a small temperature difference between the gas stream that is to be purified and the cooling medium and thus to effectively suppress the aerosol formation. The process according to the invention also avoids unwanted ice formation, i.e. the formation of solid condensate.

As heat transfer medium for warming and/or vaporizing the cooling medium to be fed to the cryocondenser, for example, in this case a gas from a mains grid or a tank can be used. Preferably, however, as heat transfer medium the cooling medium is used that was already used in the cryocondenser for cooling the gas stream that is to be purified and has been discharged from the cryocondenser. This cooling medium in this case can be used completely, in part and/or mixed with gas from a mains grid or a tank for warming and/or vaporizing the cooling medium that is to be fed to the cryocondenser. Using the cooling medium that is discharged from the cryocondenser for prewarming the cooling medium to be fed to the cryocondenser ensures already for thermodynamic reasons that the temperature of the cooling medium fed to the cryocondenser is always somewhat lower than the gas stream in the cryocondenser that is to be purified.

For transfer of the heat energy from the discharged cooling medium to the cooling medium that is to be fed to the cryocondenser, various possibilities are conceivable, such as, for example, mixtures of matter or indirect heat transfers via a third medium. Preferably, the cooling medium that is to be fed to the cryocondenser, however, is brought into immediate thermal contact in a cooling medium heat exchanger with the cooling medium that is discharged from the cryocondenser.

A further improved energy balance can be achieved by the gas stream in the cryocondenser that is to be purified being further brought into thermal contact with the cooling medium that previously was conducted through the cooling medium heat exchanger where it was in heat exchange with the cooling medium that is to be fed for the first time to the cryocondenser. The amount of the cooling medium to be used in total can be markedly decreased thereby.

Expediently, the cooling medium, before it is fed for the first time and/or later to the cryocondenser, is heated, more precisely to a temperature which is sufficient in order to bring the gas stream that is to be purified to a temperature below the dew point of the substance which is to be removed from the gas stream. A particularly suitable site for installing a heater is, in particular, the cooling medium heat exchanger itself. The heating output can then be controlled directly in dependence on the heat transfer to the cooling medium that is to be fed to the cryocondenser.

An equally energetically advantageous embodiment of the invention provides that the cooling medium, before – first or later – feed thereof to the cryocondenser is brought into heat exchange with the purified gas stream by thermal contact.

In another expedient embodiment of the invention, the gas stream that is to be purified, before feed thereof to the cryocondenser, is brought into thermal contact with the previously purified gas stream that is exiting from the cryocondenser, and precooled thereby.

The object of the invention is also solved by a device of the type mentioned at the outset, in which the cryocondenser is equipped with a gas stream feed and a gas stream discharge for the gas stream that is to be purified and with a cooling medium feed and also a cooling medium discharge, in that the cooling medium feed is thermally connected at heat exchange surfaces of a cooling medium heat exchanger to a feed for a heat transfer medium. The heat transfer medium serves to bring the temperature of the medium to be fed to the cryocondenser closer to the temperature of the gas stream in the cryocondenser that is to be purified and in this case to increase it to a value at which, firstly the aerosol formation is effectively suppressed, and secondly a cooling effect is achievable in the cryocondenser which is sufficient for the cryocondensation.

Preferably, the cooling medium feed in this case is in thermal contact with the cooling medium discharge. Such a structure ensures that only a small temperature difference occurs between the gas stream and the cooling medium in the cryocondenser, but at the same time a good cooling effect proceeds in the cryocondenser.

Advantageously, the cryocondenser is equipped with a secondary cooling medium feed which is flow-connected to a cooling medium discharge for the cooling medium that is cooled in the cooling medium heat exchanger.

Further expedient development of the invention provides a tertiary cooling medium feed which is flow-connected to the gas stream discharge of the cryocondenser. The expression "tertiary cooling medium feed" here is to serve only linguistic differentiation from the abovementioned "secondary cooling medium feed"; according to the invention the presence of such a "secondary cooling medium feed" that is flow-connected to the cooling medium discharge, however, is not a precondition for the installation of a "tertiary cooling medium feed" that is flow-connected to the gas stream discharge.

In order to lower the energy costs further, it is advantageous to connect thermally the gas stream discharge of the cryocondenser via a heat exchanger to a cooling medium feed, wherein this can be the first, the secondary and/or the tertiary cooling medium feed.

It is further advantageous when the gas stream discharge of the cryocondenser is in thermal contact with the gas stream feed of the cryocondenser in a heat exchanger.

In order to increase the efficiency of the purification action and to decrease the energy costs in operation of the device according to the invention, it is particularly expedient to equip the device according to the invention with additional installations for temperature control of the cooling medium and/or of the gas stream. These installations are, for example, temperature sensors, heaters and/or programmable control installations which are arranged within the cryocondenser, one of the abovementioned heat exchangers, or in the associated feeds or discharges, and which have the task of controlling the absolute temperature in the cryocondenser to a value at which the substance present in the gas stream condenses out at least to a large part and in this case at the same time of keeping as small as possible the temperature difference between the gas stream and the cooling medium in the various cooling medium lines thereof in the cryocondenser, in order to curb the aerosol formation.

The following exemplary embodiments of the invention are to be explained in more detail with reference to the drawings.

In schematic views:

Fig. 1: shows the flow pattern of a device according to the invention in a first embodiment and

Fig. 2: shows the flow pattern of a device according to the invention in a second embodiment.

The device 1 shown in Fig. 1 comprises a heat exchanger for purifying a gas stream by means of cryocondensation, hereinafter termed cryocondenser 2, a cooling medium heat exchanger 3 for prewarming and/or vaporizing the cooling medium that is fed to the cryocondenser 2, and a heat exchanger 4 for precooling the gas stream that is to be purified. As cooling medium, preferably in this case a liquid gas, for example nitrogen, argon or carbon dioxide, comes into consideration. In the exemplary embodiments, nitrogen (N_2) is used.

On the cryocondenser 2, a gas stream feed 5 is arranged for feeding a gas stream that is loaded with at least one condensable substance into the cryocondenser, and also a gas stream discharge 6 for removing the at least partially purified gas stream from the cryocondenser 2. In the cryocondenser 2, the gas stream is freed from the substance by purification in a manner known per se by thermal contact with a cooling medium on heat exchange surfaces 9. A cooling medium feed 7 serves for feeding the cooling medium, and a cooling medium discharge 8 serves for discharging the cooling medium that is warmed by thermal contact with the gas stream.

In the exemplary embodiment according to Fig. 1, the gas stream discharge 6 is connected to the heat exchanger 4, on the heat exchange surfaces 10 of which a thermal contact is produced between the gas stream that is cooled and purified by the cryocondensation that is performed, and the gas stream that is fed in the gas stream feed 5, as a result of which it is precooled before feed thereof to the cryocondenser 2, and thereby the energy efficiency of the device 1 is increased overall.

In order to suppress in the cryocondenser 2 the formation of aerosols in the gas stream, it is considered to be important that the temperature difference between the cooling medium and the gas stream in the region of the heat exchange surfaces 9 is as low as possible. For this purpose, in the cooling medium heat exchanger 3 a transfer of heat from the cooling medium passed through the cooling medium discharge 8 to the cooling medium to be fed to the cryocondenser 2 takes place. The cooling medium that is to be fed to the cryocondenser 2 is introduced from a source, which is not shown here, which is, for example, a liquid gas tank, via a feed 11 into the cooling medium heat exchanger 3 and there warmed by thermal contact with the cooling medium which is conducted thereto from the cooling medium discharge 8. If the cooling medium is conducted in the feed 11 in the liquid state, it vaporizes in the course of this and arrives in the gaseous state via the cooling medium feed 7 into the cryocondenser 2. The cooling medium that is cooled in the cooling medium heat exchanger 3 passes from the cooling medium discharge 8 via a secondary cooling medium feed 12 back into the cryocondenser 2. At secondary heat exchange surfaces 13, a heat exchange with the gas stream again takes place before the cooling medium is, via a secondary cooling medium discharge 14, discharged into the atmosphere, fed to reprocessing, or a further use. A possible "further use" would be, for example, the use such that the cooling medium from the secondary cooling medium discharge 14 again once or several times successively passes through the cooling medium heat exchanger 3 and the cryocondenser 2, in order there to be brought into thermal contact with the cooling medium and/or the gas stream.

The temperatures of the cooling medium that is fed in the cooling medium feeds 7, 12 can be affected by switching on a heater 15 that is arranged in the cooling medium heat exchanger 3. The heater 15 can also be controlled and optimally adapted to the respective conditions via temperature sensors in the cryocondenser 2 which are not shown here and corresponding control installations, in dependence on the temperature of the gas stream and/or further parameters, such as, for example, the dew point of a substance that is to be condensed. The substances that condensed out of the gas stream in the cryocondenser 2 are removed via a condensate outlet 16.

Instead of, or in supplementation to, the heating of the cooling medium to be fed to the cryocondenser 2 by the cooling medium that is discharged from the cryocondenser, warm cooling medium, for example gaseous nitrogen, can also be fed into the cooling medium discharge 8 from a mains grid or a tank, via the gas feed 17. In this case, the cooling medium can be discharged into the open in whole or in part via a discharge 18, or can be fed to another use. The control of the gas streams proceeds in this case via valves 19 which are manually operated or automatically actuated via a suitable control installation which is not shown here. By feeding warm gas via the gas feed 17, the heat output of the heater 15 can also be reduced.

In the case of the device 20 described in Fig. 2, functionally identical components are denoted with the same reference numbers as in Fig. 1. In contrast to the device 1 of Fig. 1, the device 20 has additional heat exchangers in which the heat content of the purified gas stream is exploited to increase the energy efficiency.

The cooling medium that was introduced from the feed 11 and vaporized or warmed in the cooling medium heat exchanger 3 is fed via a feed 21 to a heat exchanger 22, at the heat exchange surfaces 23 of the which it is then brought into thermal contact with the purified gas stream from the gas stream discharge 6. The cooling medium that is further warmed in this manner is then fed via the cooling medium feed 7 to the cryocondenser 2. The purified gas stream in the gas stream discharge 6, supplementary to the abovementioned use in the heat exchanger 22, or instead of which, can be used by means of a heat exchanger 25 to precool the cooling medium fed in the cooling medium discharge 8 before feed thereof to the cooling medium heat exchanger 3. Alternatively, or in supplementation to the heat exchangers 22, 25, tertiary heat exchange surfaces 26 in the cryocondenser 2 can also be used, at which the purified gas stream from the gas stream discharge 6 is brought into thermal contact with the unpurified gas stream via a tertiary cooling medium feed 27 and in this manner contributes to the cooling thereof. The purified gas stream that is discharged from the tertiary heat exchange surfaces 26 is then discharged into the atmosphere or fed to another use. In the exemplary embodiment according to Fig. 2, the heat exchange surfaces 26 operate in parallel to the heat exchanger 25, but in the context of the invention, other configurations are possible, in particular to connect both heat exchangers 25, 26 in series connection with one another or to provide only one of the two in a device 20 according to the invention.

In a further variant of the invention shown in Fig. 2, the cooling medium that is cooled in the cooling medium heat exchanger 3, on its way through the secondary cooling medium feed, is conducted via heat exchange surfaces 28 in the heat exchanger 22 and there brought into thermal contact with the purified gas stream. This variant serves to bring the temperature of the secondary cooling medium in the secondary heat exchange surfaces 13 closer to the temperature of the gas stream that is to be purified.

Industrial implementation of the primary, secondary and tertiary heat exchange surfaces 9, 13, 26 can be achieved, for example, in the form of tube bundles that are appropriately flow-separated from one another, around which flow passes in the cryocondenser 2.

It is a matter of course that the embodiments according to Fig. 1 and Fig. 2 do not represent the sole possible configurations of a device according to the invention, in particular, it is possible to provide only individual heat exchangers 22, 25, 26, 28, or such heat exchangers 22, 25, 26, 28 can also be installed into a device of the type shown in Fig. 1. Correspondingly, the heat exchanger 4, lines 17, 18 with valves 19 or a heater 15 can be installed into a device corresponding to the device 20, or further heating installations can be provided at various positions of the device. Also, the cooling medium from the secondary cooling medium discharge 12, or the purified gas stream from the discharge 29 can be used again to prewarm the cooling medium that is to be fed to the cryocondenser 2. In order to increase the purification action, a plurality of the above described devices 1, 20 can also be combined with one another or with other systems for gas purification.

Example:

The gas stream that is to be purified enters into the cryocondenser 2 containing an amount of 120 kg/h of nitrogen plus a loading of 60 kg/h of an organic solvent at a temperature of 0°C. By thermal contact with the cooling medium, the gas stream cools up to the exit of the cryocondenser 2 at the gas stream discharge 6 to a temperature of minus 144°C. Nitrogen stored in a liquid tank at a pressure 3.6 bar (360 000 Pa) at a temperature of minus 183°C, which is used as cooling medium, is warmed to a temperature of about minus 180°C in the heat exchanger 3 by thermal contact with the cooling medium that was removed from the cryocondenser 2 via the cooling medium discharge 8 and which has a temperature of approximately minus 20°C. Then, further warming in the heat exchanger 22 to approximately minus 160°C takes place. Between the fed cooling medium and the removed gas stream, there is therefore still a temperature difference of only 16 K.

The cooling medium from the cooling medium discharge 8 that was cooled in the heat exchanger 3 to a temperature of approximately minus 180°C is fed via a secondary cooling medium feed 12 to the cryocondenser, previously, however, in heat exchanger 22 was brought in heat exchange with the purified gas stream from the gas stream discharge 6 and brought to a temperature of minus 160°C, that is to say to at least approximately the same temperature as the cooling medium introduced via the cooling medium feed 7. In the heat exchange with the gas stream in the cryocondenser 2, the secondary cooling medium warms to minus 20°C. By thermal contact with the cooling medium in the heat exchanger 2, the purified gas stream cools to a value of approximately minus 160°C and subsequently is conducted via the tertiary cooling medium feed 27 onto the cryocondenser 2, in which it contributes to the cooling of the process gas by thermal contact with the unpurified gas stream at the heat exchange surfaces 26. It leaves the cryocondenser at a temperature of minus 20°C. In this example, no heat exchanger 25 is provided for precooling the cooling medium; however, for thermodynamic reasons, in the cooling medium heat exchanger 3, the cooling medium is heated with a heat output of approximately 0.4 kW.

List of reference signs

1. Device
2. Cryocondenser
3. Cooling medium heat exchanger
4. Heat exchanger
5. Gas stream feed
6. Gas stream discharge
7. Cooling medium feed
8. Cooling medium discharge
9. Heat exchange surfaces (in the cryocondenser)
10. Heat exchange surfaces (in the heat exchanger 4)

11. Feed
12. Secondary cooling medium feed
13. Secondary heat exchange surfaces
14. Secondary cooling medium discharge
15. Heater
16. Condensate outlet
17. Gas feed
18. Discharge
19. Valves
20. Device
21. Feed
22. Heat exchanger
23. Heat exchange surfaces
24. -
25. Heat exchanger
26. Tertiary heat exchange surfaces
27. Tertiary cooling medium feed
28. Heat exchange surfaces
29. Discharge

Eljárás és berendezés parciális kondenzáció elvégzésére

SZABADALMI IGÉNYPONTOK

1. Eljárás parciális kondenzáció elvégzésére, ahol egy, a kondenzálandó anyaggal töltött gázáramot kriokondenzátorban (2) hűtőközeggel történő hőcseréléssel hűtünk, és ezzel a gázáramban lévő anyagot legalább részben kondenzáljuk,
ahol a kriokondenzátorba (2) vezetendő hűtőközeget a kriokondenzátorba (2) történő bevezetés előtt egy hűtőközeg hőcserélőben (3) egy hőközlő közeggel érintkeztetve felmelegítjük és/vagy elgőzölögtetjük,
ahol hőközlő közegként legalább részben a kriokondenzátorból (2) kivezetett hűtőközeget alkalmazzuk,
azzal jellemezve, hogy
a feltöltött gázáramot a kriokondenzátorban (2) járulékosan a kriokondenzátorból (2) kivezetett és a hűtőközeg hőcserélőn (3) keresztülvezetett hűtőközeggel hőkapcsolatba hozzuk.
2. Az 1. igénypont szerinti eljárás, **azzal jellemezve, hogy** a hűtőközeget ezután ismét egy vagy több alkalommal egymás után keresztül vezetjük a hűtőközeg hőcserélőn (3) és a kriokondenzátoron (2), hogy ott termikus érintkezésbe hozhassuk a hűtőközeggel, illetve a gázárammal.

3. Az 1. vagy 2. igénypont szerinti eljárás, **azzal jellemezve, hogy** a hűtőközeget a kriokondenzátorba (2) történő első és/vagy későbbi bevezetése előtt hevítjük.

4. Az előző igénypontok bármelyike szerinti eljárás, **azzal jellemezve, hogy** a hűtőközeget a kriokondenzátorba (2) történő bevezetése előtt termikus érintkezésbe hozzuk a megtisztított gázárammal.

5. Az előző igénypontok bármelyike szerinti eljárás, **azzal jellemezve, hogy** a feltöltött gázáramot a kriokondenzátorban (2) és/vagy a kriokondenzátorba (2) történő bevezetése előtt termikus érintkezésbe hozzuk a megtisztított gázárammal.

6. Az előző igénypontok bármelyike szerinti eljárás, **azzal jellemezve, hogy** a hűtőközeget a kriokondenzátoron (2) történő átvezetése után és a hűtőközeg hőcserélőbe (3) történő bevezetése előtt termikus érintkezésbe hozzuk a megtisztított gázárammal.

7. Berendezés kriokondenzátorral (2), amelyben egy, a kondenzálandó anyaggal töltött gázáram hőcsere során hűtőközeggel van hűtve, és ezzel a gázáramban lévő anyag legalább részben kondenzálódik, ahol a kriokondenzátor (2) el van látva egy gázbevezetéssel (5) és egy gázkivezetéssel (6) a tisztítandó gáz számára, valamint egy hűtőközeg bevezetéssel (7) és egy hűtőközeg kivezetéssel (8), ahol a hűtőközeg bevezetés (7) egy hűtőközeg hőcserélő (3) hőcserélő felületein a hűtőközeg kivezetéssel (8) van termikusan összekapcsolva,

   azzal jellemezve, hogy a kriokondenzátor (2) másodlagos hőcserélő felületekkel (13) és egy másodlagos hűtőközeg kivezetéssel (12) van ellátva, ami a hűtőközeg hőcserélőben (3) lehűtött hűtőközeg számára egy hűtőközeg kivezetéssel van áramlástechnikailag összekapcsolva.

8. A 7. igénypont szerinti berendezés, **azzal jellemezve, hogy** a kriokondenzátor (2) egy harmadlagos hűtőközeg kivezetéssel (27) van ellátva, ami a gázkivezetéssel (6) van áramlástechnikailag összekapcsolva.

9. A 7. és 8. igénypont szerinti berendezés, **azzal jellemezve, hogy** a kriokondenzátor (2) gázkivezetése (6) egy hőcserélőben (22, 25) egy hűtőközeg bevezetéssel (7, 11, 12, 27) vagy egy hűtőközeg kivezetéssel (8, 14) van termikusan összekapcsolva.

10. A 7 – 9. igénypontok bármelyike szerinti berendezés, **azzal jellemezve, hogy** a kriokondenzátor (2) gázkivezetése (6) egy hőcserélőben (4) a hűtőközeg bevezetéssel (5) van termikusan összekapcsolva.

11. A 7 – 10. igénypontok bármelyike szerinti berendezés, **azzal jellemezve, hogy** a kriokondenzátor (2) és legalább az egyik hőcserélő (4, 22) egy házban van elrendezve.

12. A 7 – 11. igénypontok bármelyike szerinti berendezés, **azzal jellemezve, hogy** járulékos berendezésekkel (15) van ellátva a hűtőközeg és/vagy a gázáram hőmérsékletének szabályozására.

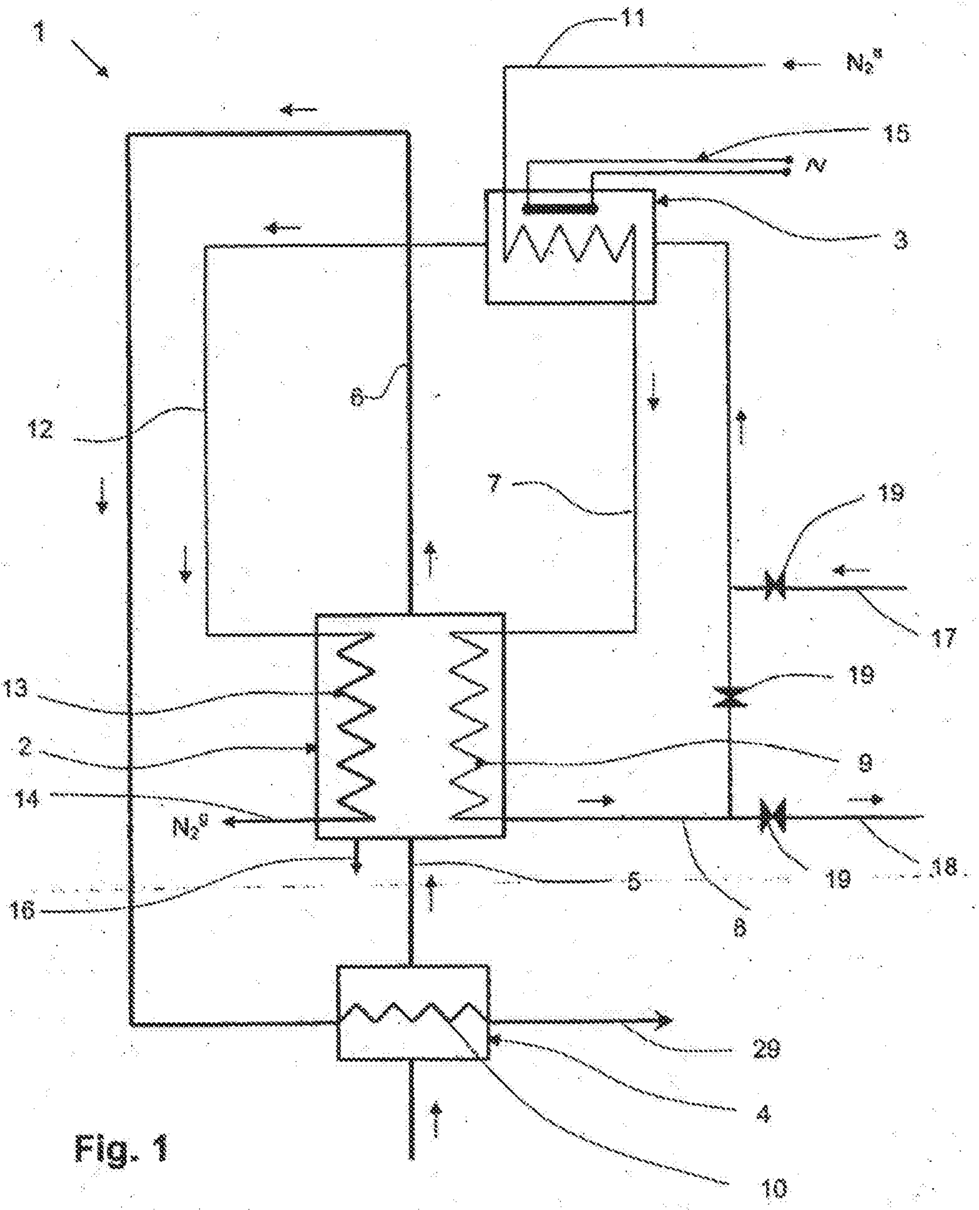

Fig. 1

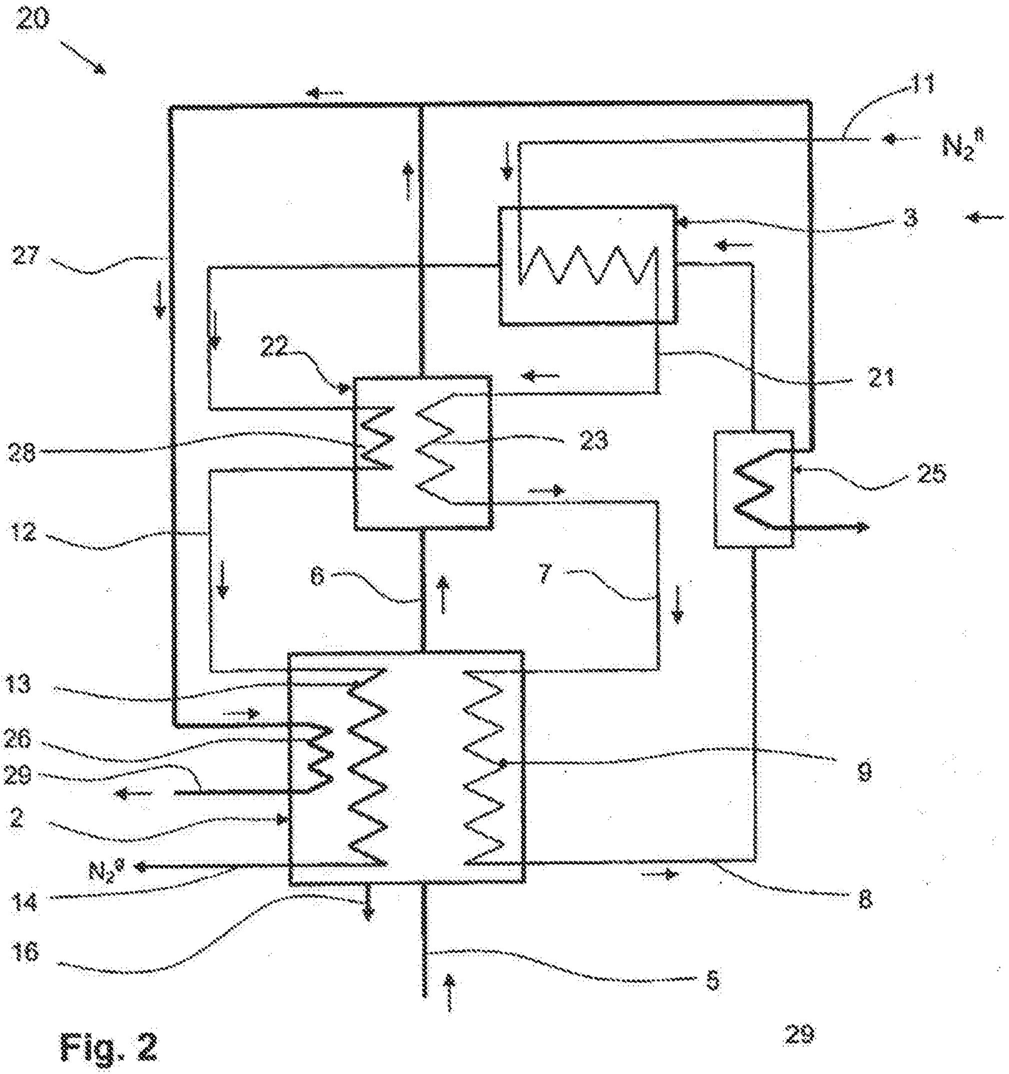

Fig. 2