A process installation for preparing a carbon dioxide (CO₂) end product from a gaseous carbon dioxide containing starting product.

The invention relates to a process installation for preparing - during use - a carbon dioxide (CO₂) end product from a gaseous carbon dioxide containing starting product having a lower degree of purity than the carbon dioxide end product, which process installation comprises a processing line, which processing line comprises at least the elements: a main inlet for the gaseous carbon dioxide containing starting product having a first degree of purity; a first purification unit for preparing liquid carbon dioxide having a second degree of purity from said gaseous carbon dioxide containing starting product using a cooling medium, said second degree of purity being higher than said first degree of purity, said first purification unit at least comprising: a cooling unit and a heat exchanging unit being in heat exchanging contact with each other; said cooling unit having a cooling inlet being in fluid communication with said main inlet and a cooling outlet for outputting said liquid carbon dioxide with said second degree of purity; and said heat exchanging unit having a heat exchanging inlet being in fluid communication with said cooling outlet and a heat exchanging outlet for outputting carbon dioxide end product having a third degree of purity, and a main outlet being in fluid communication with said heat exchanging outlet, which elements are interconnected by means of suitable first piping.
Title: A process installation for preparing a carbon dioxide (CO₂) end product from a gaseous carbon dioxide containing starting product.

DESCRIPTION

The invention relates to a process installation for preparing - during use - a carbon dioxide (CO₂) end product from a gaseous carbon dioxide containing starting product having a lower degree of purity than the carbon dioxide end product, which process installation comprises a processing line, which processing line comprises at least the elements: a main inlet for the gaseous carbon dioxide containing starting product having a first degree of purity; a first purification unit for preparing liquid carbon dioxide having a second degree of purity from said gaseous carbon dioxide containing starting product using a cooling medium, said second degree of purity being higher than said first degree of purity, said first purification unit at least comprising: a cooling unit and a heat exchanging unit being in heat exchanging contact with each other, said cooling unit having a cooling inlet being in fluid communication with said main inlet and a cooling outlet for outputting said liquid carbon dioxide with said second degree of purity, and said heat exchanging unit having a heat exchanging inlet being in fluid communication with said cooling outlet and a heat exchanging outlet for outputting carbon dioxide end product having a third degree of purity, and a main outlet being in fluid communication with said heat exchanging outlet, which elements are interconnected by means of suitable first piping.

Such process installation is for example known from the European patent application no. 0194795 in the name of Kins Development Ltd. In the installation described a carbon dioxide end product is prepared based on a gaseous carbon dioxide containing starting product. Such gaseous carbon dioxide containing starting product is for example a by-product of a beer brewery. The degree of purity of the carbon dioxide starting product is lower than the degree of the carbon dioxide end product, which end product is often indicated as a ‘pure’ or ‘food grade’ carbon dioxide end product. This allows the ‘food grade’ carbon dioxide end product to be used in other applications, in particular for the carbonation of soft drinks or beer.

In the aforementioned European patent application no. 0194795 the gaseous carbon dioxide containing starting product with a low degree of purity is being subjected to a first purification process stage, during which liquid carbon dioxide having a
higher degree of purity is being prepared from said lower degree gaseous carbon dioxide containing starting product using a cooling medium. In this first purification stage so-called non-condensable gaseous impurities having a higher condensation temperature than carbon dioxide are being discharged and the liquid, condensed carbon dioxide being prepared is in fact used as the cooling medium as it is being fed back towards the first purification process stage for cooling said gaseous carbon dioxide containing starting product by means of heat exchange.

Herewith it is avoided to use other coolant agents, such as ammonia, glycol or Freon, the use of which are undesirable due to the risk of contamination in a food environment such as a beer or soft drink brewery.

With the known process installation it is possible to prepare a carbon dioxide end product having a high degree of purity, which end product is outputted towards the end user for further consumption, for example for carbonation of beer or soft drinks.

It is to be emphasized that in this patent application the carbon dioxide end product having a high degree of purity is not necessarily intended for food consumption applications, such as carbonation of beer or soft drinks, but could also be used for non-food consumption applications, such as the purification of biogas from a biomass fermentation application, where carbon dioxide from the fermentation biogas is being obtained as an end product with a high degree of purity.

However the known process installations are operated such, that carbon dioxide end product having a high degree of purity is being prepared in a continuous manner. Due to the nature and quality of the carbon dioxide end product any surplus will not be purged towards the atmosphere but stored in suitable storage tanks. As long as there is any consumption of carbon dioxide end product at the main outlet by the user, for example for carbonating soft drinks, the process installation will be operated at a so-called equilibrium between production, consumption and storage capacity.

When there is no or little consumption of carbon dioxide end product at the main outlet of the process installation said equilibrium will be disturbed. In particular the known process installation require additional cooling capacity to compensate for the absent or low consumption, which is very energy intensive and difficult to control as it involves the controlling of an additional cooling unit.

It is an object of the invention to provide a process installation of the above
kind, which process installation can be operated in a controlled and energy efficient manner for any kind of consumption level of carbon dioxide end product at the main outlet.

According to the invention the process installation hereafter further comprises a recirculation unit at least comprising a recirculation inlet being in fluid communication with said heat exchanging outlet; a compressor unit, a condenser unit and an expansion unit for preparing liquid carbon dioxide end product having said third degree of purity, and a recirculation outlet for outputting liquid carbon dioxide end product having said third degree of purity, said recirculation outlet being in fluid communication with said heat exchanging inlet.

Hereafter an energy efficient recirculation of carbon dioxide end product is feasible regardless of the actual consumption of the carbon dioxide end product at the main outlet of the installation. After the intake of carbon dioxide end product in a gaseous state at the recirculation inlet side liquefaction is done in a controlled and energy efficient manner.

In particular the process installation further comprises a second purification unit for preparing liquid carbon dioxide end product having said third degree of purity from said liquid carbon dioxide having said second degree of purity, said third degree of purity being higher than said second degree of purity, said second purification unit at least comprising a purification inlet being in fluid communication with said cooling outlet and a purification outlet being in fluid communication with said heat exchanging inlet.

In this purification stage carbon dioxide end product is being prepared having a third, highest degree of purity. This carbon dioxide end product is being returned to the heat exchanging inlet of the first purification unit and serves as the cooling medium for the first purification step of the gaseous carbon dioxide starting product.

In particular said second purification unit comprises a reboiler unit interconnection said purification inlet and said purification outlet, which reboiler unit is in heat exchanging contact with the main inlet, wherein said reboiler unit further comprises at least one storage unit for storing said liquid carbon dioxide having said third degree of purity, wherein further said at least one storage unit is in fluid communication with said heat exchanging inlet.

In particular said second purification unit further comprises a stripper unit interconnecting said purification inlet with said reboiler, as well as having a gas stripper
interconnection between said reboiler unit and main inlet. According to an embodiment of the invention said recirculation liquid outlet is in fluid communication with said reboiler unit. With this piping configuration a proper, energy efficient recirculation of the carbon dioxide end product is obtained, wherein the carbon dioxide end product obtained from the recirculation unit is reintroduced in a liquid state into the processing line towards the heat exchanging inlet. In particular the carbon dioxide end product obtained from the recirculation unit is being combined or mixed with the carbon dioxide end product being obtained in the second purification unit, thus avoiding any dilution of the degree of purity of the carbon dioxide end product.

In a further embodiment the recirculation unit further comprises at least a recirculation flash gas outlet for outputting gaseous carbon dioxide end product having said third degree of purity, which recirculation flash gas outlet is in fluid communication with said main outlet. Carbon dioxide end product being liquefied in the recirculation unit still contains an amount of dissolved gaseous carbon dioxide that is being released as flash gas and purged back into the main outlet. Herewith an additional energy efficiency is obtained as the flash gas (end product) is not introduced into the first piping towards the heat exchanging unit, nor towards the cooling inlet.

In another embodiment said recirculation flash gas outlet is in fluid communication with said cooling inlet. In the event of no consumption of gaseous end product at the main outlet said gaseous flash gas can be reintroduced into the main inlet towards the cooling unit.

In particular said recirculation flash gas outlet is in heat exchanging contact with said expansion unit, which expansion unit comprises an expansion valve and an expansion storage unit. With this configuration the recirculated flash gas is reintroduced and brought in heat exchanging contact with the liquefied recirculated carbon dioxide end product. Herewith the flash gas is being warmed up, whereas the liquefied recirculated carbon dioxide is being cooled in order to further reduce the amount of flash gas dissolved.

In a specific embodiment said at least one storage unit and said expansion storage unit form one single storage unit. This allows the process installation to be constructed in a simplified and less expensive manner, as only one storage unit for storing liquefied carbon dioxide end product being obtained in either the second purification unit and in the recirculation unit.
In addition said cooling unit further comprises a purge outlet for purging non-condensable gases. Such non-condensable gases are considered by-products of the process, which has created the gaseous carbon dioxide containing starting product and can be amongst others, but not limited be $\text{N}_2$, $\text{O}_2$, $\text{CH}_4$ and more. Such gasses have a higher condensation temperature than carbon dioxide and are purged out of the process installation towards for example the atmosphere.

In another embodiment the process installation further comprises a superheater unit interconnecting said heat exchanger outlet with said main outlet.

The invention will now be explained in more detail with reference to a drawing, in which:

Figure 1 shows a first embodiment of a process installation according to the invention;

Figure 2 shows a second embodiment of a process installation according to the invention;

Figure 3 shows a third and fourth embodiment of a process installation according to the invention;

Figure 4 shows a fifth embodiment of a process installation according to the invention;

Figure 5 shows a sixth, seventh and eight embodiment of a process installation according to the invention;

Figure 6 shows a schematic view of a further embodiment of a process installation according to the invention.

For a clear understanding of the detailed description below identical parts depicted in the accompanying drawings are denoted with like reference numerals.

Figure 1 discloses a first simplified yet schematic embodiment of a process installation according to the invention. The process installation 10 comprises a processing line for preparing a carbon dioxide end product using a gaseous carbon dioxide containing starting product. The process installation 10 of the embodiment depicted in Figure 1 consists of a processing line which is composed of a first piping 15 and several interconnected elements. With reference numeral 11 a main inlet is depicted for the intake of a gaseous carbon dioxide containing starting product having a first degree of purity.
In this patent application it should be understood that with the first degree of purity it is meant a carbon dioxide containing product having a lower degree of purity in connection to the degree of purity of the carbon dioxide end product.

With the process installation according to the invention a carbon dioxide end product is obtained with a degree of purity which is (significantly) higher than the first degree of purity of the gaseous carbon dioxide containing starting product.

The inlet 11 is further connected to a first purification unit 12. The first purification unit 12 comprises two elements: a cooling unit 121 and a heat exchanging unit 122. The cooling unit 121 and the heat exchanging unit 122 are in heat exchanging contact with each other. The heat exchanging contact surface between both units 121 and 122 is depicted in the Figures 1-5 by means of the schematic bold line 120.

In all Figures 1-6 the main inlet 11 is interconnected with the cooling unit 121 by means of a cooling inlet 121a. In the cooling unit 121 the gaseous carbon dioxide containing starting product (with the first degree of purity) is cooled down and liquefied using a cooling medium, such that at the cooling outlet 121b liquefied carbon dioxide is outputted. This liquefied carbon dioxide has a second degree of purity, which second degree of purity is higher than the first degree of purity of the gaseous carbon dioxide starting product which has entered the cooling unit 121 via the cooling inlet 121a. The first purification in the cooling unit 121 is established as any non-condensable gases present in the gaseous carbon dioxide containing starting product are being purged out of the cooling unit 121 via a purge outlet 14.

As depicted in Figure 1 the liquefied carbon dioxide with the second degree of purity is outputted at the cooling outlet 121b and is circulated through piping 15 towards a heat exchanging inlet 122a of the heat exchanger 122. In fact the cooling medium used to liquefy the gaseous carbon dioxide containing starting product being introduced via the cooling inlet 121a into the cooling unit 121 is the liquefied carbon dioxide with the second degree of purity, which has been outputted via the cooling outlet 121b.

The first purification stage in the first purification unit 12 takes place by means of the cooling down and the liquefaction of the gaseous carbon dioxide containing starting product (with the first, lowest degree of purity) by means of heat exchange across the heat exchanging surface 120 using as a cooling medium the already liquefied carbon dioxide having the second, higher degree of purity, which is being reintroduced via the heat exchanging inlet 122a into the heat exchanging unit 122.
Due to the heat exchanging transfer across the heat exchanging surface 120 the gaseous carbon dioxide containing starting product is being liquefied due to the fact that the already liquefied carbon dioxide having the second degree of purity undergoes a pressure drop and needs energy to evaporate in the heat exchanging unit. This energy is being extracted across the heat exchanging surface 120, which generates a cold front at the other side of the heat exchanging surface and liquefies carbon dioxide from the gaseous carbon dioxide containing starting product. Evaporated carbon dioxide having the second degree of purity is delivered via the heat exchanging outlet 122b and via piping 15 towards a main outlet 13.

The carbon dioxide end product outputted via the heat exchanging outlet 122b towards the main outlet 13 is considered having a high degree of purity, often indicated as a ‘pure’ or ‘food grade’ carbon dioxide end product (when using the end product in a food consumption application). This end product can be distributed via the main outlet 13 towards a dedicated process installation of an end user, who might use the carbon dioxide end product for several application. One application can be the carbonation of soft drinks or beer. Please note however that in this patent application the carbon dioxide end product having a high degree of purity is not necessarily intended for food consumption applications, such as carbonation of beer or soft drinks, but could also be used for non-food consumption such as the purification of biogas from a biomass fermentation application.

A process installation 10 as depicted in a schematic manner in Figure 1 (and Figures 2-6) are operated such that carbon dioxide end product having a high ‘food grade’ degree of purity is being prepared in a continuous manner. Due to the nature and quality of the carbon dioxide end product any surplus will not be purged to the outer atmosphere but will be stored in suitable storage tanks. As long as there is any consumption of carbon dioxide end product at the main outlet 13 by the end user, for example for carbonating soft drinks, the process installation 10 will be operated at a so-called equilibrium between production, consumption and storage capacity.

When there is no or little consumption of carbon dioxide end product at the main outlet 13 of the process installation 10 said equilibrium will be disturbed. In particular the known process installation according to the state of the art will require additional cooling capacity to compensate for the absent or low consumption at the main outlet 13, which is difficult to control as it involves the controlling of an additional cooling unit. Hereto
the process installation 10 according to the invention as depicted in Figure 1 can be
operated in a controlled and energy efficient manner for any kind of consumption level of
carbon dioxide end product at the main outlet 13.

In addition the process installation 10 is provided with a recirculation unit,
depicted with reference numeral 50. The recirculation unit 50 is composed of a further
processing line consisting of a second piping 54 and interconnected elements depicted
with reference numerals 51-52-53. The recirculation unit 50 has a recirculation inlet 50a
which is connected with the main outlet 13. Furthermore, the recirculation unit is
composed of a line of elements being a compressor unit 51, in particular any type of
compressor unit with additional equipment elements (such as a filter, etc.), a condenser
unit 52 and an expansion unit 53.

In the event of no or little consumption of carbon dioxide end product (with
a high degree of purity) at the main outlet 13, said gaseous carbon dioxide end product is
drawn in via the recirculation inlet 50a towards a compressor unit 51. After compression
by the compressor unit 51 the compressed carbon dioxide end product is being fed
towards a condenser unit 52 and then towards an expansion unit 53. The expansion unit
53 will be explained in more detail with reference to Figures 5 and 6. However, the
subsequent stage of compression by the compressor unit 51, cooling down by the
condenser unit 52 and expansion via the expansion unit 53 result in liquefied carbon
dioxide end product having the same high degree of purity as the gaseous carbon dioxide
end product, which exits the heat exchanging outlet 122b.

Said liquefied carbon dioxide end product is being fed towards the
recirculation outlet 50b, which according to the invention is in fluid communication with the
heat exchanging inlet 122a (via first piping 15).

Herewith an energy efficient recirculation of carbon dioxide end product
(with the high degree of purity) is feasible regardless of the actual consumption of the
carbon dioxide end product at the main outlet 13 of the process installation 10. After the
intake of carbon dioxide end product in the gaseous state at the recirculation inlet 50a
liquefaction is performed via the stage of the compressor unit 51, the condenser unit 52
and the expansion unit 53 in a controlled and energy efficient manner.

Reference numeral 55 denotes a recirculation flash gas outlet for outputting
gaseous carbon dioxide end product having the third, high degree of purity, which
recirculation flash gas outlet 55 is in fluid communication with the main outlet 13 by
means of outlet piping 56. This because carbon dioxide end product being liquefied in the recirculation unit near the stage of the expansion unit 53 will contain an amount of dissolved gaseous carbon dioxide that will be released as flash gas and purged back into the main outlet 13 via the recirculation flash gas outlet 55 and outlet piping 56.

Purging flash gas via the flash gas outlet 55 towards the main outlet 13 is considered energy efficient as it is herewith avoided that the flash gas end product is to be introduced via the recirculation outlet 50b and being mixed with liquefied carbon dioxide into the first piping towards the heat exchanging unit 122 and affects the efficiency of the heat exchanging unit. It is observed that the inlet 122a of the heat exchanging unit 122 is being fed with liquefied carbon dioxide and also inserting flash gas towards the heat exchanging unit 122 will reduce the energy efficiency of the heat exchanging unit 122 during operation.

It is noted that in Figure 1 the gaseous carbon dioxide end product at the heat exchanging unit 122b and the main outlet 13 has a higher degree of purity than the degree of purity of the gaseous carbon dioxide containing starting product at the main inlet 11 and the cooling inlet 121a. After the first purification step in the first purification unit 12 the liquefied carbon dioxide at the cooling outlet 121b has a second degree of purity, which second degree of purity is higher than the first degree of purity. In the embodiment of Figure 1 the liquid carbon dioxide with the second degree of purity is identical to the gaseous carbon dioxide end product with the third degree of purity which is being outputted at the heat exchanging outlet 122b and the main outlet 13.

It is to be understood that in the embodiment of the process installation 10 according to the invention as depicted in Figure 1 the liquid carbon dioxide at the cooling outlet 121b and the gaseous carbon dioxide end product at the main outlet 13 have the same degree of purity. In other words in the embodiment of Figure 1 the second degree of purity (obtained after the first purification stage at the first purification unit 12) belonging to the liquid carbon dioxide product at the cooling outlet 121b is identical to the third degree of purity belonging to the gaseous carbon dioxide end product at the main outlet 13.

In addition to Figure 1 the second embodiment of the process installation 10 as disclosed in Figure 2 comprises a second purification unit which is schematically denoted with reference numeral 16. The second purification unit 16 is accommodated in the first piping 15 and is provided with a purification inlet 16a, which is in fluid communication with the cooling outlet 121b. Furthermore the second purification unit 16
has a purification outlet 16b which is in fluid communication with the heat exchanging inlet 122a.

The second purification unit 16 is being provided with liquefied carbon dioxide with the second degree of purity being prepared with the first purification stage in the first purification unit 12 and being outputted via the cooling outlet 121b. In the second purification unit 16 liquefied carbon dioxide end product having a third degree of purity is being prepared, with the third degree of purity being higher than the second degree of purity of the liquefied carbon dioxide outputted at the cooling outlet 121b. The liquefied carbon dioxide with the third degree of purity is considered being the carbon dioxide end product with the highest degree of purity.

This liquefied carbon dioxide end product with the third, highest degree of purity is being fed towards the heat exchanging inlet 122a as a cooling medium for preparing liquid carbon dioxide with the second degree of purity in the first purification unit 12 using heat exchanging across the heat exchanging surface 120. The carbon dioxide end product having the third, highest degree of purity is being outputted via the heat exchanging outlet 122b towards the main outlet 13 in a gaseous state.

As in Figure 1 also this second embodiment of the process installation 10 according to the invention is provided with the recirculation unit 50, for its functionality reference is made towards the detailed description in connection with Figure 1. Also in this embodiment of Figure 2 the recirculation outlet 50b is in direct fluid communication with the heat exchanging inlet 122a for recirculating liquefied carbon dioxide end product having the third, highest degree of purity, which liquefied carbon dioxide end product has been introduced into the recirculation unit 50 in a gaseous state via the main outlet 13 and the recirculation inlet 50a.

Also in this embodiment of the process installation according to the invention flash gas is being purged downstream from the expansion unit 53 via flash gas outlet 55 and flash outlet piping 56 back towards the main outlet 13.

Figure 3 discloses further embodiments of a process installation 10 according to the invention. In Figure 3 the second purification unit 16 comprises a reboiler unit 161 which interconnects the purification inlet 16a with the purification outlet 16b. In addition the reboiler unit 161 further comprises at least one storage unit 162 for storing liquid carbon dioxide having the third degree of purity.
In one embodiment the reboiler unit 161 is in direct fluid communication with the heat exchanging inlet 122a via the purification outlet 16b, yet in another embodiment the reboiler unit 161 is in indirect fluid communication with the heat exchanging inlet 122a, as the reboiler unit 161 first connects to the storage unit 162, and which storage unit 162 is in direct fluid communication via the purification outlet 16b with the heat exchanging inlet 122a via piping 15.

In either embodiment wherein the reboiler unit 161 is in direct fluid communication with the heat exchanging inlet 122a or via at least one intermediate storage unit 162 with said heat exchanging inlet 122a, the liquid carbon dioxide end product having the third, highest degree of purity is being fed towards the heat exchanging unit 122 for preparing liquid carbon dioxide having a second degree of purity from the gaseous carbon dioxide containing starting product having the first degree of purity.

In this embodiment of the process installation encompassing two purification stages using first purification unit 12 and second purification unit 16 it is noted that the first degree of purity is lower than the second degree of purity, which in turn is lower than the third degree of purity, which is considered the highest degree of purity of the carbon dioxide end product. As stated earlier the carbon dioxide end product having the third, highest degree of purity is fed towards to main outlet 13 for further use, for example for the carbonation of soft drinks or beer.

Apart from the multiple configurations of the second purification unit 16 in Figure 3 the third and fourth embodiment of the process installation according to the invention relate to the connection of the recirculation outlet 50b (see Figures 1 and 2) with the heat exchanging inlet 122a.

Whereas in Figures 1 and 2 the recirculation outlet 50b is in direct fluid communication with the heat exchanging inlet 122a, Figure 3 shows two alternative connections wherein the recirculation outlet 50b is in indirect fluid communication with the heat exchanging inlet 122a. These two indirect fluid communications are depicted in dashed lines 50b' and 50b". In the third embodiment the recirculation outlet 50b' is in direct fluid communication with the reboiler unit 161, whereas in the fourth embodiment the recirculation outlet 50b" is in direct fluid communication with the at least one storage unit 162. In either embodiment the recirculation outlets 50b' and 50b" are in fluid
communication with the heat exchanging inlet 122a, albeit in an indirect communication manner.

However, in either third and fourth embodiment liquid carbon dioxide end product having the third, highest degree of purity is being introduced into the second purification unit where liquid carbon dioxide end product having the third degree of purity is being prepared from the liquid carbon dioxide having the second degree of purity. Also here in both embodiments no dilution of the carbon dioxide end product will occur as the carbon dioxide end product is being reintroduced into the process line of the process installation 10 at locations where carbon dioxide product having the same degree of purity exists in the process line.

Also in Figure 3 the flash gas outlet 55 communicates directly with the main outlet 13 via flash outlet piping 56.

In Figure 4 a fifth embodiment of the process installation 10 according to the invention is disclosed, wherein the second purification unit is further explained. In Figure 4 reference numeral 163 denotes a stripper unit which interconnects the cooling outlet 121b by means of piping 15 with the reboiler unit 161. The stripper column 163 also contains a gas stripper interconnection 164 which interconnects the reboiler 161 and the main inlet 11, in particular the cooling inlet 121a. In the stripper unit any gas still dissolved in the liquid carbon dioxide having the second degree of purity can escape from the liquid carbon dioxide and reintroduced via the gas stripper interconnection 164 towards the cooling inlet 121a.

The purified liquid carbon dioxide enters the reboiler 161 as the liquid carbon dioxide end product having the third, highest degree of purity. In addition to the earlier detailed figurative description of the second purification unit 16 it is to be noted that in this embodiment of Figure 4 the main inlet 11 is in heat exchanging contact with the liquid carbon dioxide end product having the third, highest degree of purity contained in the reboiler unit 161. The gaseous carbon dioxide starting product having the first, lowest degree of purity and being fed via the main inlet 11 is herewith pre-cooled prior to the entrance via the cooling inlet 121a into the cooling unit 121 for the first purification step.

This results in a further energy efficiency.

Figure 5 discloses a sixth, seventh and eighth embodiment of a process installation 10 according to the invention. Reference numerals 50b, 50b', 50b'' disclose the three embodiments of the (direct and indirect) different and possible fluid
communications of the recirculation outlet with the heat exchanging inlet 122a. The dashed line depicted with 50b shows the direct fluid communication of the recirculation outlet 50b with the heat exchanging inlet 122a, whereas the recirculation outlet 50b' is in direct fluid communication with the reboiler unit 161 and the recirculation outlet 50b'' is in direct fluid communication with the at least one storage unit 162 of the second purification unit. These two latter interconnections 50b' and 50b'' are still in fluid communication with the heat exchanging inlet 122a, albeit in an indirect manner.

Figure 5 discloses in more detail several process configurations of the flash gas outlet 55 as well as a more detailed depiction of the recirculation unit 50 in particular the expansion unit 53. The expansion unit 53 is composed of an expansion valve 531 which interconnects the condenser unit 52 with a recirculation storage unit 532. Compressed and condensed gaseous carbon dioxide end product having the third, highest degree of purity expands after the expansion valve 531 and is collected in a liquid form in the recirculation storage unit 532. Any flash gas in the expansion storage unit 532 may evaporate (evade) and can be separated via the flash gas outlet 55.

In a first embodiment as already depicted in Figures 1-4 the flash gas outlet 55 is in direct fluid communication with the main outlet 13 via flash outlet piping 56. In Figure 5 two alternative embodiments of this process configuration are depicted. In the first process configuration the flash gas outlet 55 is in direct fluid communication with the main outlet 13 upstream from the recirculation inlet 50a via flash outlet piping 56, whereas in another embodiment the flash gas outlet 55 is in direct fluid communication with the main outlet 13 at a downstream level from the recirculation inlet 50a via flash outlet piping 56'. A reason for outputting flash gas upstream or downstream from the recirculation inlet 50a can be the different temperature of the flash gas. Flash gas which is to be distributed directly to an end user (end consumer) can have another temperature than for example the flash gas temperature, which is recirculated via the recirculation inlet 50a towards the compressor unit 51.

It is to be noted that the main outlet 13 in the Figures 1-5 is depicted as a single outlet, however, it will be clear for the skilled man that different outlets 13 can be configured depending on the conditions of the gaseous carbon dioxide end product (temperature, pressure, volume rate) etc. to be delivered towards specific different end consumers.
In yet another embodiment the flash gas outlet 55 is interconnected via flash outlet piping 56" with the main inlet 11/cooling inlet 121a. Although herewith carbon dioxide end product having the highest degree of purity is being mixed with the carbon dioxide containing starting product having the lowest degree of purity, this process configuration can be beneficial under certain operational conditions wherein an excess amount of flash gas is being produced, which cannot be introduced into the main outlet.

Yet another process configuration of the flash gas outlet 55 is shown in Figure 5 by means of reference numeral 56"'. In this process configuration the flash gas outlet 55 is in heat exchanging contact with the recirculation storage unit 132 by means of a flash outlet piping 56"' being submerged in the liquid carbon dioxide end product having the third degree of purity, that is being collected in the recirculation storage unit 532. Herewith the flash gas through said outlet piping 56"' is being warmed up by the liquid carbon dioxide end product contained in the recirculation storage unit 532 and then returned to the main outlet 13 either via the flash outlet piping 56 or piping 56'. Figure 6 shows in more detail a possible process configuration of the process installation according to the invention. In this Figure 6 the multiple embodiments depicted in Figures 2-5 are shown. Depending on the operational conditions of the process installation any embodiment of the recirculation unit 50 can be activated using suitable control devices 57 incorporated in the piping 15 and 54.

In other embodiments (not shown) the flash outlet piping 56 can be directly returned to the recirculation unit 50, in particular flash outlet piping 56 can be connected to the second piping 54, upstream from either the compressor unit 51 or condenser unit 52 using suitable control devices 57. Also is it feasible to connect the flash outlet piping 56 directly to the compressor unit 51 or the condenser unit 52.

In addition to that in Figure 6 a further embodiment is depicted by means of a superheater unit 60 being incorporated in the main outlet 13. The superheater unit 60 heats the gaseous carbon dioxide end product having the third, highest degree of purity being outputted from the heat exchanging unit 122 before the end product is fed towards the end consumer for example for carbonating soft drinks or beer (or other non-food consumption applications).

The superheater unit 59 is located such that the flash outlet piping 56 is connected upstream from the superheater 59 with the main outlet 13. Flash outlet piping
56' is connected downstream from the superheater 59 with the main outlet 13. Reference numeral 60 denotes a pre-heater unit.

Likewise reference numeral 58 denotes a heat exchanging unit in a separate outlet 13' for delivering flash gas end product with different properties.

It is to be noted that the superheaters 59 and 60 can be located at different locations in the processing line depending on the final application of the carbon dioxide end product towards the end consumers. Herewith it is possible to deliver the carbon dioxide end product at different temperature and pressure levels, e.g. 5, 8 or 10 bar.

In Figure 6 reference numeral 165 denotes a separate storage unit for storing liquid carbon dioxide end product having the third degree of purity. It is remarked that this separate storage unit 165 can replace the individual storage units 162 and 532 of the second purification unit 16 and the recirculation unit 50. In that embodiment said individual storage units 162 and 532 are combined and form one general storage unit 165 resulting in more simplified and hence cheaper process installation. However the central storage unit 165 can be obviated, requiring said individual storage units 162 and 532 of both the second purification unit 16 and the recirculation unit 50. This individual storage can be achieved using larger dimensions of the piping.
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expansion valve
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second piping
recirculation flash gas outlet
recirculation flash gas outlet piping (first embodiment)
recirculation flash gas outlet piping (second embodiment)
recirculation flash gas outlet piping (third embodiment)
recirculation flash gas outlet piping (fourth embodiment)
control devices
separate condenser
superheater
pre-heater
CONCLUSIES

1. Een procesinstallatie voor het tijdens bedrijf bereiden van een koolstofdioxide (CO₂) eindproduct uit een gasvormig koolstofdioxide bevattende startproduct, dat een lager zuiverheid bezit dan het koolstofdioxide eindproduct, welke procesinstallatie een procesleiding omvat, welke proceslijn tenminste de elementen omvat:

   een hoofdinlaat voor het gasvormig koolstofdioxide bevattende startproduct, dat een eerste zuiveringsgraad bezit;

   een eerste zuiveringseenheid voor het bereiden van vloeibaar koolstofdioxide met een tweede zuiveringsgraad uit het gasvormige koolstofdioxide bevattende startproduct onder gebruikmaking van een koelmiddel, waarbij de tweede zuiveringsgraad hoger is dan de eerste zuiveringsgraad, welke eerste zuiveringseenheid tenminste omvat:

   * een koeleenheid en een warmtewisselseenheid die in warmtewisselend contact met elkaar staan,

   * waarbij de koeleenheid een koelinlaat bezit die in stroomverbinding staat met de hoofdinlaat en een koeluitlaat voor het afgeven van de vloeibare koolstofdioxide met de tweede zuiveringsgraad, en

   waarbij de warmtewisselseenheid een warmtewisselinlaat bezit die in stroomverbinding staat met de koeluitlaat alsmede een warmtewisseluitlaat voor het afgeven van koolstofdioxide eindproduct met een derde zuiveringsgraad,

   een hoofduitlaat die in stroomverbinding staat met de warmtewisseluitlaat, welke elementen door middel van geschikte eerste leidingdelen met elkaar zijn verbonden, waarbij de procesinstallatie verder omvat:

   een recirculatie-eenheid ten minste omvattende:

   * een recirculatie-inlaat die in stroomverbinding staat met de warmtewisseluitlaat;

   * een compressoreenheid, een condensatoreenheid en een expansie-eenheid voor het bereiden van vloeibaar koolstofdioxide eindproduct met de derde zuiveringsgraad, en

   * een recirculatie-uitlaat voor het afgeven van vloeibaar koolstofdioxide eindproduct met de derde zuiveringsgraad, waarbij de recirculatie-uitlaat in
stroomverbinding staat met de warmtewisselinlaat.
2. Procesinstallatie volgens conclusie 1, waarbij de procesinstallatie verder omvat een tweede zuiveringseenheid voor het bereiden van vloeibaar koolstofdioxide eindproduct met de derde zuiveringsgraad vanuit het vloeibare koolstofdioxide met de tweede zuiveringsgraad, waarbij de derde zuiveringsgraad hoger is dan de tweede zuiveringsgraad, welke tweede zuiveringseenheid ten minste omvat:
   * een zuiveringsinlaat die in stroomverbinding staat met de koeluitlaat en
   * een zuiveringsuitlaat die in stroomverbinding staat met de warmtewisselinlaat.
3. Procesinstallatie volgens conclusie 2, waarbij de tweede zuiveringseenheid een reboilereenheid omvat die de zuiveringsinlaat en de zuiveringsuitlaat met elkaar verbindt, waarbij de reboilereenheid in warmtewisselend contact staat met de hoofdinlaat.
4. Procesinstallatie volgens conclusie 3, waarbij de reboilereenheid verder tenminste een opslagsenheid omvat voor het opslaan van het vloeibare koolstofdioxide met de derde zuiveringsgraad.
5. Procesinstallatie volgens conclusie 4, waarbij de ten minste ene opslageenheid in stroomverbinding staat met de warmtewisselinlaat.
6. Procesinstallatie volgens een van de conclusies 3-5, waarbij de tweede zuiveringseenheid verder een strippereenheid omvat, welke de zuiveringsinlaat verbindt met de reboilereenheid, alsook een gasstripper die de reboilereenheid en de hoofdinlaat met elkaar verbindt.
7. Procesinstallatie volgens een van de conclusies 3-6, waarbij de recirculatie-uitlaat in vloeistofverbinding staat met de reboilereenheid.
8. Procesinstallatie volgens conclusie 7, waarbij de recirculatie-uitlaat in stroomverbinding staat met de ten minste ene opslageenheid.
9. Procesinstallatie volgens een van de conclusies 1-8, waarbij de recirculatie-eenheid verder ten minste een recirculatie flash gasuitlaat omvat voor het afgeven van gasvormig koolstofdioxide eindproduct met de derde zuiveringsgraad.
10. Procesinstallatie volgens conclusie 9, waarbij de recirculatie flash gasuitlaat in stroomverbinding staat met de hoofduitlaat.
11. Procesinstallatie volgens conclusie 9 of 10, waarbij de recirculatie flash gasuitlaat in stroomverbinding staat met de koeluitlaat.
12. Procesinstallatie volgens conclusie 9 of 10, waarbij de recirculatie flash
gasuitlaat in warmtewisselend contact staat met de expansie-eenheid.

13. Procesinstallatie volgens conclusie 12, waarbij de expansie-eenheid een expansieklep en een expansie-opslageenheid omvat.


15. Procesinstallatie volgens een van de voorgaande conclusies, waarbij de koeleerheid verder een purge-uitlaat omvat voor het purgen van niet-condenseerbare gassen.

16. Procesinstallatie volgens een van de voorgaande conclusies, waarbij de procesinstallatie verder een superheateereenheid omvat welke de warmtewisseluitlaat verbindt met de hoofduitlaat.
Fig. 2
ABSTRACT

The invention relates to a process installation for preparing - during use - a carbon dioxide (CO₂) end product from a gaseous carbon dioxide containing starting product having a lower degree of purity than the carbon dioxide end product, which process installation comprises a processing line, which processing line comprises at least the elements: a main inlet for the gaseous carbon dioxide containing starting product having a first degree of purity; a first purification unit for preparing liquid carbon dioxide having a second degree of purity from said gaseous carbon dioxide containing starting product using a cooling medium, said second degree of purity being higher than said first degree of purity, said first purification unit at least comprising: a cooling unit and a heat exchanging unit being in heat exchanging contact with each other, said cooling unit having a cooling inlet being in fluid communication with said main inlet and a cooling outlet for outputting said liquid carbon dioxide with said second degree of purity, and said heat exchanging unit having a heat exchanging inlet being in fluid communication with said cooling outlet and a heat exchanging outlet for outputting carbon dioxide end product having a third degree of purity, and a main outlet being in fluid communication with said heat exchanging outlet, which elements are interconnected by means of suitable first piping.
### SAMENWERKINGSVERDRAG (PCT)

**RAPPORT BETREFFENDE NIEUWHEIDSONDERZOEK VAN INTERNATIONAAL TYPE**

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### I. CLASSIFICATIE VAN HET ONDERWERP
(bij toepassing van verschillende classificaties, alle classificatiesymbolen opgeven)

Volgens de internationale classificatie (IPC)

| B01D53/00 | F25J3/06 |

### II. ONDERZOCHTE GEBIEDEN VAN DE TECHNIEK

Onderzochte minimumdocumentatie

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Onderzochte andere documentatie dan de minimum documentatie, voor zover dergelijke documenten in de onderzochte gebieden zijn opgenomen

### III. GEEN ONDERZOEK MOGELIJK VOOR BEPAALDE CONCLUSIES
(opmerkingen op aanvullingsblad)

### IV. GEBREK AAN EENHEID VAN UITVINDING
(opmerkingen op aanvullingsblad)

Form PCT/ISA 201 A (11/2000)
ONDERZOEKSRAPPORT BETREFFENDE HET RESULTAAT VAN HET ONDERZOEK NAAR DE STAND VAN DE TECHNIEK VAN HET INTERNATIONALE TYPE

A. CLASSIFICATIE VAN HET ONDERWERP
INV. B01D53/00 F25J3/06
ADD.

Volgens de Internationale Classificatie van octrooien (IPC) of zowel volgens de nationale classificatie als volgens de IPC.

B. ONDERZOEKDE GEBIEDEN VAN DE TECHNIEK

Onderzochte minimum documentatie (classificatie gevolgd door classificatiesymbolen)
B01D F25J

Onderzochte andere documentatie dan de minimum documentatie, voor dergelijke documenten, voor zover dergelijke documenten in de onderzochte gebieden zijn opgenomen

Tijdens het onderzoek geraadpleegde elektronische gegevensbestanden (naam van de gegevensbestanden en, waar uitvoerbaar, gebruikte trefferwoorden)
EPO-Internal, WPI Data

C. VAN BELANG GEACHTDE DOCUMENTEN

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☐ Verdere documenten worden vermeld in het vervolg van vak C.
X Leden van dezelfde octrooifamilie zijn vermeld in een bijlage

* Speciale categorieën van aangehaalde documenten
  *A* niet tot de categorie X of Y behorende literatuur die de stand van de techniek beschrijft
  *D* in de octrooiaanvraag vermeld
  *E* van derde octrooiaanvraag, gepubliceerd op of na de indieningsdatum, waarin dezelfde uitvinding wordt beschreven
  *L* om andere redenen vermelde literatuur
  *O* niet-schrijftijds stand van de techniek
  *P* tussen de voorraadsdatum en de indieningsdatum gepubliceerde literatuur

*T* na de indieningsdatum of de voorraadsdatum gepubliceerde literatuur die niet bezwaarlijk is voor de octrooiaanvraag, maar wordt vermeld ter verheldering van de theorie of het principe dat ten grondslag ligt aan de uitvinding

*X* de conclusie wordt als niet nieuw of niet inventief beschouwd ten opzichte van deze literatuur

*Y* de conclusie wordt als niet inventief beschouwd ten opzichte van de combinatie van deze literatuur met andere geciteerde literatuur van dezelfde categorie, waarbij de combinatie voor de volmaak voor de hand liggend wordt geacht

Datum waarop het onderzoek naar de stand van de techniek van internationaal type werd voltooid
8 juni 2015

Naam en adres van de instantie
European Patent Office, P.B. 5818 Patentlaan 2
NL-2280 HV Rijswijk
Tel: (+31-70) 340-2040,
Fax: (+31-70) 340-3016

De bevoegde ambtenaar
Gruber, Marco

Formulier PCT/ISA/201 (tweede blad) (Januari 2004)
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**WRITTEN OPINION**

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**International Patent Classification (IPC)**

INV. B01D5300 F25J306

**Applicant**

Haffmans B.V.

---

This opinion contains indications relating to the following items:

- ☒ Box No. I  Basis of the opinion
- ☐ Box No. II  Priority
- ☐ Box No. III  Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☐ Box No. IV  Lack of unity of invention
- ☒ Box No. V  Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- ☐ Box No. VI  Certain documents cited
- ☐ Box No. VII  Certain defects in the application
- ☐ Box No. VIII  Certain observations on the application

---

**Examiner**

Gruber, Marco

Form NL237A (Dekblad) (July 2006)
Box No. I  Basis of this opinion

1. This opinion has been established on the basis of the latest set of claims filed before the start of the search.

2. With regard to any nucleotide and/or amino acid sequence disclosed in the application and necessary to the claimed invention, this opinion has been established on the basis of:

   a. type of material:
      - ☐ a sequence listing
      - ☐ table(s) related to the sequence listing

   b. format of material:
      - ☐ on paper
      - ☐ in electronic form

   c. time of filing/furnishing:
      - ☐ contained in the application as filed.
      - ☐ filed together with the application in electronic form.
      - ☐ furnished subsequently for the purposes of search.

3. ☐ In addition, in the case that more than one version or copy of a sequence listing and/or table relating thereto has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that in the application as filed or does not go beyond the application as filed, as appropriate, were furnished.

4. Additional comments:

Box No. V  Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

   Novelty      Yes: Claims  2-16
                No: Claims  1

   Inventive step
                Yes: Claims  1-16
                No: Claims

   Industrial applicability
                Yes: Claims  1-16
                No: Claims

2. Citations and explanations

   see separate sheet
Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1 Reference is made to the following document:
   D1 EP 2 685 189 A1 (AIR LIQUIDE [FR]) 15 januari 2014
   (2014-01-15)

2 The present application does not meet the criteria of patentability, because the subject-matter of claim 1 is not new.

Document D1 discloses (ref. abstract) a process installation for producing a carbon dioxide end product from a gaseous carbon dioxide containing starting product (e.g. flue gas) with a lower purity of carbon dioxide than the end product, the process line of the process installation comprising (ref. Fig. 5 and col. 5)

- a main inlet (45) for the intake of a gaseous carbon dioxide (e.g. flue gas) containing starting product having a first degree of purity;

- a first purification unit (43) for generating liquid carbon dioxide with a second degree of purity from the gaseous carbon dioxide by using a cooling medium, whereby the second degree of purity is higher than the first one, the first purification unit comprising at least

- a cooling unit (43) and a heat exchanging unit (43) which are in heat exchanging contact with each other, whereby

- the cooling unit comprises a cooling inlet (line coming from compressors C) being in fluid connection with the main inlet (41) and a cooling outlet (line 47) for outputting liquefied carbon dioxide having a second degree of purity (ref. col. 5, l. 22,23), which second degree of purity is higher than the first degree of purity of the gaseous carbon dioxide starting product; and whereby

- the heat exchanger comprising a heat exchanging inlet (lines downstream of valves V4 or V5) and a heat exchanging outlet (line 65) for outputting liquid carbon dioxide end product having a third degree of purity, and

- a main outlet (61) connected to the heat exchanging outlet (65) whereby the process installation further comprises

- a recirculation unit at least comprising

- a recirculation inlet (63) being in fluid communication with said heat exchanging outlet (line 65);
- a compressor unit (p2), a condenser unit (separator 49) and an expansion unit (V4) for preparing liquid carbon dioxide end product having said third degree of purity, and

- a recirculation outlet (e.g. line 53) for outputting liquid carbon dioxide end product having said third degree of purity, said recirculation outlet being in fluid communication with said heat exchanging inlet (line downstream of V4).

D1 is therefore considered novelty destroying for the subject matter of claim 1.

3

It is, at present, not clear to which extent the subject matter of the dependent claims causes unexpected or surprising effects with respect to what is disclosed in the above mentioned prior art. Therefore, no inventive activity can be acknowledged for the time being.