(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 4 012 249 A1

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:
15.06.2022 Bulletin 2022/24

(21) Application number: **21020118.2**

(22) Date of filing: **03.03.2021**

(51) International Patent Classification (IPC):
F17C 11/00 *(2006.01)*

(52) Cooperative Patent Classification (CPC):
F17C 11/002

(84) Designated Contracting States:
AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL PT RO RS SE SI SK SM TR
Designated Extension States:
BA ME
Designated Validation States:
KH MA MD TN

(30) Priority: **11.12.2020 EP 20020608**

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(54) METHOD AND AN APPARATUS FOR SUPPLYING ETHYNE OF A DESIRED PURITY TO A CONSUMER

(57) The invention relates to a method (200) of supplying ethyne of a desired purity to a consumer (150), comprising withdrawing (210) ethyne from a container (112, 114) in which ethyne is solved in a solvent under pressure, determining (220) a concentration of the solvent within the withdrawn ethyne (10) and transporting (240) the withdrawn ethyne (10) to the consumer (150) in dependence (230) of the determined concentration, as well as an apparatus (100) for performing such a method (200).

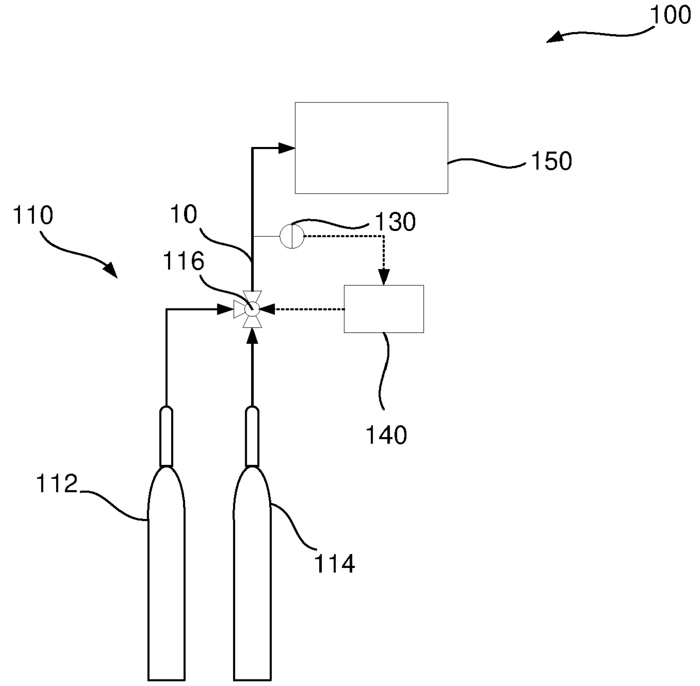

Fig. 1

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Description

[0001] The present invention relates to a method and an apparatus for supplying ethyne of a desired purity to a consumer.

Background of the invention

[0002] High-quality (or purity) ethyne (also referred to as acetylene in the applied sciences) supply is required by some heat treatment processes, like Low Pressure Carburizing (LPC) and Diamond-Like Carbon coating (DLC), in the industry which requires a high standard of surface performance, especially for automobile industry. Typical purities are 99.5% for LPC, and 99.7% or 99.9% for DLC.

[0003] Typically, ethyne is offered in a dissolved state to customers. In most regions of the world, acetone is widely used as the solvent due to its properties. However, acetone is quite volatile, so solvent withdrawing is inevitable in the applications, and solvent concentration differs depending on the solvent type, temperature, gas flow rate and cylinder pressure, etc. The solvent (acetone vapor CH_3COCH_3) sent to LPC furnaces can lead to negative impacts, including contaminating the pipeline, valves, mass flow controllers, gas injection nozzles, producing variances to the process control (by adding extra oxygen), creating soot and/or tar, and lowering the efficiency of the vacuum system, gauges and pump, and decreasing yield and product quality, etc.

[0004] Solvent withdrawing is well known by gas suppliers and users, and some measures are taken to alleviate the impact to the process, including, for example, limiting acetylene withdrawal rate to a maximum value, e.g. 500 l/h per cylinder (or other container), defining a minimum residual cylinder pressure or weight in use (switch-over pressure), typically in the range from 3 bar to 8 bar, or installing filters made of porous material. All of the mentioned measures for purity control are based on empirical observations and are therefore either prone to failure in application or cause excessive costs (e.g. by defining a higher residual pressure and therefore wasting more ethyne than necessary).

[0005] Putting aside the real performance of the above-mentioned actions, it is difficult to measure or analyze the acetone concentration in the acetylene gas.

[0006] Existing solutions for determining a composition of a gas mixture include gas chromatography, which is a highly precise, but difficult to use, expensive and fragile technique. It is therefore not easily integrated into a production setup in the heat treatment industry.

[0007] Therefore, a robust and easy to implement solution for determining the solvent concentration within the withdrawn ethyne is desired in order to directly control the ethyne purity.

Disclosure of the invention

[0008] According to the invention a method of supplying ethyne (also known as acetylene) of a desired purity to a consumer as well as an apparatus for performing such a method with the features of the independent claims are proposed. Advantageous embodiments are subject-matter of the dependent claims and the description herein below.

[0009] The invention relies on determining a concentration of a solvent, particularly acetone, within a stream of ethyne withdrawn from a container and transporting the withdrawn ethyne depending on the determined concentration. It is therefore possible, to only transport ethyne if the purity requirement is fulfilled, thereby ensuring quality and safety of downstream process products and equipment, respectively. In some embodiments, the purity of the withdrawn ethyne may also be improved, if the determined concentration does not fulfill the purity requirement.

[0010] According to the invention, a method of supplying ethyne of a desired purity to a consumer, comprises withdrawing ethyne from a container in which ethyne is solved in a solvent under pressure, determining a concentration of the solvent within the withdrawn ethyne and transporting the withdrawn ethyne to the consumer in dependence of the determined concentration. In some embodiments, the solvent may comprise acetone and/or dimethylformamide or other organic compounds. As mentioned above, this enables stable quality of downstream process products and improves safety and maintenance effort for downstream equipment

[0011] Advantageously, determining the concentration of the solvent comprises determining a density of the withdrawn ethyne and/or reacting a part of the withdrawn ethyne with oxygen and determining the amount of oxygen consumed in the reaction. Both of these determination methods are robust, accurate and easy to implement within existing or new facilities and do not require expensive or overly complex equipment

[0012] Preferably, the withdrawn ethyne is transported to the consumer, when the determined concentration of the solvent is below a threshold concentration. Thereby, a minimum purity of ethyne can be set and guaranteed.

[0013] Preferably, a measure is taken, when the determined concentration of the solvent exceeds a threshold concentration. This enables control of the purity of ethyne supplied to downstream consumers, especially in situations when this purity is lower than the desired purity or approaching a minimum purity.

[0014] The measure, in such a case, advantageously comprises one or more of issuing a warning signal, stopping withdrawal of ethyne from the container and starting withdrawal of ethyne from a second container. In some embodiments, the measure may also comprise purifying the withdrawn ethyne by separating the solvent at least partially from the ethyne. This enables a variety of counter measures to ensure the purity of ethyne delivered to the consumer meets quality requirements.

[0015] In embodiments in which the measure comprises purifying the ethyne, the method comprises removing the at least one solvent at least partially from the withdrawn ethyne affording purified ethyne by controlled condensation of the at least one solvent, and providing the purified ethyne to the consumer.

[0016] Preferably, removing the at least one solvent is at least partially effected by cryotrapping, particularly at a pressure of more than 100 or 150 kPa (high pressure) and/or by adsorbing the at least one solvent, particularly at a pressure of below 150 or 100 kPa (so-called medium or middle pressure, also referred to as low pressure herein). This provides a very effective and highly controllable possibility for ethyne purification. It is to be noted, however, that cryotrapping may also be performed at the lower pressure (i.e. in a middle or medium pressure pipe) which provides benefits regarding operating security as ethyne under high pressure conditions tends to be thermodynamically unstable.

[0017] The relative content of ethyne in the purified ethyne advantageously exceeds 99.0%, 99.5%, 99.8%, 99.9% or 99.99%. In such qualities, remaining impurities have no or at least only acceptable adversary effects on downstream processes and/or equipment The threshold concentration is preferably below 10%, 5%, 3%, 1%, 0.5%, 0.1%, 0,01% or 0.001% by volume. This provides a large range of relevant purities and, therefore, flexible possibilities of process control or regulation. In advantageous embodiments, the threshold (or thresholds) may be chosen by a user of the invention.

[0018] An apparatus for supplying ethyne of a desired purity to a consumer, according to the invention, comprises means for connecting at least one container configured to store ethyne solved in a solvent to the apparatus, a concentration determining device, configured to determine a concentration of the solvent within a stream of ethyne, and means for transporting ethyne to the consumer with or without an intermedium buffer vessel, depending on a concentration value determined by the concentration determining device. The apparatus may comprise (further) means for performing a method as described herein. Such an apparatus profits from the advantages mentioned in connection with the method according to the invention, above.

[0019] Particularly, the apparatus may comprise at least one trap for trapping at least one solvent from a mixture containing ethyne and the at least one solvent, and means for transporting ethyne from the one or more connector(s) through the trap to the consumer, so as to purify the withdrawn ethyne, if necessary.

[0020] Such a trap may comprise a cryotrap, configured to condense the at least one solvent and to not condense ethyne. This is based on different condensation temperatures at a given pressure, wherein ethyne typically exhibits a lower condensation temperature as compared to the relevant solvent(s), particularly acetone or dimethylformamide.

[0021] Alternatively or additionally, the trap may comprise at least one adsorber with an adsorbent selectively adsorbing the at least one solvent over ethyne. Such adsorbents typically comprise highly porous materials such as active carbon or porous ceramics. Depending on the size of the pores in such an adsorbent, condensation temperatures may be strongly increased locally, due to surface effects. Therefore, an adsorber may have a similar effect on residual solvent concentration as the above mentioned cryotrap. In some embodiments, a combination of cryotrapping and adsorbing of the at least one solvent may be implemented, e.g. by cooling the adsorber to cryogenic temperatures or by arranging a cryotrap upstream of the adsorber. This latter arrangement may provide the advantage, that rough purification of ethyne may be effected in the cryotrap, whereas fine purification may be effected in the adsorber due to a much higher contact surface between the ethyne and the adsorbent in comparison to the contact surface between a wall of the cryotrap and the ethyne.

[0022] Further advantages and embodiments of the invention will be discussed in connection with the appended drawings and the description thereof.

[0023] It is to be understood that the features mentioned and discussed herein are not only useable in the combinations explicitly mentioned, but can also be used in different combinations thereof or alone, without departing from the scope of the present invention.

[0024] The invention is described herein below with reference to an exemplary embodiment that is illustrated schematically in the drawings.

Description of the drawings

[0025]

Figure 1 shows an advantageous embodiment of an apparatus according to the invention in a schematic block diagram.

Figure 1A shows another advantageous embodiment of an apparatus according to the invention, comprising a solvent trap.

Figure 2 shows an advantageous embodiment of a method according to the invention in a simplified flow diagram.

Figure 3 schematically shows an advantageous embodiment of an apparatus with an exemplary embodiment of a concentration determining device, usable in connection with the invention.

Figure 4 schematically shows an advantageous embodiment of an apparatus with another exemplary embodiment of a concentration determining device, usable in connection with the invention.

Detailed Description

[0026] In Figure 1, an advantageous embodiment of an apparatus according to the invention is depicted in a schematic block diagram and collectively referred to as 100. An advantageous embodiment of a method according to the invention is schematically shown in Figure 2 and collectively referred to as 200. Generally, references to components of an apparatus in the following refer to the apparatus 100 as shown in Figure 1, whereas references to steps of a method generally refer to the method 200 as illustrated in Figure 2.

[0027] The apparatus 100 comprises a gas supply unit 110, a concentration determining device 130, a treatment unit 150 and a computing unit 140. The gas supply unit may, for example, comprise a first container 112, a second container 114 and a selection means 116, which may be e.g. a three-way-valve. The first 112 and second 114 containers may each comprise one or more storage tanks and are configured to store ethyne (aka acetylene) in a dissolved state, i.e. mixed with a solvent, for example acetone and/or dimethylformamide.

[0028] The selection means 116 is configured to selectively withdraw a gas 10 from at least one of the first 112 and second 114 containers. In some embodiments, it may also be configured to withdraw the gas 10 from both first and second containers 112, 114, simultaneously. The selection means 116 is controlled by the computing unit 140, particularly electronically.

[0029] The concentration determining device 130 is configured to determine a concentration of the solvent, present in the containers 112, 114 within the withdrawn gas 10 and is arranged between the gas supply unit 110 and the treatment unit 150. In use, the concentration determining unit 130 delivers a signal containing information about the concentration of the solvent to the computing unit 140. Specific embodiments of such a concentration determining device 130 are described herein below in connection with Figures 3 and 4.

[0030] The computing unit 140 may be provided, by way of example, as a microcomputer, a computing center, a server or a control unit, configured to control a complete operation of the apparatus 100.

[0031] The treatment unit 150 may comprise a furnace configured to controlled heating of an article, a substrate or other objects to be treated, for example, a furnace configured to perform a (low pressure) carburization (LPC) process of a steel article, a furnace for diamond-like carbon coating (DLC) or similar processes requiring ethyne of high purity. The treatment unit 150 may also be controlled by the computing unit 140.

[0032] During use of the apparatus 100, the method 200 may be applied. in a first step 210 of the method 200, gas 10 is withdrawn from one of the first 112 and second 114 containers, for example from the first container 112. Step 210 may, therefore, comprise controlling the selecting means 116 to selectively connect the first container 112 to piping leading the withdrawn gas 10 to the treatment unit 150, passing the concentration determining device 130. Optionally, selecting means 116 may also be configured for controlling a downstream pressure of the gas 10. In such a case, step 210 may also comprise controlling selection means 116 to set or regulate a target pressure in the withdrawn gas 10.

[0033] In a concentration determining step 220, a concentration of a solvent, for example acetone or dimethylformamide, within the withdrawn gas 10 is determined. Details regarding the method of determining the concentration will be discussed below in connection with the specific embodiments of concentration determining devices 130.

[0034] In a decision step 230, the determined solvent concentration is compared to a threshold value, which optionally may be selected by a user or may be preset. If the solvent concentration is below the threshold value, the method 200 continues with a supply step 240, in which the withdrawn gas 10 is transported to the treatment unit 150 and used for the treatment process performed there. The method 200 may then return to step 210 and continue withdrawing gas 10 from the first container 112.

[0035] If, however, in step 230, the threshold value is reached or exceeded, the method 200 continues with an action step 250, in which a measure is taken in order to return the solvent concentration to below the threshold value. Such a measure may comprise one or more of issuing a warning signal, e.g. to a user of the apparatus 100 or the computing unit 140, stopping withdrawal of ethyne from the first container 112 and starting withdrawal of ethyne from the second container 114. Particularly, a control signal may be sent to the selecting means 116, such that the first container 112 is disconnected from the supply line to the treatment unit 150 and the second container 114 is connected to the supply line in its stead. The method 200 may then continue with step 210, now withdrawing the gas 10 from the second container 114 instead of the first container 112. This enables seamless operation of the treatment unit 150 while warranting the required or desired purity of the supplied gas 10.

[0036] The discussed issuance of a warning signal to a user of the apparatus enables the user to replace the first container 112 with a fresh first container 112, so that in a later step 250, the selecting means 116 may switch the gas supply unit 110 back to the first container 112 without adversely affecting the purity of gas 10.

[0037] In Figure 1A, an advantageous variation of the apparatus 100, as described with reference to Figure 1, is schematically shown and collectively referred to with 100A. The apparatus 100A additionally comprises a solvent trapping device 135. The solvent trapping device 135 in the shown embodiment is connected to the piping upstream of it via a three-way-valve, controlled by the computing unit 140 according to the solvent concentration determined.

[0038] The apparatus 100A (as also 100) may comprise a pressure reducing valve to set a gas pressure to a pressure level required by treatment unit 150. The pressure reducing valve may be regulated or controlled pneumatically through a pressure control device. In alternative embodiments, the pressure control may also be effected by electronic, hydraulic, magnetic or any other suitable means.

[0039] Downstream of the pressure reducing valve a decomposition barrier may be arranged. A decomposition barrier, also known as flashback arrestor, is typically configured to stop a decomposition front running from downstream it into an upstream direction in order to prevent a dangerous chain reaction, in which gas supply unit 110 might otherwise be critically damaged or destroyed. Such a decomposition front may arise from too high temperatures in downstream components, particularly in treatment unit 150. During such a decomposition event, ethyne exothermally reacts to the elements carbon and hydrogen, thereby causing a rise in pressure due to thermal expansion. The decomposition barrier prevents the decomposition front from expanding upstream by diverting flow and decelerating the reaction. An automatically closing valve at the entrance of the decomposition barrier may be provided to physically disconnect the barrier from upstream components of apparatus 100/100A.

[0040] Regarding the position, in which the solvent trapping device 135 may be suitably installed, there positions in a high pressure region of apparatus 100A, i.e. upstream of the mentioned pressure reducing valve, and positions in a low pressure region of apparatus 100A, i.e. downstream of the pressure reducing valve and preferably also downstream of the decomposition barrier. Typically, the high pressure region is operated at a pressure in the range of more than 100 or 150 kPa up to 2.5 MPa, while the low (or medium resp. middle) pressure region may be operated at a pressure in the range of 20 kPa to below 150 or 100 kPa. Particularly, the separation limit between the two pressure regions (100 kPa or 150 kPa) may depend on local legislation and/or standards.

[0041] An arrangement of the solvent trapping device 135 in the high pressure region is preferred for embodiments in which the solvent trapping is effected by lowering the temperature of gas 10 to a temperature level at which the saturation pressure of the solvent is at or below a pressure level that corresponds to an acceptable partial pressure level of the solvent within the ethyne stream provided to the treatment unit 150. Therefore, a cryotrap may be installed in the high pressure region. The cryotrap may particularly comprise a vessel through which the gas 10 is conducted and which comprises a heat exchanger, wherein the medium to which heat is transferred may comprise liquid nitrogen, liquid argon or any other suitable medium capable of being operated at cryogenic temperatures, particularly at temperature levels below -20 °C. Therefore, in use, the solvent, if contained within the gas 10 withdrawn from gas supply unit 110, will at least partially condense at the wall of the vessel of the cryotrap, such that a remaining partial pressure of the solvent is below a tolerable value. A regeneration of the cryotrap may simply be effected by emptying the vessel, e.g. in predefined time intervalls or when the vessel is filled to a certain degree or level, as may be determined by a sensor within the cryotrap.

[0042] In embodiments in which the solvent trapping is effected by adsorbing solvent molecules on an adsorber, the preferred position is in the low pressure region of apparatus 100A in order to prevent ethyne adsorption to the adsorber. In other words, the lower pressure increases adsorption selectivity towards the solvent. Such an adsorber may, for example, be configured for temperature swing adsorption (TSA), pressure swing adsorption (PSA) or vacuum pressure swing adsorption (VPSA). Advantageously, two adsorbers may be provided in parallel and operated alternately, such that during an adsorption phase, in which a first adsorber is provided with the gas 10 withdrawn from gas supply unit 110, a second adsorber is regenerated, i.e. adsorbed solvent is withdrawn from the second adsorber, e.g. by lowering the pressure (PSA/VPSA) or increasing the temperature (TSA) and optionally flushing with a purge gas (e.g. gas 10 or a partial stream of purified gas 10). This operation scheme may be reversed periodically, such that each of the first and second adsorbers is regenerated and used for purification alternatingly.
The solvent removed from the trapping device 135 (cryotrap and/or adsorber) may be collected in a solvent stream 14.

[0043] In cases, in which an apparatus 100A comprising a solvent trapping device 135 is used, step 250, described above in connection with Figure 2, may comprise controlling the three-way-valve 118 to open the path towards the solvent trapping device 135 and closing the direct path towards the treatment unit 150. In some embodiments, the concentration determining device 130 may also be arranged downstream the solvent trapping device 135, thereby enabling a determination after which time, a regeneration of the solvent trapping device 135 may be necessary or a determination, when a changeover between the first 112 and second 114 containers of the gas supply unit 110 is necessary. This may be the case, when the solvent concentration in the withdrawn raw ethyne 10 upstream the solvent trapping device 135 increases to such high amounts that the purification capacity of the solvent trapping device is insufficient to lower this concentration to a concentration below the acceptable threshold.

[0044] In Figure 3 an advantageous embodiment of an apparatus 100 with an exemplary embodiment of a concentration determining device 130, usable in connection with the invention is schematically shown.

[0045] The depicted apparatus 100 corresponds, in its essential parts, to the apparatus 100 as described in connection with Figure 1, above. Attention is drawn to the specific design of the concentration determining device 130, which in this example comprises a combustion chamber 132, configured to react a partial stream 13 of the withdrawn gas 10 with oxygen 136 to an exhaust gas product 15. The combustion chamber 132 may be equipped with suitable catalyst materials in order to enable or accelerate the reaction of the gas of partial stream 13 with the oxygen 136. The device 130 further comprises a sensor 134 for determining an oxygen concentration within the exhaust gas product 15. This sensor 134 may, for example, be provided in the form of a lambda sensor, particularly a broadband lambda sensor, such that a signal provided by the sensor contains information about the concentration of oxygen within the exhaust gas product Combined with an information about the amounts of oxygen 136 and gas in partial stream 13, this enables determining a composition of the partial stream 13. This determination is based on different oxygen consumption during combustion by ethyne (2.5 molecules of oxygen per molecule of ethyne) and solvent (4 molecules of oxygen per molecule of acetone, or 4.25 molecules of oxygen per molecule of dimethylforamide), respectively. By determining the oxygen content in the exhaust gas product 15 while controlling the feed streams 136, 13, the amount of oxygen consumed by the reaction in the combustion chamber 132 can be easily calculated and the composition of the two-compound stream 13 can be deduced from this information. The temperature of the sensor 134 may be controlled to be in a range in which the sensor 134 exhibits a high sensitivity towards oxygen concentration, e.g. by a sensor heater. This is an important influence parameter regarding the overall performance of the invention in this embodiment.

[0046] In step 220, therefore, when using a concentration determining device 130 as shown in Figure 3, a partial stream 13 of the withdrawn gas 10 is directed to the combustion chamber 132. This partial stream 13 can amountto, for example, less than 10%, 5%, 1%, 0.1% or 0.01% of the withdrawn gas 10. The amount of the partial stream 13 may be controlled with a mass flow controller, for example. At the same time, an oxygen gas stream 136 is provided to the combustion chamber 132, again under controlled conditions, such that a ratio between the amount of partial stream 13 and oxygen gas 136 in combustion chamber 132 is known. The mixture resulting in combustion chamber 132 may then be ignited in order to react the gas of partial stream 13 completely with the oxygen of stream 136. Therefore, it is advantageous to control stream 136 regarding quantity such that an over stoichiometric amount of oxygen is present within the combustion chamber at any time, to ensure complete oxidation reaction. The produced exhaust gas 15 is then analyzed with the sensor 134, e.g. a conventional lambda sensor or another type of electrochemical oxygen probe, such that a signal of the sensor includes information about the oxygen content within the exhaust gas 15. Such a signal from an electrochemical sensor (i.e. a concentration element) may be analyzed using the Nernst equation or a mathematical model derived from it

[0047] In a similar embodiment, the sensor signal may be used to control the amount of oxygen 136 provided to the combustion chamber 132, e.g. by a mass flow controller. Preferably, the amount of oxygen in such a case is controlled in such a manner, that the signal of sensor 134 remains constant, while the amount of partial stream 13 is also kept at a constant flow. Then, the amount of oxygen 136 provided to the combustion chamber 132 can be used for determining the ratio of ethyne to solvent in partial stream 13.

[0048] In Figure 4 an advantageous embodiment of an apparatus 100 with another exemplary embodiment of a concentration determining device 130, usable in connection with the invention is schematically illustrated. In this case, the working principle of concentration determining device 130 is based on a measurement of a mass density of the withdrawn gas 10, e.g. by measuring buoyancy acting upon a probe body 135 within the gas 10. This relies on the different densities of ethyne (~1.1 kg/m^3 at standard temperature and pressure) and the solvent vapor (~2.6 kg/m^3 for acetone, ~3 kg/m^3 for dimethylforamide), respectively. The relative densities, therefore, correspond to about 0.9 for ethyne, about 2 for acetone and roughly 2.5 for dimethylformamide, as compared to air. Therefore, the higher the measured density of the withdrawn gas 10 (i.e. the higher the buoyancy), the higher the solvent concentration. A calibration of gas densities for different solvent contents is sufficient to calculate the solvent concentration. Therefore, in such a case, step 220 comprises determining the density of the gas stream 10, e.g. by measuring the buoyancy of a probe body 135 within the gas 10 and calculating the solvent concentration from the density determined.

[0049] As already mentioned, not all the features discussed herein need to be strictly realized in the described combination. It may, for example, be advantageous to perform some of the steps of method 200 in a different, e.g. reversed, order or to combine several steps or to perform some of the steps simultaneously or in parallel to one another. As an example, determining the concentration of the solvent within gas 10 (step 220) may be performed simultaneously with providing the gas 10 to the treatment unit 150 (step 240). In such a case, the threshold value is preferably selected such that even after exceeding the threshold concentration, the minimum purity requirements for ethyne, as acceptable for the treatment in step 240, are still met This means that a switch over (step 250) to the second container 114 is still timely enough, even after exceeding the threshold in step 230, if performed directly after the threshold value was exceeded. A similar argumentation may hold for different sets of method steps. Even the stepwise arrangement of method 200 altogether may be altered in favor of a continuous performance, so that the existence of distinct steps may be avoided altogether, in some embodiments. Therefore, the stepwise performance of method 200 is to be understood solely for illustration purposes and in no way limiting the scope of the present invention.

Claims

1. Method (200) of supplying ethyne of a desired purity to a consumer (150), comprising
withdrawing (210) ethyne from a container (112, 114) in which ethyne is solved in a solvent under pressure,
determining (220) a concentration of the solvent within the withdrawn ethyne (10) and
transporting (240) the withdrawn ethyne (10) to the consumer (150) in dependence (230) of the determined concentration.

2. Method (200) according to claim 1, wherein determining (220) the concentration of the solvent comprises
determining a density of the withdrawn ethyne (10) and/or
reacting a part (13) of the withdrawn ethyne (10) with oxygen (136) and determining the amount of oxygen consumed in the reaction.

3. Method (200) according to claim 1 or 2, wherein the withdrawn ethyne (10) is transported (240) to the consumer (150), when the determined concentration of the solvent is below (230) a threshold concentration.

4. Method (200) according to any of the preceding claims, wherein a measure is taken (250), when the determined concentration of the solvent exceeds (230) a threshold concentration.

5. Method (200) according to claim 4, wherein the measure (250) comprises one or more of issuing a warning signal, stopping withdrawal of ethyne from the container (112) and starting withdrawal of ethyne from a second container (114).

6. Method according to claim 4, wherein the measure (250) comprises
removing the at least one solvent (14) at least partially from the withdrawn ethyne (10) by controlled condensation of the at least one solvent affording purified ethyne, and
providing the purified ethyne to the consumer (150).

7. Method (200) according to claim 6, wherein removing (250) the at least one solvent (14) is at least partially effected by cryotrapping, preferably at a pressure of more than 100 or 150 kPa and/or adsorbing the at least one solvent (14), preferably at a pressure of below 150 or 100 kPa.

8. Method (200) according to claim 6 or 7, wherein a relative content of ethyne in the purified ethyne exceeds 99.0%, 99.5%, 99.8%, 99.9%, 99.99% or 99.999%.

9. Method (200) according to any one of claims 3 to 8, wherein the threshold concentration is below 10%, 5%, 3%, 1%, 0.5%, 0.1%, 0,01% or 0.001% by volume.

10. Method (200) according to any of the preceding claims, wherein the solvent comprises acetone and/or dimethylformamide.

11. Apparatus (100) for supplying ethyne of a desired purity to a consumer (150), comprising
means (116) for connecting at least one container (112, 114) configured to store ethyne solved in a solvent to the apparatus (100),
a concentration determining device (130), configured to determine a concentration of the solvent within a stream of ethyne (10), and
means for transporting ethyne to the consumer (150), depending on a concentration value determined by the concentration determining device (130).

12. Apparatus (100) according to claim 11, comprising means (130, 140, 135) configured to perform a method (200) according to any one of claims 1 through 10.

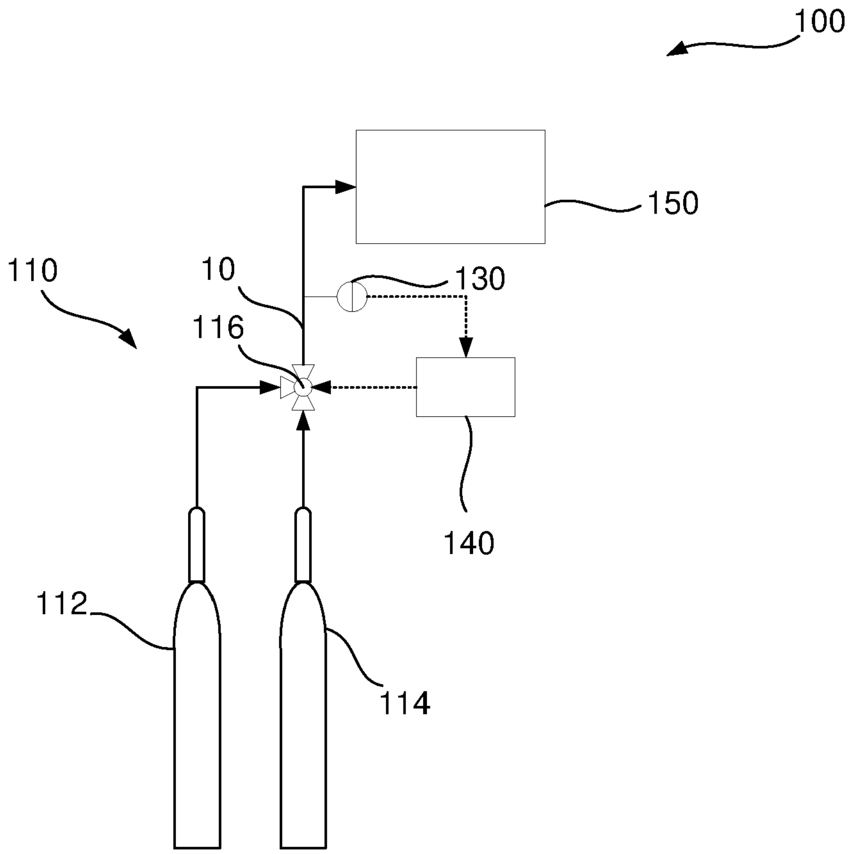

Fig. 1

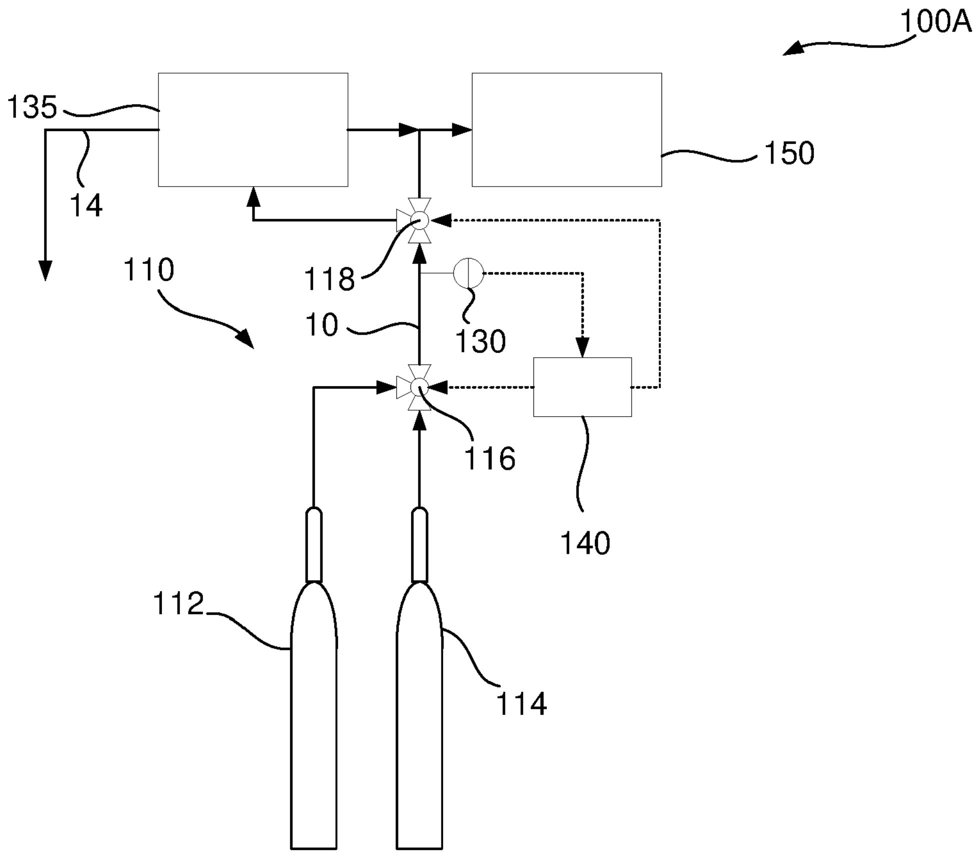

Fig. 1A

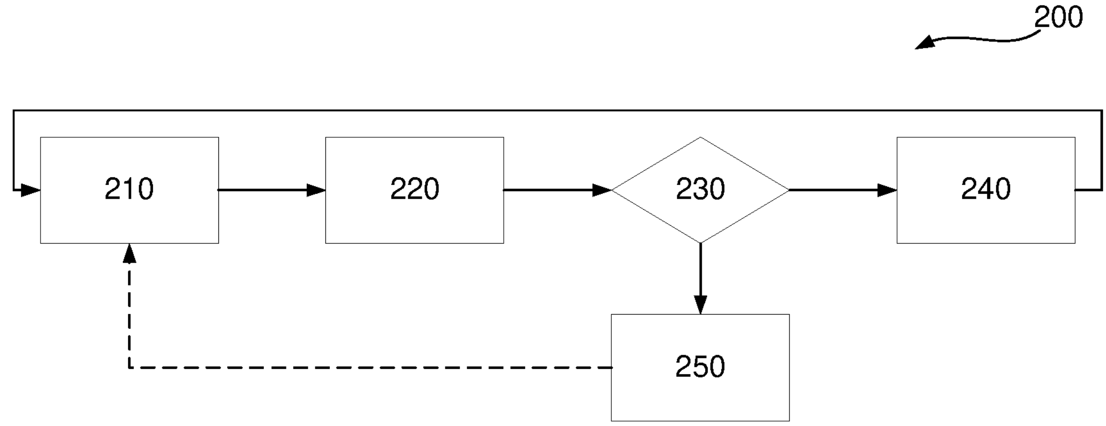

Fig. 2

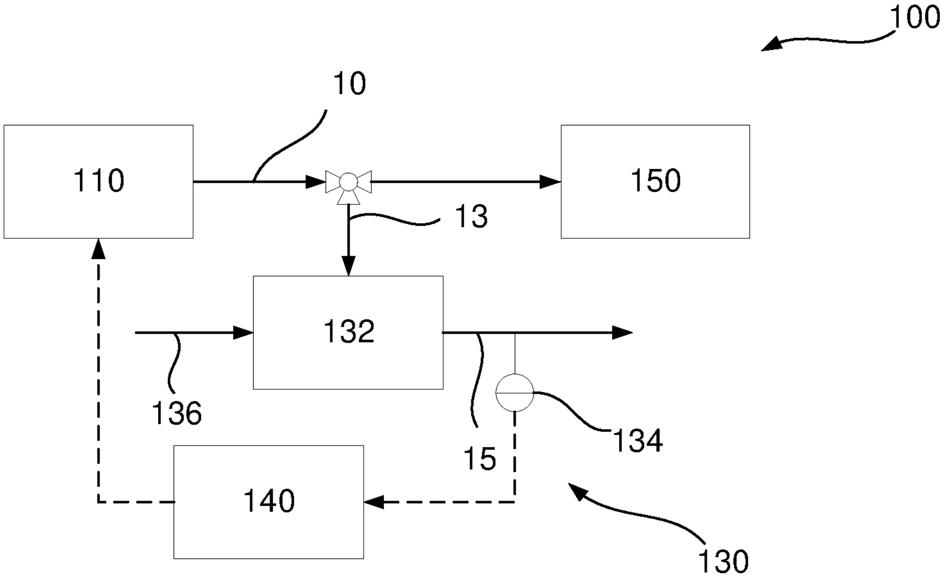

Fig. 3

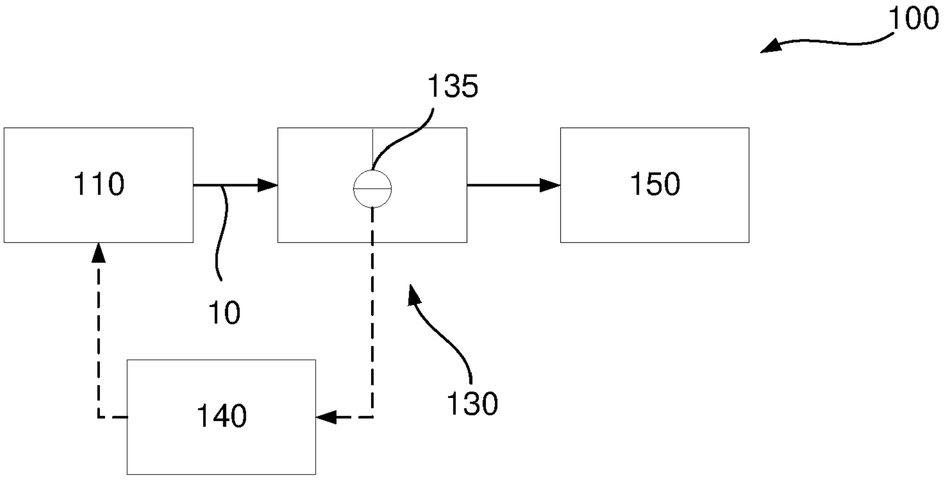

Fig. 4

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EUROPEAN SEARCH REPORT

Application Number
EP 21 02 0118

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	JP 2013 194004 A (TAIYO NIPPON SANSO CORP) 30 September 2013 (2013-09-30) * abstract; figures; example 1 * * paragraph [0025] - paragraph [0028] * -----	1-12	INV. F17C11/00
A	JP S58 84896 A (NICHIGO ACETYLEN) 21 May 1983 (1983-05-21) * abstract * -----	1-12	

TECHNICAL FIELDS SEARCHED (IPC)
F17C

The present search report has been drawn up for all claims

1

Place of search	Date of completion of the search	Examiner
The Hague	23 July 2021	Lapeyrère, Jean

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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23-07-2021

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
JP 2013194004 A	30-09-2013	NONE	
JP S5884896 A	21-05-1983	JP S601352 B2	14-01-1985
		JP S5884896 A	21-05-1983

EPO FORM P0459

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82