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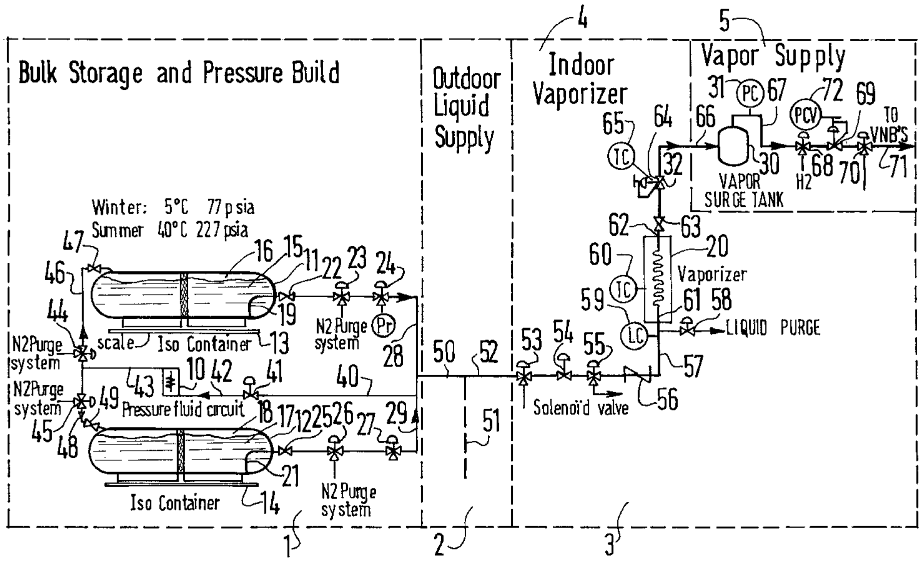
(54) **Title:** SYSTEM AND METHOD FOR HIGH FLOW DELIVERY OF NH3 FOR FLAT DISPLAY PANEL MANUFACTURE



(57) **Abstract:** A system for high flow delivery of a liquefied product such as liquid ammonia to a flat display panel manufacture location or the like comprising means for bulk storage of liquefied product which includes at least one container to store said product in liquid phase with a gaseous phase of said product located above said liquid product, liquid supply delivery means for flowing said liquid product from said bulk storage means to main vaporizer means while keeping said liquid product substantially in liquid form throughout said delivery to said main vaporizer means and main vaporizer means to vaporize said liquid product in response to the request of gaseous product made at the location of manufacture.

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System and method for high flow delivery of NH3 for flat display panel manufacture

The invention relates to a system and method for high flow delivery of liquefied products such as NH3 for use in the manufacture of flat panel display. NH3 gaseous product is particularly useful for the cleaning-up of CVD reactors or the like.

Today the requirement of the manufacturers of these panels is the generation of flow rates of liquefied product from 2000 liters/min down to no flow in a matter of seconds. Delivery equipments and systems need to be able to respond to the high and variable flow requirements while maintaining a given delivery pressure, which does not fall below a minimum delivery pressure.

It is known from US-A-6 032 483 a system and method of delivery of a vapor phase product to a point of use, which describes a system and method to deliver liquid phase product to a vaporizer to form vapor to supply high gas flow rates to a point of use which system requires the use of a pump. The pump could experience mechanical failure that would stop all product flow. However, unless special design consideration are given to assure product remains in the liquid phase, a gaseous phase will be present in the line when there are long delivery lines to the point of vaporization. The presence of gaseous phase will cause problems delivering liquid.

US-A-6 647 930 describes a system and method to deliver high flow rates of liquids. A condenser is required to assure liquid delivery to a secondary containment tank where vaporization occurs. The secondary containment tank needs to be custom designed to allow for piping to be run inside for containment of heat transfer fluid, liquid product entry, gas phase withdrawal, pressure, temperature, and level measurement.

US-A-6 637 212 discloses a NH3 gas delivery system having a constant impurity level in the liquid phase and gaseous phase, by completely vaporizing all of the liquefied gas and the soluble impurities that enter the vaporization unit. Accordingly, contrary to the present invention, the invention disclosed in this patent uses a non conventional vaporizer.

The difficulties with supplying bulk flows of liquefied gases including delivery at a point of use which may be far away from the source, are met by the existing systems as described above, can be summarized as follows :

- Delivery of liquefied product from a source in the gas phase is limited to how much energy can be transferred to liquid phase to produce the gaseous phase. Inadequate vaporization of the liquid phase to produce gas phase results in a reduction of head-space pressure and resultant limiting of the gas flow rate that can be delivered.
- The pressure drop due to the distribution lines of gases from a source container over long distance creates another restriction to the maximum flow rates that can be achieved.
- There are also limitation to how quickly the systems can respond to the varying point of use flow rates (In many cases, there are several points of use connected to the same supply system, with several lines having various lengths, with a requirement of product at each point of use which is unpredictable in time and independent from each others).
- The larger source containers, will normally be located outdoors experiencing wide variations in temperature and humidity that affect the pressure and capability to transfer energy to the source container.(the requirement may be e.g. from about 5°C, $5x10^5$ Pascal (winter) to 40°C, $15x10^5$ Pascal (summer))
- Large source containers can concentrate impurities in the remaining liquid phase when withdrawing gas phase.
- Liquid phase delivery product needs to be maintained in the liquid phase over long distances outdoors.
- Vapor surge capacity needs to be large to compensate for flow requirement changes.

The above drawbacks of the prior art systems and methods are solved by the present invention, wherein according to one embodiment a pressure build circuit (vaporizer) is used at the source, potentially being an ISO-Container of more than 20,000 litre capacity, to help maintain pressure in the container during high flow withdrawal of liquid. This pressure maintained in the gaseous headspace above liquid in the container helps to keep liquid phase during delivery, while helping delivery of this liquid without the need of a pump. The NH3 product is preferably delivered from the NH3 container source in liquid phase to a main vaporizer located at a remote distance from the source. Vaporization is preferably done indoors near the point of use of the NH3 gas minimizing the distance to transport gas phase product. According to a preferred embodiment, back pressure regulation means (back pressure regulator) is used to minimize the size of the gas surge tank needed to compensate for potential flow requirement changes.

Vaporisers useful for the invention are for example electrical type vaporizers or shell and tube heat exchanger type vaporizers, which can be operated for high flow of vapor phase product with a liquid phase feed. These vaporizers have two modes of operation : a feed back mode and a feed through mode. The feed back mode is when the vaporizer is used as a pressure builder for the source tank, the feed through mode being when the vaporizer provides gas product to point of use or the head space of the source tank.

The advantages of the invention can be summarized as follows :

- the use of liquid phase delivery from the source reduces size piping needed for the same required pressure drop over long distances for example in case of multiple customer fabs or multiple plants of the same customer compared to the use of a gas delivery system.
- The main energy requirements for vaporization can be accomplished indoors in a more controlled environment with a smaller volume of liquid.
- The main vaporizer is able to supply a gas line comprising a surge tank downstream, together assuring response to a variation of zero flow to maximum flow while maintaining required delivery pressure.

- The use of a back pressure regulation system such as a back pressure regulator, located downstream of the main vaporizer provides liquid delivery from the source container to the main vaporizer while the gas surge tank size can be minimized.
- Ambient temperature and humidity variations and their drawbacks can be offset : the use of a pressure build vaporizer to stabilize the temperature and pressure of the liquid source (NH3) which pressure build vaporizer comprises a liquid delivery from the source (container) to a vaporizer located in the vicinity of said container(s).This vaporizer generates a vapour phase from the liquid phase and said vapour phase is sent back to the head space of the source container.
- According to a different embodiment of the invention the pressure drop in the gaseous phase above the liquid phase in the source container can be avoided by using a pump to deliver the compensation liquid from the liquid source up to the main vaporizer.
- The use of such pressure build up vaporizer allows for pressure build up from the outlet of the vaporizer to the head space of the container and / or for the flow to meet the manufacturers flow rate requirements.
- According to the invention, concerns with impurity build up in the vaporizer or liquid supply thereto are minimal since liquid withdrawal will minimize impurity build up in bulk storage tank, allowing less accumulation of said impurities at the bottom of the container, compared to the situation where gaseous withdrawal is provided.
- However, the vaporizer includes preferably a liquid purge valve located at the bottom of said vaporizer to allow for timed and / or qualitative releases of impurities which may accumulate therein. According to another embodiment of the invention, liquid purge is

done after disconnection and reconnection of the vaporizer for maintenance.

The invention is further disclosed by way of two different embodiments disclosed on the following drawings :

- Fig. 1 is the preferred embodiment of the invention.
- Fig. 2 is an alternate embodiment using a pump to circulate the liquid.

The system and process according to the invention comprises usually three different portions (from upstream to downstream following the flow of NH3) :

a) first portion comprising bulk storage means:

a bulk storage means including preferably pressure build-up means is provided upstream: this bulk storage means may comprise one or a plurality of containers such as iso-containers, having preferably a large capacity for the use envisioned herein (flat display panel manufacture). These containers (usually two) are usually connected in parallel (serial / parallel is possible) and the PLC controller of the system allows to switch from one to the other, when necessary (the switch is usually controlled by level detection, or by using a scale located under the containers which gives an indication of the volume of liquid still contained in the container). Each container is provided with a piping system which may preferably reach the bottom of the container to allow the flow of most or all of the liquid contained therein. This piping system may comprise different valving systems (check valve, close valve, three ways valve to purge the piping anytime it is needed and at least one control valve controlled by the PLC system to open or close the line, which exit is connected to the exit of other control valves of other containers forming a common liquid supply pipe and to the piping system of second portion (liquid supply means) of the system. Also connected to this common liquid supply pipe is a feeding pipe and a control valve to feed the liquid to the pressure build-up circuit comprising a pressure build-up vaporizer to vaporize the liquid withdrawn from the common liquid supply line to generate a vapor phase, which pressure depends on the energy (heat) provided to the vaporizer; this gaseous phase under pressure is then distributed to the headspace of at least one (preferably all of them) of the containers through a control valve (one per container at least) to build and maintain the appropriate pressure (usually between 10^5 and 10^6 Pascal) (150 – 200 psig) in this headspace, in order to push the liquid in the piping system located in the container when the appropriate valves are open, to meet the requirement of liquid needed downstream. This pressure build circuit may be replaced by a pump, e.g. located in the common liquid supply pipe (see fig. 2).

b) <u>second portion or liquid supply means</u> : this second portion comprises at least one liquid supply line to transport the liquid from the first portion a) to the third portion c). In case of several lines, each of them may provide the liquid to a separate third portion c), unless there are specific reasons to provide several supply lines to the same third portion c). Usually this second portion is located outdoor (as usually first portion is), with a temperature which may vary from - 20°C to 80°C. These lines need also to be insulated in such a way that no or substantially no gaseous phase is present in said line portions (purge valves / check valves associated with appropriate abatement, if necessary, may be provided on these lines, if appropriate). This insulation may be provided in addition to the pressure build circuit and / or back pressure regulator means (of third portion) to avoid any substantial gaseous phase of product until reaching the main vaporizer.

c) <u>third portion or vaporizer and gaseous supply means</u> : it is usually located indoor, even though not mandatory. It usually comprises two sub portions (from upstream to downstream) :

- the upstream portion or main vaporizer means receives the liquid phase product from portion b) through different valving system (including purge), said liquid being supplied through a pipe to the bottom (usually) of a main vaporizer having optionally but preferably a liquid purge system to purge liquid and possible residues from the bottom of the vaporizer. The main vaporizer is preferably heat controlled, the vaporized product being recovered at the top (or close to) of the main vaporizer and this gaseous phase is then circulated through a pipe to the point of use of such products. Preferably a vapor surge tank of appropriate size (depending on the pressure requirements) is provided in this pipe with a pressure control (via a pressure sensor) located downstream of this tank. Appropriate valving means controlled by the customer's request at the point of use is also provided, with a connection downstream (close to the point of use) to be able to flow the stream of product, when purging of the lines is done, to an abatement system (wet and / or dry).

Of course, an electronic controller such as a PLC is used to control the opening or the closing of the valves in response to pressure detector signals, temperature detector signals, request of product signals from the customer (e.g. reactor cleaning), alarm signals, empty container signals, purge signals, etc ...

Examples of different embodiments of the invention are now described on the following drawings which represent :

Fig. 1, the preferred embodiment of the invention

Fig 2, another embodiment using a pump;

On fig 1 are represented the different portions and subparts of the first embodiment :

Bulk Storage and Pressure Build

Bulk supply will normally be accomplished with large source containers as shown an fig. 1 and 2 as ISO-containers. These source containers will normally be stored outside and experience the temperature fluctuations of ambient temperatures. To compensate for low outdoor temperatures and withdrawal of liquid phase bulk material a pressure build circuit 10 receives a portion of the liquid phase to a pressure build vaporizer(s) that produces gas phase to return to the head space of the bulk container. This is the pressure build circuit (electrical vaporizer connected to a bulk tank). The head space pressure then can drive the delivery of the liquid phase bulk material to the (indoor) main vaporizer(s), which may be located several kilometres away.

Vacuum / pressure purge can be done using N2 or other appropriate purge gas during disconnection / reconnection of the old / new source container. Purging needs to be done on the liquid and gas connections to the source container.

Outdoor Liquid Supply

The outdoor liquid supply could be experiencing temperature and humidity variations of the outdoor environment for several kilometres to supply several customer fabrication facilities. Vacuum jacketing, or insulation could be used for the tubing to avoid heating or cooling of tubing in high or low temperature extremes.

Indoor Vaporizer

When the liquid supply reaches the inside of the fabrication facility it will enter into a vaporizer(s) to provide the heat exchange to produce the vapor phase product. The heater means to the vaporizer(s) can be left on at all times. The valve upstream of the vaporizer(s) is opened or closed based on the pressure in the surge tank downstream. A back pressure regulator is used immediately downstream of the main vaporizer for dual purposes. Operated at high pressures it helps assure single-phase liquid supply to the vaporizer(s) while also allowing the gas surge tank size to be minimized.

There is also the possibility to provide a liquid purge at the bottom of the vaporizer(s) in the case where impurity levels have become high due to contamination of the liquid feed. This allows for some basic purification of the source material if necessary.

Indoor Vapor Supply

The vapor supply line coming from the vaporizer(s) shown in red could be insulated up to the forward pressure regulator. Heating may or may not be required depending on the gas and flow rate required. A surge tank 30 is provided to compensate for the change in customer flow rate requirements. For the case when the customer is not running and in the next moment requires full design capacity, the surge tank is sized to supply until the main vaporizer 20 comes up to full delivery capacity. For the case where the customer is running full flow then stops production completely the pressure measured with the pressure sensor 31 at the surge tank 30 will rise above the upper authorized pressure limit. The upper authorized pressure limit when reached will trigger closure of the valve 32 upstream of the main vaporizer 20. The surge tank 30 in this case may be sized to receive the gas that will be produced by the vaporization of the maximum remaining liquid in the vaporizer. Preferably the vaporizer used allows flow backward when closed so that there is no need to size the surge tank taking into account this consideration.

On fig. 1, a bulk storage means including pressure build-up means 1 is provided upstream: this bulk storage means comprises two containers 11 and 12 such as iso-containers, having preferably a large capacity for the use envisioned herein (flat display panels). These containers are each located above a scale 13, 14. Each container is filled up with liquid 15, 17 having respectively a gaseous headspace 16, 18 above. The liquid is withdrawn through pipes 19, 21 (alternatively) then flown through valves 22, 23, 24 and pipe 28, respectively through valves 25, 26, 27 and pipe 29 to pipe 50 on the one hand which is part of the outdoor liquid supply 2 and to pipe 40 which feeds the pressure build circuit 10 with liquid 15 or 17. Pipe 40 is connected to valve 41, pipe 42 then the input of the vaporizer of the pressure build circuit 10 which delivers in pipe 43 the gaseous product under pressure so vaporized (the pressure build circuit comprises, e.g. electrical heating means to vaporize the liquid and build a pressurized gaseous phase). This gaseous product is then fed through valve 44, pipe 46 and valve 47 to the gaseous headspace 16 above liquid 15 to build up pressure in container 11, respectively through valve 45 pipe 48, valve 49 to the gaseous headspace 18 above liquid 17 to build up pressure in container 12. The PLC controller of the system (not represented on the drawing) allows to switch from one container 11 to the other 12 or vice versa, when necessary (the switch is usually controlled by level detection, or by using the scales 13, 14 which give an indication of the volume of liquid still contained in each of the containers). The piping system may comprise different valving system (check valve, close valve, three ways valve to purge the piping anytime it is needed with e g high purity nitrogen and at least one control valve controlled by the PLC system to open or close the line.

The second portion or liquid supply means 2 comprises at least one common liquid supply line 50 to transport the liquid from the first portion (bulk storage 1) to the main vaporizer 20.There may be several branches 51, 52 etc... connected to this common line 50 (only one being represented) for feeding a vaporizer 20 (there may be several vaporizers and gaseous supply sections like 3 on fig 1). Usually this second portion 2 is located outdoor (as usually first portion 1 is), with a temperature which may vary from –20°C to 80°C. These lines 50, 51, 52 need to be insulated in such a way that no or substantially no gaseous phase is present in said line portions (purge valves / check valves associated with appropriate abatement, if necessary, may be provided on these lines, if appropriate).

The third portion or vaporizer and gaseous supply means 3 is usually located indoor, even though not mandatory. It usually comprises two sub portions (from upstream to downstream) 4 and 5:

- the upstream portion 4 or main vaporizer means receives the liquid phase product from pipe 52 through different valving system (including purge) 53, 54, 55, 56 , said liquid being supplied through the pipe 57 to the input 61 at the bottom (usually) of the main vaporizer 20 having optionally but preferably a liquid purge system 58 to purge liquid and possible residues from the input 61 at the bottom of the vaporizer 20 . The main vaporizer 20 is heat controlled, the vaporized product being recovered at the top 62 (or close to) of the main vaporizer 20 and this gaseous phase is then circulated through valve 63, 64 and a pipe 66 either directly to the point of use of such products or preferably to a vapor surge tank 30 of appropriate size (depending on the pressure requirements), from which the gaseous product may be delivered to the point of use (VMB) through a valving system 68, 69 (pressure regulator), 70 and pipe 71.

Of course, an electronic controller such as a PLC (represented as 130 on fig 2) is used to control the opening or the closing of the valves in response to pressure detector signals, temperature detector signals, request of product signals from the customer (e.g. reactor cleaning), alarm signals, empty container signals, purge signals, etc sent by detectors like 59 60 65 72 31 or others with no specific references on the drawings. On this drawing a back pressure regulator 64 is used to maintain liquid delivery up to the main vaporizer (in cooperation with other means as explained hereabove).

Figure 2 shows an alternate embodiment accomplishing the same function by using a pump104 instead of the pressure build circuit to deliver the liquid to indoor vaporizers such as 20. On this fig 2 the same devices have the same reference as on fig 1. The pump 104 is fed through the valve 101 and pipe 102 and delivers pressurized liquid in valves 105, 106 to the pipe 57; the PLC 130 is controlling the signals from various sensors, valves or the like such as 132, 131, 134, 135, 136, 133, 140, 141, 142 etc....

In this embodiment the pump is used to pump liquid from the containers (bottom) with no pressure build circuit means provided contrary to the embodiment of fig 1.

CLAIMS

1. A system for high flow delivery of a liquefied product such as liquid ammonia to a flat display panel manufacture location or the like comprising means for bulk storage of liquefied product which includes at least one container to store said product in liquid phase with a gaseous phase of said product located above said liquid product ,liquid supply delivery means for flowing said liquid product from said bulk storage means to main vaporizer means while keeping said liquid product substantially in liquid form throughout said delivery to said main vaporizer means and main vaporizer means to vaporize said liquid product in response to the request of gaseous product made at the location of manufacture;

2. A system according to claim 1, wherein pressure build circuit means is provided as part of the bulk storage means, having one end which receives liquid product and delivers on the other end gaseous product which feeds the space above at least one of the containers storing the liquid product in order to maintain a pressure above said liquid sufficient to supply substantially only liquid product by the related at least one container to the liquid supply delivery means .

3. A system according to one of claims 1 or 2, wherein the exit of said main vaporizer means is connected through a valving system to a vapor surge tank having an appropriate size related to the request of the manufacture location

4. A system according to one of claims 1 to 3, further comprising PLC means to control the flow of liquid and/or gaseous product up to the point of use in the manufacture location.

5. A system according to one of claims 1 to 4 further comprising back pressure regulation means located downstream of the main vaporizer which means contributes to provide liquid delivery from the container to the main vaporizer.

6. A system in accordance with claim 5 comprising a surge tank located downstream the main vaporizer, wherein the back pressure regulation means is located upstream of said surge tank.

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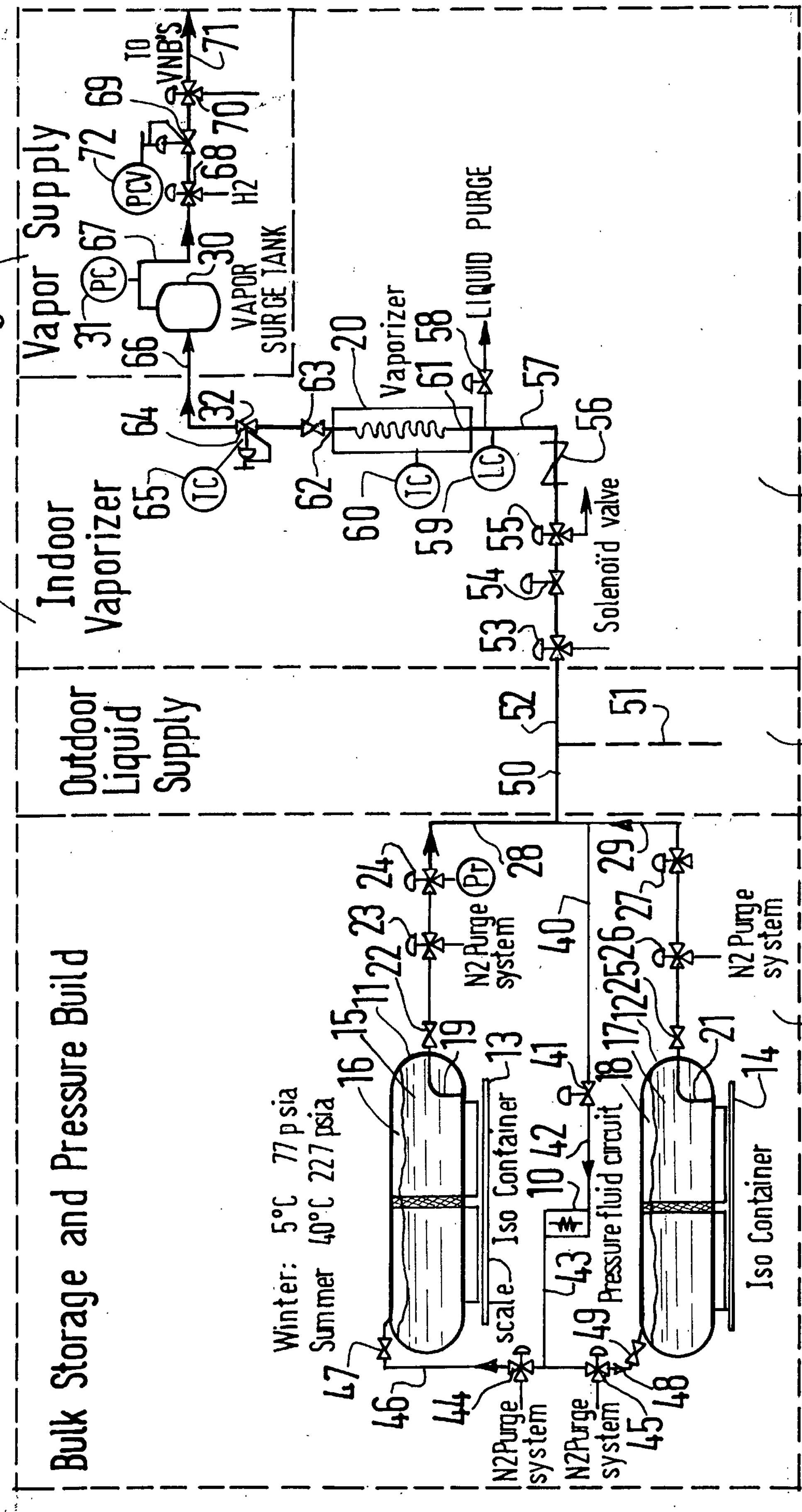
Bulk Storage and Pressure Build
Outdoor Liquid Supply
Indoor Vaporizer
Vapor Supply
Winter: 5°C 77 psia
Summer 40°C 227 psia
Iso Container
Iso Container
scale
Pressure fluid circuit
N2 Purge system
N2 Purge system
N2Purge system
N2Purge system
Pr
Solenoid valve
TC
TC
LC
PC
PCV
Vaporizer
LIQUID PURGE
VAPOR SURGE TANK
H2
TO VNB'S

FIG.1

2/2

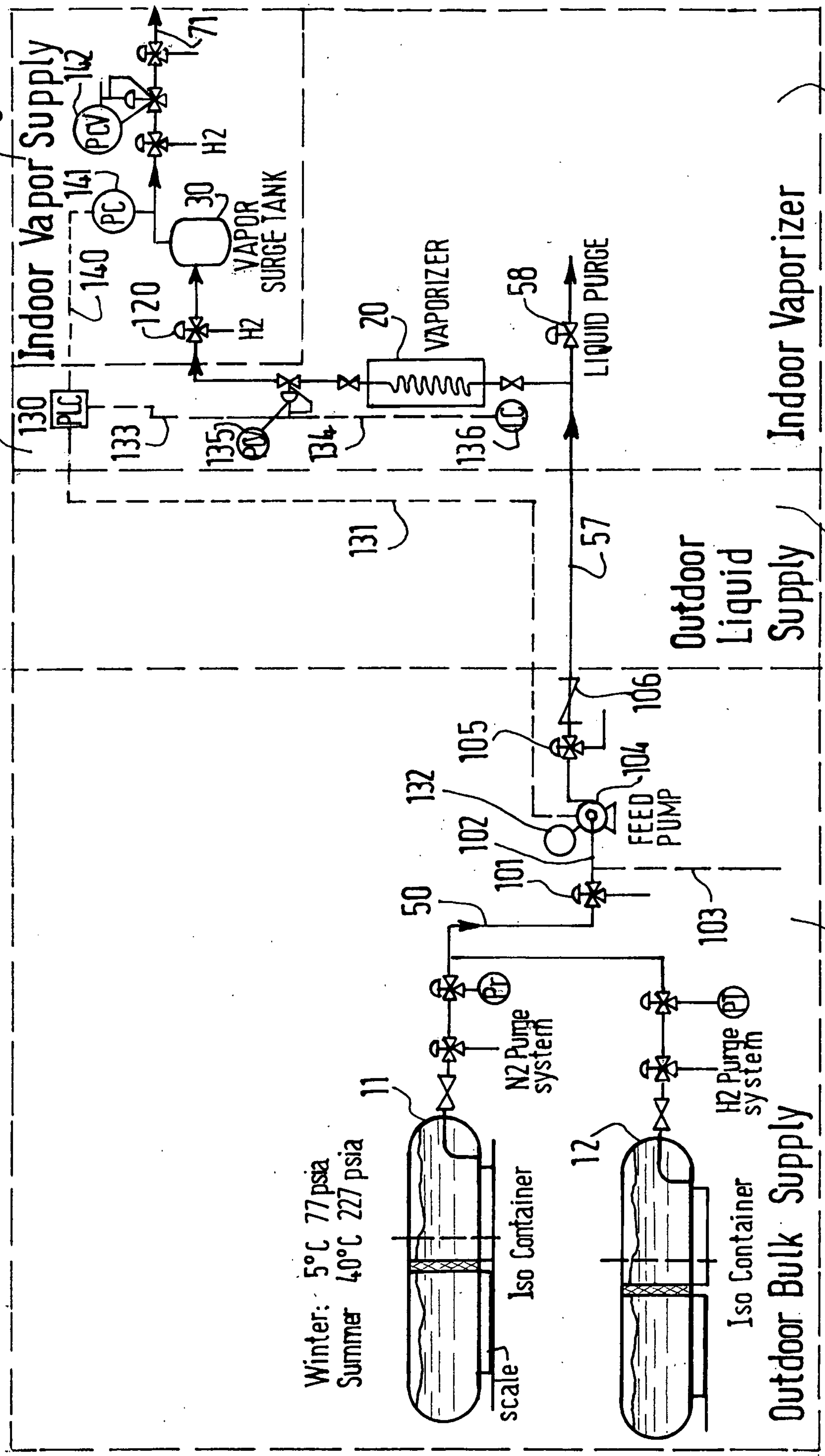
Indoor Vapor Supply
5
4
3
2
1
71
142
PCV
H2
141
PC
140
30
VAPOR SURGE TANK
120
H2
130
PLC
133
135
134
136
20
VAPORIZER
58
LIQUID PURGE
Indoor Vaporizer
131
57
Outdoor Liquid Supply
106
105
104
132
102
FEED PUMP
101
103
50
PT
N2 Purge system
H2 Purge system
11
12
Winter: 5°C 77psia
Summer 40°C 227 psia
Iso Container
scale
Outdoor Bulk Supply

FIG.2

INTERNATIONAL SEARCH REPORT

International Application No
PCT/IB2004/001815

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 F17C7/04 F17C9/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 F17C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
15 December 2004	22/12/2004

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Nicol, B

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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INTERNATIONAL SEARCH REPORT

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