



(12) **EUROPEAN PATENT APPLICATION**

(43) Date of publication:
05.11.2014 Bulletin 2014/45

(51) Int Cl.:
F25J 1/02 (2006.01)

(21) Application number: **14166079.5**

(22) Date of filing: **25.04.2014**

(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

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(30) Priority: **02.05.2013 NL 2010751**

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(54) **Method and system for at least partially liquefying methane containing gas**

(57) The invention relates to a method for at least partially converting methane-containing gas to a liquid state, comprising the steps of:

- compressing in a compressor methane-containing gas which is supplied from a gas source;
- feeding the compressed methane-containing gas from the compressor to a cooling unit, the gas then being cooled such that the methane-containing gas is brought at least partially into a liquid state and a methane-containing mixture is created;
- feeding from the cooling unit to a heat exchanger the cooled methane-containing mixture which is then further cooled in the heat exchanger such that the methane-containing gas remaining in the methane-containing mixture is converted to a liquid state;
- releasing pressure from the methane-containing mixture, thereby creating a fraction of liquid and a fraction of flash-gas; and
- feeding the fraction of liquid to a storage vessel.

The invention further relates to a system herefor.

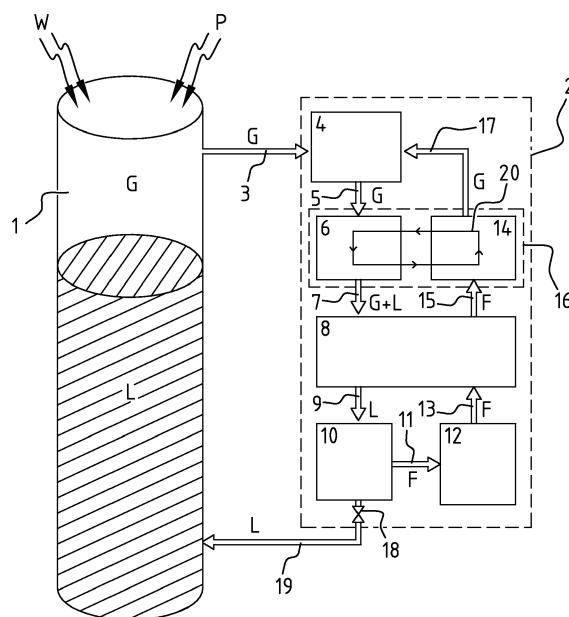


FIG. 1

Description

[0001] The invention relates to a method and a system with which methane-containing gas can be at least partially liquefied.

[0002] An example of methane-containing gas is so-called boil-off gas at LNG storage and filling stations resulting from heat leakage and from pressure release from the truck tanks during filling. These gases accumulate above the LNG liquid in the storage vessel and in this gaseous state are not suitable for filling a vehicle running on LNG. Using the invention this boil-off gas resulting from heat leakage and/or pressure release can be converted back to a liquid state.

[0003] It is known to reliquefy methane-containing gas by cooling it to about -160°C under atmospheric conditions, for instance using a Stirling engine or liquid nitrogen.

[0004] The invention has for its object to provide a method and a system with which methane-containing gas, such as LNG boil-off gas, can be at least partially liquefied in industrial manner and which preferably have advantages in respect of energy.

[0005] This object is achieved according to the invention with the method for at least partially converting methane-containing gas, in particular boil-off gas, to a liquid state, comprising the steps of:

- compressing in a compressor methane-containing gas which is supplied from a gas source;
- feeding the compressed methane-containing gas from the compressor to a cooling unit, the gas then being cooled in the cooling unit such that the methane-containing gas is brought at least partially into a liquid state and a methane-containing liquid gas mixture is created;
- feeding from the cooling unit to a heat exchanger the cooled methane-containing mixture which is then further cooled in the heat exchanger such that the methane-containing gas remaining in the methane-containing mixture is converted to a liquid state;
- releasing pressure from the methane-containing mixture, thereby creating a fraction of liquid and a fraction of flash-gas; and
- feeding the fraction of liquid to a storage vessel.

[0006] It is possible with the method to (re)liquefy a methane-containing gas. In the case the methane-containing gas is a boil-off gas which results for instance due to heat leakage and/or due to pressure release from the fuel tank of a vehicle being fuelled, it can be fed back to the storage vessel after being reliquefied. This results in a closed system wherein emission of gas to the surrounding area is prevented.

[0007] It is on the other hand possible with the method according to the invention to use for instance natural gas as source, whereby LNG can be produced on-site. It is

also possible to upgrade biogas to green gas/bio-natural gas.

[0008] An increased efficiency is achieved by compressing the gas to a pressure around or above the critical pressure of methane in the step of compressing the methane-containing gas with the compressor.

[0009] According to a preferred embodiment, this step comprises of compression to a pressure of around 100 bar.

[0010] Tests have shown that the methane-containing gas is converted in desired quantities to a liquid state when the compressed gas is cooled in the cooling unit to a temperature in the range of -55°C to -60°C.

[0011] According to a further preferred embodiment, in the step of further cooling in the heat exchanger the methane-containing mixture is further cooled to a temperature in the range of -75°C to -80°C. Tests have shown that at these temperatures all the methane-containing gas remaining in the methane-containing mixture is converted to a liquid state.

[0012] According to a very advantageous preferred embodiment, the energy efficiency of the system is increased significantly by feeding the fraction of flash-gas back to the heat exchanger, where it functions as coolant for cooling the methane-containing mixture in the heat exchanger.

[0013] A further increase in efficiency of the system is achieved when the cooling unit comprises a heat exchanger, wherein a coolant circuit with a coolant flows through a further heat exchanger in which the coolant is cooled. This pre-cooling increases the efficiency of the coolant circuit.

[0014] According to a further preferred embodiment, the flash-gas is fed from the heat exchanger for cooling the methane-containing mixture to a plate exchanger in which the flash-gas is heated and in which the coolant of the coolant circuit is supercooled.

[0015] According to yet another preferred embodiment, the flash-gas is heated in the plate exchanger to a temperature above 0°C, after which the flash-gas is fed via a conduit from the cooling unit and to the compressor. This recirculation of the flash-gas provides for an exact mass balance and increases production capacity. Heating to a temperature above 0°C moreover causes the recirculation to take place under conditions which are favourable for the lifespan of the compressor.

[0016] According to yet another preferred embodiment, the method further comprises the steps of:

- feeding the methane-containing liquid from the heat exchanger to an intermediate storage;
- controlling the flash pressure using a pressure regulator;
- having the methane-containing liquid flash in the intermediate storage, wherein a fraction of liquid and a fraction of flash-gas is created;
- feeding the fraction of liquid to a storage vessel; and
- supplying the flash-gas via the pressure regulator to

the heat exchanger, this gas functioning there as coolant for cooling the methane-containing mixture. The pressure regulator controls the pressure, whereby the intermediate storage is in optimal condition during flashing so as to maximize the fraction of liquid. Flashing is understood to mean that the liquid undergoes a pressure reduction, wherein a gas fraction is created by the reduction of the enthalpy.

[0017] According to yet another preferred embodiment, during release of pressure from the methane-containing mixture, the pressure thereof is reduced to a pressure level which is 1 to 2 bar lower than the pressure in the storage vessel.

[0018] According to yet another preferred embodiment, the flash-gas fed back from the intermediate storage via the pressure regulator to the second heat exchanger has a temperature in the range of about -135°C to -120°C. Tests have shown that the desired conversion of liquid is guaranteed at this temperature.

[0019] According to yet another preferred embodiment, the flash-gas is heated in the second heat exchanger to a temperature in the range of -45°C to -75°C, more particularly about -50°C.

[0020] According to yet another preferred embodiment, the method further comprises the following steps of:

- monitoring the liquid level in the intermediate storage;
- closing the flash-gas pressure regulator when a threshold value of the liquid level is exceeded, whereby the pressure temporarily rises to a value of 1 to 2 barg above the pressure in the storage vessel; and
- opening a drain valve at the bottom of the intermediate storage, whereby the produced liquid is returned to the storage vessel at a temperature colder than that in the storage vessel.

[0021] According to yet another preferred embodiment, the storage vessel is also the gas source and the methane-containing gas is a boil-off gas. In such a configuration a recirculation takes place and the boil-off gas, resulting for instance due to heat leakage and/or due to pressure release from the fuel tank of a vehicle being fuelled, is relieved. Once in liquid form, it is fed back to the storage vessel, thereby creating a closed system wherein emission of gas to the surrounding area is prevented.

[0022] According to yet another preferred embodiment, the gas source is a (bio-)natural gas conduit and the methane-containing gas is a (bio-)natural gas. Using natural gas as source enables production of LNG from natural gas on-site. It is also possible to upgrade biogas to green gas/bio-natural gas.

[0023] According to yet another preferred embodiment, the flash-gas is discharged from the cooling unit

to a CNG compressor. Dutch 'Slochteren' gas comprises for instance about 14.3% nitrogen. When such low-calorific (bio-)natural gas is guided through the system, LNG is on the one hand produced and a fraction of flash-gas is on the other created in which a nitrogen residue is present. This flash-gas is hereby unsuitable for recirculation because it entails the danger of separation. In the case of separation the upper layer can become heavier, whereby the centre of gravity is raised and a storage vessel can roll over. This flash-gas is preferably discharged to a CNG compressor. LNG and CNG filling stations can in this way be combined in simple manner.

[0024] The invention further relates to a system for at least partially converting methane-containing gas, in particular boil-off gas, to a liquid state, comprising a conversion unit, comprising:

- a gas source with methane-containing gas;
- a compressor in gas connection with the gas source and configured to receive methane-containing gas in gaseous form from the gas source, wherein the compressor is configured to compress the received methane-containing gas to a pressure around or above the critical pressure of methane;
- a cooling unit in fluid connection with the compressor and configured to receive and cool the methane-containing gas from the compressor, whereby the methane-containing gas is brought at least partially into a liquid state and a methane-containing mixture is created; and
- a heat exchanger in fluid connection with the cooling unit and configured to receive and further cool the methane-containing mixture from the cooling unit such that the remaining methane-containing gas in the methane-containing mixture is converted to a liquid state;
- means for releasing pressure from the liquid methane-containing mixture, thereby creating a fraction of liquid and a fraction of flash-gas; and
- a liquid connection between the conversion unit and storage vessel for storage of methane-containing gas in liquid state, such as LNG.

[0025] According to a preferred embodiment of the system, the fraction of flash-gas is fed back to the heat exchanger and functions there as coolant for cooling the methane-containing mixture in the heat exchanger.

[0026] According to a further preferred embodiment of the system, the cooling unit comprises a heat exchanger and a coolant circuit flowing with a coolant through a further heat exchanger in which the coolant is cooled.

[0027] According to yet another preferred embodiment, the system further comprises:

- an intermediate storage arranged in fluid connection with the heat exchanger; and
- a pressure regulator configured to control the flash pressure and in fluid connection with the heat ex-

changer such that a fraction of flash-gas, created during flashing of the methane-containing liquid mixture in the intermediate storage, is feedable via the pressure regulator to the heat exchanger so that it can be utilized in the heat exchanger as cooling medium for cooling the methane-containing mixture.

[0028] The invention further relates to a system configured for application of the method as described in this application.

[0029] The operation of the present invention is now elucidated with reference to the figures, in which:

Figure 1 shows a system according to the invention wherein LNG boil-off gas is at least partially liquefied; and

Figure 2 shows a system according to the invention, wherein methane-containing (natural) gas is at least partially liquefied.

[0030] Boil-off gas G is a methane-containing gas which results in LNG storage and at filling stations from heat leakage W and/or by pressure release P from the vehicle fuel tanks to storage vessel 1 during refuelling. These gases G accumulate above the LNG liquid L in storage vessel 1. In the discussed embodiments this is at a pressure of between 6 and 12 barg, although other embodiments with higher pressures of up to for instance 24 barg can also be envisaged.

[0031] The invention comprises a conversion unit 2 configured to convert methane-containing gas at least partially to a liquid state. It can for instance thus convert boil-off gas G back at least partially to a liquid state so that it can be returned as LNG liquid L to storage vessel 1.

[0032] Because conversion unit 2 applies a flash pressure higher than the atmospheric pressure, a cooling to a temperature in the range of about -135°C to -120°C will suffice. The exact temperature depends on the pressure in storage vessel 1. Because this temperature is less low than the temperature of about -160° required according to the prior art, the system and method according to the invention are an energetic improvement. The energetic advantages are:

- a less low temperature owing to the increased condensation pressure;
- a forced cooling to -60°C is sufficient to reach a final temperature of -120°C to -135°C; and
- simple switching on and off of the system, wherein there is no energy consumption at all in switched-off mode.

[0033] The operation of conversion unit 2 according to the invention will now be elucidated with reference to figure 1. Because the operation is elucidated in figure 1 for an LNG system, conversion unit 2 will also be referred to below as 'Zero Boil-off unit' 2 in relation to figure 1.

[0034] Zero Boil-off unit 2 will compress the boil-off

gases G in a compressor 4 to a pressure around or above the critical pressure of methane, after which the gas G is cooled in a heat exchanger 6 to -55°C to -60°C and thereby becomes partially liquid.

[0035] The mixture of gas G and liquid L then flows through a second heat exchanger 8 in which the gas G, in counterflow with the flash-gas F, is further cooled to a temperature of -75°C to -80°C and completely liquefied.

[0036] First heat exchanger 6 has a coolant as cooling medium, while second heat exchanger 8 has gas, in particular flash-gas F, as cooling medium.

[0037] The pressure on the liquid is then released to a pressure 1 to 2 bar lower than the pressure in storage vessel 1. In this step the liquid flashes in an intermediate storage 10, which results in 45% by mass to 55% by mass of flash-gas F, the remnant remaining liquid L with a temperature lower than the saturation conditions of storage vessel 1. The gas created in this step is referred to as flash-gas F and flows out of intermediate storage 10 via a pressure regulator 12, which controls the flash-gas pressure, and back to second heat exchanger 8.

[0038] The flash-gas F fed back from intermediate storage 10 via pressure regulator 12 to second heat exchanger 8 has a temperature in the range of about -135°C to -120°C and is heated in second heat exchanger 8 to about -50°C.

[0039] It is noted that a decrease to a pressure about 1 to 2 bar below the pressure in storage vessel 1 will give the liquid L a temperature about 1°C to 2°C lower than the temperature of the liquid LNG L in storage vessel 1. This is advantageous because it brings about an additional cooling of the LNG present in storage vessel 1. The production of boil-off gas will hereby decrease.

[0040] It is noted that intermediate storage 10, pressure regulator 12 and drain valve 18 increase the controllability and energy efficiency, although they are not essential for the operation of the system. If immediately after cooling in second heat exchanger 8 pressure is released from the liquid without conditions optimal for the purpose being provided by pressure regulator 12, the liquid still flashes, be it in less favourable ratios. The flash-gas F can then serve as cooling medium for second heat exchanger 8 and the methane-containing gas converted to liquid L can be carried via a conduit 19 into storage vessel 1.

[0041] Once the flash-gas F has functioned as cooling medium for second heat exchanger 8, the flash-gas F flows further via a plate exchanger 14 and is heated to a temperature above 0°C, after which the flash-gas F is recirculated in a particularly advantageous embodiment to the inlet of Zero boil-off unit 2 and fed to compressor 4.

[0042] Flowing on the secondary side in plate exchanger 14 where the flash-gas F is heated is coolant, for instance of the type R404A, which is used for cooling to -55°C/-60°C in first heat exchanger 6. This coolant is supercooled in this plate exchanger 14 and provides for an improvement of up to 6.5% of the energy efficiency of cooling unit 16.

[0043] It is noted that, while coolant type R404A is particularly suitable for this application in energetic terms, it is also possible for another type of coolant to be applied, such as type R410A.

[0044] In an advantageous embodiment cooling unit 16 is an assembly comprising first heat exchanger 6 and plate exchanger 14, as well as a coolant circuit 20 running through these two exchangers 6, 14. Cooling unit 16 is optimized for the integration of the energy flows: heat exchanger 14 ensures that the flash energy is recovered and transferred to coolant circuit 20, whereby less energy need be supplied overall for the same quantity of cooling energy.

[0045] In intermediate storage 10 the LNG level is monitored, and if a threshold value is exceeded the flash-gas pressure regulator 12 is closed, whereby the pressure rises temporarily to a value of 1 to 2 barg above the pressure in storage vessel 1. At that moment a drain valve 18 is opened at the bottom of intermediate storage vessel 10, whereby the produced liquid L is fed back to storage vessel 1 at a temperature colder than that in storage vessel 1.

[0046] The pressure in storage vessel 1 can vary between for instance 6 and 9 barg or less, and the above stated threshold value is preferably a fixed value below the pressure prevailing in storage vessel 1.

[0047] It is noted that application of intermediate storage 10 is particularly advantageous, though not essential per se. If an intermediate storage 10 were however not present, the liquid L would have to be drained off directly from second heat exchanger 8 to storage vessel 1, which would have the drawback that the pressure of storage vessel 1 will then also equalize with that in the heat exchanger. The system would hereby become less energy-efficient.

[0048] Typical mass flows received by compressor 4 for the system shown in figure 1 are 12 kg/hour inflow of boil-off gas G from storage vessel 1 and about 12-14 kg/hour recirculated flash-gas F. Flowing therefore from compressor 4 to first heat exchanger 6 of cooling unit 16 is a mass flow of about 24-26 kg/hour of gas G, and about 12 kg/hour of liquefied boil-off gas can be fed back as liquid LNG from intermediate storage 10 to storage vessel 1.

[0049] The liquid LNG fed back from intermediate storage 10 to storage vessel 1 has, depending on the pressure, a temperature in the range of about -135°C to -120°C. The flash-gas F fed back from intermediate storage 10 via pressure regulator 12 to second heat exchanger 8 also has a temperature in the range of about -135°C to -120°C.

[0050] Because compressor 4 increases the pressure to a pressure higher than the critical pressure of methane, a pressure results which is also higher than atmospheric pressure, whereby the cooling temperatures need be less low than in prior art cooling methods. The system and the method according to the invention are hereby more energy-efficient than the above stated alternative

systems. The energetic advantages are:

- a less low temperature owing to the increased condensation pressure;
- a forced cooling to -60°C is sufficient to reach a final temperature of -120 to -135°C; and
- simple switching on and off of the system, wherein there is no energy consumption at all in switched-off mode.

[0051] Figure 2 shows an embodiment of the system suitable for producing LNG from natural gas on-site. It is further possible to upgrade biogas to green gas/bio-natural gas. Because the embodiment of figure 2 is highly similar to the embodiment of figure 1 in terms of operation and construction, only the differences are further elucidated.

[0052] The greatest difference is that compressor 4 also has a gas feed 22 from a gas mains supply. Gas supplied from a gas mains supply can comprise nitrogen: the Netherlands 'Slochteren' gas consists for instance of about 14.3% nitrogen. A nitrogen residue will hereby also be present in the flash-gas F. This flash-gas F is hereby unsuitable for recirculation because it entails the danger of separation. The flash-gas F is therefore preferably fed through via a conduit 24 to a CNG compressor 26. An LNG and CNG filling station can in this way be combined in simple manner.

[0053] The above described embodiment is intended only to illustrate the present invention and not to limit the scope of the invention in any way. The rights described are defined by the following claims, within the scope of which many modifications can be envisaged.

Claims

1. Method for at least partially converting methane-containing gas, in particular boil-off gas, to a liquid state, comprising the steps of:

- compressing in a compressor (4) methane-containing gas which is supplied from a gas source (1, 22);
- feeding the compressed methane-containing gas from the compressor (4) to a cooling unit (16), the gas then being cooled in the cooling unit (16) such that the methane-containing gas is brought at least partially into a liquid state and a methane-containing mixture is created;
- feeding from the cooling unit (16) to a heat exchanger (8) the cooled methane-containing mixture which is then further cooled in the heat exchanger (8) such that the methane-containing gas remaining in the methane-containing mixture is converted to a liquid state;
- releasing pressure from the methane-containing mixture, thereby creating a fraction of liquid

- (L) and a fraction of flash-gas (F); and
 - feeding the fraction of liquid (L) to a storage vessel (1).
2. Method as claimed in claim 1, wherein the step of compressing the methane-containing gas with the compressor (4) comprises of compression to a pressure around or above the critical pressure of methane and/or compression to a pressure around 100 bar.
 3. Method as claimed in any of the foregoing claims, wherein in the step of cooling in the cooling unit (16) the compressed gas is cooled to a temperature in the range of - 55°C to -60°C.
 4. Method as claimed in any of the foregoing claims, wherein in the step of further cooling in the heat exchanger (8) the methane-containing mixture is cooled to a temperature in the range of -75°C to -80°C.
 5. Method as claimed in any of the foregoing claims, wherein the fraction of flash-gas (F) is fed back to the heat exchanger (8) and functions as coolant for cooling the methane-containing mixture in the heat exchanger (8).
 6. Method as claimed in any of the foregoing claims, the cooling unit (16) comprising a heat exchanger (6) and a coolant circuit (20) with a coolant which flows through a further heat exchanger (14) in which the coolant is cooled.
 7. Method as claimed in claim 6, wherein the flash-gas (F) is fed from the heat exchanger (8) for cooling the methane-containing mixture to a plate exchanger (14) in which the flash-gas (F) is heated and in which the coolant of the coolant circuit (20) is supercooled.
 8. Method as claimed in claim 7, wherein the flash-gas (F) is heated in the plate exchanger to a temperature above 0°C, after which the flash-gas (F) is fed via a conduit (17) from the cooling unit (17) to the compressor (4).
 9. Method as claimed in any of the foregoing claims, further comprising the steps of:
 - feeding the methane-containing liquid from the heat exchanger (8) to an intermediate storage (10);
 - controlling the flash pressure using a pressure regulator (12);
 - having the methane-containing liquid flash in the intermediate storage (10), wherein a fraction of liquid (L) and a fraction of flash-gas (F) is created;
 - feeding the fraction of liquid (L) to a storage vessel (1); and
 - supplying the flash-gas (F) via the pressure regulator (12) to the heat exchanger (8), the gas functioning there as coolant for cooling the methane-containing mixture.
 10. Method as claimed in claim 9, wherein during release of pressure from the methane-containing mixture, the pressure thereof is reduced to a pressure level which is 1 to 2 bar lower than the pressure in the storage vessel (1);
 - wherein the flash-gas (F) fed back from the intermediate storage (10) via the pressure regulator (12) to the second heat exchanger (8) preferably has a temperature in the range of about -135°C to -120°C; and
 - wherein the flash-gas (F) is preferably heated in the second heat exchanger (8) to a temperature in the range of - 45°C to -75°C, more particularly about -50°C.
 11. Method as claimed in any of the claims 9 and 10, further comprising the following steps of:
 - monitoring the liquid level in the intermediate storage (10);
 - closing the flash-gas pressure regulator (10) when a threshold value of the liquid level is exceeded, whereby the pressure temporarily rises to a value of 1 to 2 barg above the pressure in the storage vessel (1); and
 - opening a drain valve (18) at the bottom of the intermediate storage (10), whereby the produced liquid L is returned to the storage vessel (1) at a temperature colder than that in the storage vessel (1).
 12. Method as claimed in any of the foregoing claims, wherein the storage vessel (1) is also the gas source and the methane-containing gas is a boil-off gas;
 - and/or wherein the gas source is a (bio-)natural gas conduit and the methane-containing gas is a (bio-)natural gas; and
 - wherein the flash-gas (F) is preferably discharged from the cooling unit (16) to a CNG compressor (26).
 13. System for at least partially converting methane-containing gas, in particular boil-off gas, to a liquid state, comprising a conversion unit (2), comprising:
 - a gas source with methane-containing gas;
 - a compressor (4) in gas connection (3) with the gas source (1, 22) and configured to receive methane-containing gas in gaseous form from

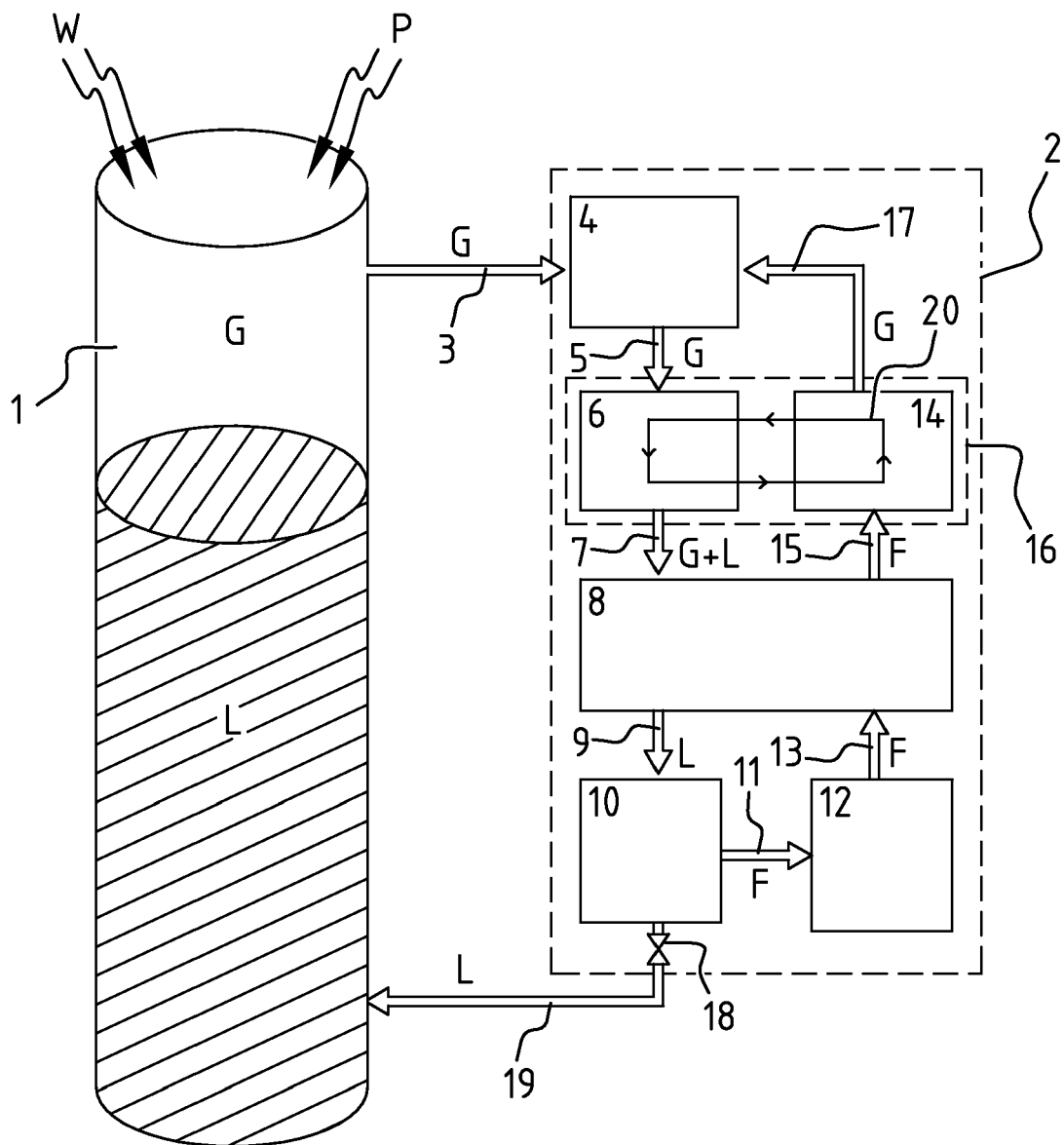
the gas source, wherein the compressor (4) is configured to compress the received methane-containing gas to a pressure around or above the critical pressure of methane;

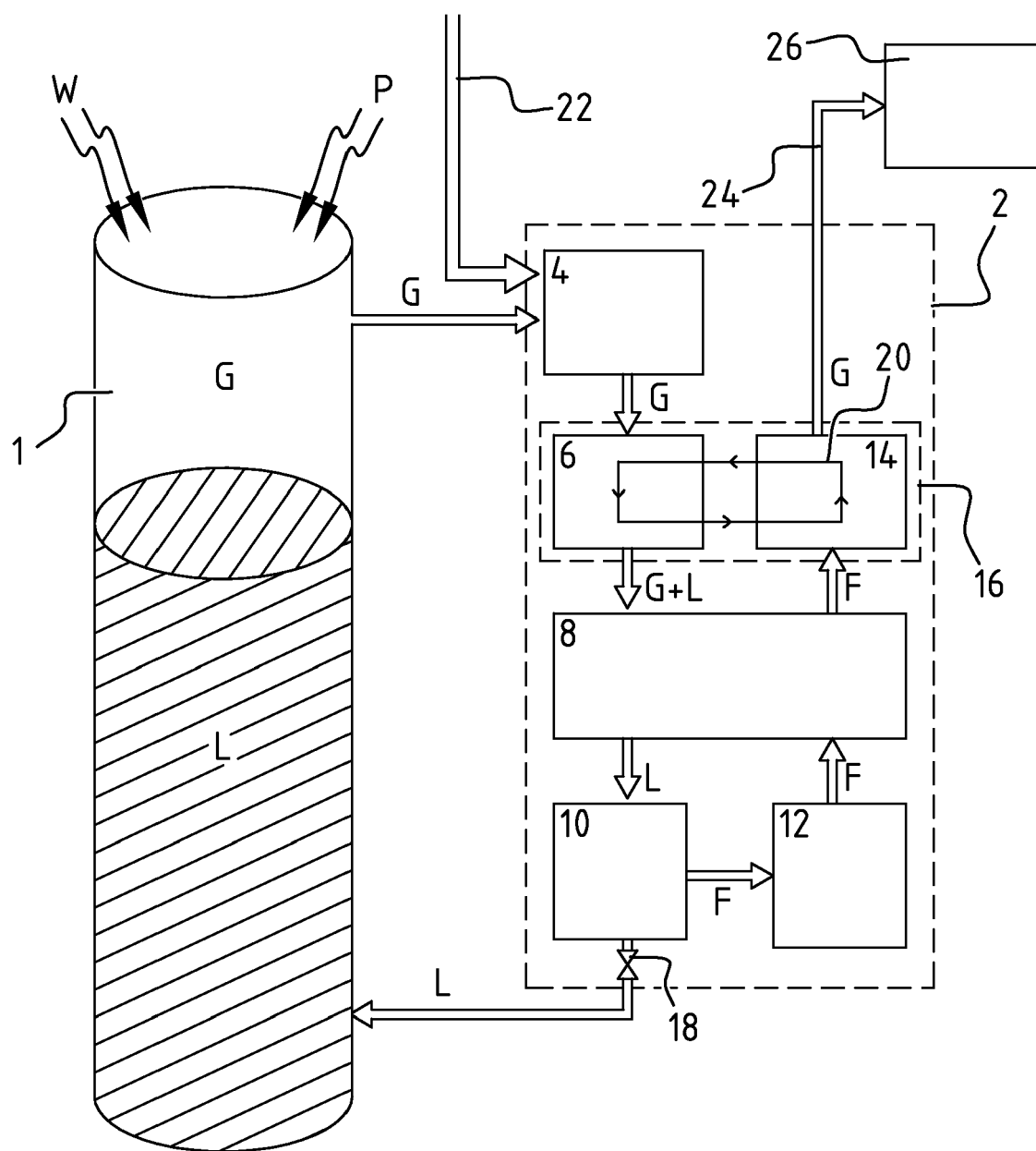
- a cooling unit (16) in fluid connection (5) with the compressor (4) and configured to receive and cool the methane-containing gas from the compressor, whereby the methane-containing gas is brought at least partially into a liquid state and a methane-containing mixture is created; and
- a heat exchanger (8) in fluid connection (7) with the cooling unit (16) and configured to receive and further cool the methane-containing mixture from the cooling unit such that the remaining methane-containing gas in the methane-containing mixture is converted to a liquid state;
- means for releasing pressure from the liquid methane-containing mixture, thereby creating a fraction of liquid (L) and a fraction of flash-gas (F); and
- a liquid connection (19) between the conversion unit (2) and storage vessel (1) for storage of methane-containing gas in liquid state, such as LNG.

14. System as claimed in claim 13, wherein the fraction of flash-gas (F) is fed back to the heat exchanger (8) and functions as coolant for cooling the methane-containing mixture in the heat exchanger (8).

15. System as claimed in claim 13 or 14, the cooling unit (16) comprising a heat exchanger (6) and a coolant circuit (20) flowing with a coolant through a further heat exchanger (14) in which the coolant is cooled;

- and/or further comprising:
- an intermediate storage (10) arranged in fluid connection (9) with the heat exchanger (8); and
- a pressure regulator (12) configured to control the flash pressure and in fluid connection with the heat exchanger (8) such that a fraction of flash-gas (F), created during flashing of the methane-containing liquid mixture in the intermediate storage (10), is feedable via the pressure regulator to the heat exchanger (8) so that it can be utilized in the heat exchanger (8) as cooling medium for cooling the methane-containing mixture.

FIG. 1

FIG. 2