

METHOD FOR DIFFUSION OF A TRACER GAS AND METHOD FOR TESTING THE LEAKTIGHTNESS OF A MEMBRANE

Technical field

5 The invention relates to the field of leaktight and thermally insulating membrane tanks for the storage and/or transportation of a fluid, such as a cryogenic fluid.

 The invention more particularly relates to a method for testing the leaktightness of a leaktight membrane of a tank using a tracer gas and to a method
10 for diffusion of a tracer gas

Technological background

 The document KR1020100050128 discloses a method for testing the leaktightness of a membrane of a leaktight and thermally insulating tank for the storage of LNG. The tank comprises a multilayer structure and successively exhibits,
15 from the outside towards the inside, a secondary insulating space, a secondary leaktightness membrane, a primary insulating space and a primary leaktightness membrane intended to be in contact with the liquefied natural gas present in the tank. The method is more particularly targeted at detecting leaks through the welding beads which make it possible to connect, in leaktight fashion, the metal sheets of the primary
20 leaktightness membrane. The method provides for the injection of a tracer gas into the primary insulating space and then for the movement of a detection apparatus equipped with a tracer gas analyser, inside the tank, along the welding beads of the primary leaktightness membrane. Thus, if the detection apparatus detects the presence of the tracer gas, it can be concluded that there exists a defect of
25 leaktightness of the primary leaktightness membrane. In such a method, the injection of the tracer gas into the primary insulating space is critical, since the detection method can only guarantee reliable results if the tracer gas has diffused homogeneously into the whole of the primary insulating space.

Summary

An idea at the basis of the invention is to provide a method for the diffusion of tracer gas which makes it possible to obtain a reliable diffusion of the tracer gas throughout the thermally insulating space.

5 According to one embodiment, the invention provides a method for injection of a tracer gas into a thermally insulating space of a leaktight and thermally insulating tank, the leaktight and thermally insulating tank exhibiting a polyhedral shape defined by a plurality of tank walls which are attached to a carrier structure, each of the tank walls comprising a thermally insulating barrier comprising insulating elements which
10 are anchored to the carrier structure and a membrane resting against the insulating elements of the thermally insulating barrier, the thermally insulating barriers of each tank wall communicating with the thermally insulating barrier of the other tank walls and together defining the thermally insulating space; the said plurality of tank walls including a bottom wall, the said injection method comprising injecting a tracer gas
15 into the thermally insulating space by means of at least one device for injection of tracer gas which passes through the membrane of the bottom wall and which emerges in the thermally insulating barrier of the bottom wall.

Advantageously, prior to the stage of injection of the tracer gas by means of at least one device for injection of tracer gas, the membrane is detached from the
20 thermally insulating barrier in the region of the device for injection of the tracer gas by introducing a lifting tool through a hole made in the membrane and by positioning said lifting tool so as to be interposed between the membrane and the thermally insulating barrier of the bottom wall.

An advantageous feature of the invention is that the tracer gas is introduced
25 and transported from the inner space of the vessel to said thermally insulating space.

The term "inner space of the vessel" means the functional area of the vessel, i.e., its storage space for a fluid, such as a cryogenic fluid, as opposed to the outer, or external, area of the vessel.

Classically, the tracer gas is transported and introduced into the thermally
30 insulating space by or via the external, or outer, zone of the tank, which presents a certain number of disadvantages - including those presented above - when one seeks in particular to carry out a leak test of the aforementioned membrane, in order to

ensure in particular that the fluid, contained in the tank, is not in danger of entering the thermally insulating space.

As the tracer gas exhibits a lower vapour density than that of air, it rises in the thermally insulating space. Consequently, the injection of the tracer gas via the bottom, through the membrane of the bottom wall, makes it possible to ensure a diffusion of the tracer gas in the thermally insulating space which is faster and more homogeneous than when the tracer gas is injected through a pipe passing through a dome made in the ceiling wall of the tank, as practised in the prior art.

According to embodiments, such a method for injection of a tracer gas can comprise one or more of the following characteristics.

According to one embodiment, during the implementation of the injection method, each of the walls comprises, from the outside towards the inside of the tank, a secondary thermally insulating barrier resting against the carrier structure, a secondary membrane resting against the secondary thermally insulating barrier, and optionally a primary thermally insulating barrier resting against the secondary membrane and a primary membrane which rests against the primary thermally insulating barrier and which is intended to be in contact with the liquefied gas present in the tank.

According to one embodiment, the secondary thermally insulating barriers of the tank walls are in communication with one another and together define a secondary thermally insulating space, and a tracer gas is injected into the secondary thermally insulating space by means of at least one device for injection of tracer gas which passes through the secondary membrane of the bottom wall and which emerges in the secondary thermally insulating barrier of the bottom wall.

According to another embodiment, the primary thermally insulating barriers of the tank walls are in communication with one another and together define a primary thermally insulating space, and a tracer gas is injected into the primary thermally insulating space by means of at least one device for injection of tracer gas which passes through the primary membrane of the bottom wall and which emerges in the primary thermally insulating barrier of the bottom wall.

According to one embodiment, the tracer gas is injected exclusively by means of devices for the injection of the tracer gas which pass through the membrane

of the bottom wall and which emerge in the thermally insulating barrier of the bottom wall.

According to one embodiment, the thermally insulating space is placed under negative pressure before the stage of injecting the tracer gas into the thermally
5 insulating space. This makes possible, first, a better diffusion of the tracer gas and makes it possible, secondly, to more quickly achieve a satisfactory level of concentration of the tracer gas.

According to one embodiment, the thermally insulating space is placed under negative pressure down to a set negative pressure of between -40 kPa and -80 kPa,
10 for example of the order of -60 kPa.

According to one embodiment, the tracer gas is injected into the thermally insulating space until the pressure inside the said thermally insulating space is slightly greater than atmospheric pressure, for example between 1 and 6 kPa relative and advantageously of the order of 2 kPa relative. This makes it possible, on the one hand,
15 to place the thermally insulating space under excess pressure, in order to promote the migration of the tracer gas through the defective welds of the membrane so as to increase the effectiveness of the test of leaktightness of the membrane, and, on the other hand, to remain within an excess pressure range which is lower than the maximum excess pressures capable of being accepted by the membrane in order to
20 prevent the latter from being torn.

According to one embodiment, the tracer gas exhibits a lower vapour density than that of air.

According to one embodiment, the tracer gas is chosen from helium, ammonia and a mixture of nitrogen and molecular hydrogen.

25 According to one embodiment, the tracer gas is injected in the form of a mixture additionally comprising air and/or nitrogen.

According to one embodiment, the bottom wall exhibits a rectangular shape exhibiting two axes of symmetry, the said two axes of symmetry delimiting four regions of the bottom wall, and the tracer gas is injected by means of at least four devices for
30 injection of tracer gas which each pass through the membrane of the bottom wall and which emerge in the thermally insulating barrier of the bottom wall, each of the four

regions of the bottom wall being equipped with one of the four devices for injection of tracer gas.

According to one embodiment, each of the four injection devices passes through the leaktightness membrane close to the centre of the respective region of the bottom wall.

According to one embodiment, each device for injection of tracer gas comprises a coupling member which is welded in leaktight fashion to the membrane of the bottom wall at the periphery of a hole which is made in the said membrane, the coupling member being combined with a pipe connected to a source of tracer gas.

According to one embodiment, the coupling member comprises a tubular coupling which is combined with the pipe connected to the source of tracer gas and a metal ring which is, on the one hand, welded in leaktight fashion to the membrane of the bottom wall, at the periphery of the hole which is made in the said membrane, and, on the other hand, welded in leaktight fashion to the tubular coupling.

According to one embodiment, prior to the stage of injection of the tracer gas by means of at least one injection device, the membrane is detached from the thermally insulating barrier in the region of the device for injection of the tracer gas by introducing a lifting tool through the coupling member and the hole made in the membrane and by positioning the said lifting tool so that a hook of the said lifting tool is inserted between the membrane and the thermally insulating barrier of the bottom wall.

According to one embodiment, the lifting tool is held in a position in which the hook is inserted between the membrane and the thermally insulating barrier of the bottom wall during the stage of injection of the tracer gas by means of at least one injection device.

According to one embodiment, each injection device additionally comprises at least one injection pipe which is connected in leaktight fashion to the hole which is made in the said membrane, the said injection pipe extending inside a corrugation of the membrane and comprising at least one drill hole at each intersection between the corrugation in which the said injection pipe extends and a perpendicular corrugation.

Another idea at the basis of the invention is to provide a method for testing the leaktightness of a membrane which is reliable and rapid to carry out.

According to one embodiment, the invention provides a method for testing the leaktightness of a membrane of a leaktight and thermally insulating tank, the leaktight and thermally insulating tank exhibiting a polyhedral shape defined by a plurality of tank walls which are attached to a carrier structure, each of the tank walls

5 comprising a thermally insulating barrier comprising insulating elements which are anchored to the carrier structure and a membrane resting against the insulating elements of the thermally insulating barrier, the thermally insulating barriers of the tank walls communicating with one another and together defining the thermally insulating space; the method for testing the leaktightness comprising:

- 10 - injecting the tracer gas into the thermally insulating space;
- providing a leak detection device comprising a detection hood, configured in order to define, with the membrane, a leaktight detection chamber, and a vacuum pump which is, on the one hand, coupled to the detection chamber and, on the other hand, to an analytical apparatus;
- 15 - positioning the detection hood against a face of the membrane opposite the thermally insulating space, the detection hood being positioned so that the detection chamber is positioned facing a portion of a welding bead to be tested;
- placing the detection chamber under negative pressure using the vacuum pump;
- 20 - conveying the gas phase present in the detection chamber towards the analytical apparatus; and
- analysing, using the analytical apparatus, the said gas phase and delivering a variable representative of a concentration of tracer gas in the said gas phase.

According to one embodiment, the invention provides a leak detection device

25 for testing the leaktightness of a membrane of a leaktight and thermally insulating tank, the leak detection device comprising a detection hood, configured in order to define, with the membrane, a leaktight detection chamber, and a vacuum pump which is, on the one hand, connected to the detection chamber and, on the other hand, to an analytical apparatus.

30 According to embodiments, the method for testing the leaktightness of a membrane and/or the leak detection device can comprise one or more of the following characteristics.

According to one embodiment, the injection of the tracer gas into the thermally insulating space is carried out using the abovementioned method for injection of a tracer gas. However, according to another embodiment, the injection of the tracer gas into the thermally insulating space can also be carried out according to
5 another method.

According to one embodiment, the analytical apparatus is a mass spectrometer.

According to one embodiment, the detection hood exhibits an elongated shape.

10 According to one embodiment, the detection hood comprises a central core and a leaktightness seal which are attached to one another and are arranged in order to define, with the membrane to be tested, the detection chamber.

According to one embodiment, the leaktightness seal comprises a casing attached to the central core and a peripheral leaktightness lip which extends the
15 casing.

According to one embodiment, the casing of the leaktightness seal exhibits a bottom which covers the upper surface of the central core and a peripheral wall which matches the periphery of the central core.

According to one embodiment, the peripheral leaktightness lip is bent
20 towards the outside of the detection hood and is configured in order to be flexed and to be flattened against the membrane when the detection chamber is placed under negative pressure.

According to one embodiment, the portion of the welding bead is traversed by at least one corrugation of the membrane.

25 According to one embodiment, the peripheral leaktightness lip is shaped in order to fit the geometry of the said at least one corrugation.

According to one embodiment, the portion of the welding bead is traversed by at least two corrugations, for example three corrugations, which are parallel to the membrane, and the peripheral leaktightness lip is shaped in order to fit the geometry
30 of the said corrugations.

According to one embodiment, the peripheral leaktightness lip comprises at least two indentations exhibiting a shape corresponding to that of a corrugation of the membrane projecting towards the inside of the tank, the said indentations being intended to straddle the said corrugation.

- 5 According to one embodiment, the at least one corrugation of the membrane projects towards the inside of the tank, the detection hood being positioned against the membrane so that the indentations straddle the corrugation.

 According to one embodiment, the peripheral leaktightness lip comprises at least two regions in projection exhibiting a shape corresponding to that of a
10 corrugation of the membrane projecting towards the outside of the tank.

 According to one embodiment, the detection hood is positioned against the membrane so that the regions in projection are inserted into the corrugation.

 According to one embodiment, the leaktightness seal is made of an elastomer material exhibiting a Shore A hardness of between 20 and 50.

- 15 According to one embodiment, the elastomer material of the leaktightness seal is chosen from polyurethane elastomer, silicone, nitrile and Viton®.

 According to one embodiment, the detection hood is equipped with a clamping system capable of flattening the peripheral leaktightness lip against the membrane to be tested.

- 20 According to one embodiment, prior to placing the detection chamber under negative pressure, the clamping system is actuated so as to guarantee the leaktightness of the detection chamber.

 According to one embodiment, when the leaktightness seal comprises at least two indentations exhibiting a shape corresponding to that of a corrugation of the
25 membrane projecting towards the outside of the tank and traversing the portion of the welding bead, the clamping system comprises a clamp at each of the indentations, each clamp comprising two branches respectively positioned on either side of the indentation and configured in order to apply an effort of clamping the peripheral leaktightness lip against the corrugation of the membrane.

- 30 According to one embodiment, the detection chamber is placed under negative pressure down to an absolute pressure value of between 50 and 1000 Pa, for example of the order of 100 Pa absolute.

According to one embodiment, the gas phase is analysed for a time of greater than or equal to 5 seconds.

According to one embodiment, the variable representative of a concentration of tracer gas in the said gas phase is compared with a threshold, and it is determined
 5 that the leaktightness of the portion of the welding bead is defective when the variable representative of a concentration of tracer gas in the said gas phase is greater than the said threshold.

According to one embodiment, the leak detection device additionally comprises a homogenization chamber which is positioned between the detection
 10 chamber and the analytical apparatus, the detection hood comprising a gas inlet equipped with a valve, the homogenization chamber and the gas inlet being coupled to two opposite ends of the detection chamber.

According to one embodiment, the valve of the gas inlet is closed during the placing of the detection chamber under negative pressure, the stage of conveying the
 15 gas phase present in the detection chamber comprising:

- opening the valve of the gas inlet and conveying the gas phase present in the detection chamber towards the homogenization chamber; and
- conveying the gas phase from the homogenization chamber towards the analytical apparatus.

20 According to one embodiment, the invention provides a method for monitoring the diffusion of the tracer gas into the thermally insulating space.

According to one embodiment, the method for monitoring the diffusion of the tracer gas is carried out during the abovementioned method for testing the leaktightness. However, in another embodiment, it can also be carried out
 25 independently.

According to one embodiment, the monitoring of the diffusion of the tracer gas into the thermally insulating space comprises:

- sampling the gas present in the thermally insulating space using a plurality of sampling devices each traversing the membrane of one of the tank walls; and
- 30 - conveying the sampled gas towards an analytical apparatus, such as a mass spectrometer, for example.

According to one embodiment, the sampling devices are advantageously positioned close to corner regions formed at the junction between two or three tank walls.

5 According to one embodiment, each gas sampling device comprises a cover plate which is combined with a membrane using a mastic bead surrounding a region of the membrane devoid of welding at the junction between two elements of the membrane, the cover plate exhibiting a hole and a coupling intended to be coupled to a pipe connected to the analytical apparatus, the said coupling being welded in leaktight fashion at the periphery of the hole of the cover plate.

10 According to one embodiment, the gas sampling device comprises a coupling member which exhibits a joining piece intended to be coupled in leaktight fashion to a pipe connected to the analytical equipment, a shoulder and a threaded lower rod which projects from the shoulder, passes through the secondary membrane and is screwed into a threaded boring made in a plate attached to the thermally
15 insulating barrier, the gas sampling device additionally comprising an annular leaktightness seal which is threaded onto the lower rod and which is sandwiched between the secondary membrane and the shoulder, the coupling member exhibiting a drill hole which emerges, on the one hand, at the joining piece and, on the other hand, in the thermally insulating space.

20 **Brief description of the figures**

A better understanding of the invention will be obtained and other aims, details, characteristics and advantages of the latter will become more clearly apparent during the following description of several specific embodiments of the invention, given solely by way of illustration and without limitation, with reference to the
25 appended drawings.

- **Figure 1** is a diagrammatic illustration of a multilayer structure of a wall of a membrane tank.

- **Figure 2** is a partial diagrammatic view of a leaktight and thermally insulating tank illustrating devices for injection of tracer gas which are positioned
30 through a membrane of the bottom wall of the tank.

- **Figure 3** is a detailed perspective view of a hole made through the membrane of a bottom wall of the tank.

- **Figure 4** is a detailed perspective view of a device for injection of tracer gas according to a first embodiment.

- **Figure 5** is a sectional view of the device for injection of tracer gas of Figure 4 and of a lifting tool introduced through the injection device so as to detach
5 the membrane from the thermally insulating layer.

- **Figure 6** is a perspective view of a device for injection of tracer gas according to one embodiment.

- **Figure 7** is a diagrammatic perspective view of a tank illustrating the arrangement of devices for injection of tracer gas according to a second embodiment.

10 - **Figure 8** is a detailed top view illustrating an opening, a tubular coupling and a multiroute distribution member of a device for injection of tracer gas according to the second embodiment.

- **Figure 9** is a sectional view illustrating an opening and a tubular coupling of an injection device according to the second embodiment.

15 - **Figure 10** is a partial view of an injection pipe of a device for injection of tracer gas according to the second embodiment.

- **Figure 11** is a sectional view of the injection pipe of Figure 10 in a region of intersection between two corrugations of the membrane.

20 - **Figure 12** is a top view of a region devoid of welding at the junction between two sheets of a membrane which is intended to receive a gas sampling device according to a first embodiment.

- **Figure 13** is a top view of a gas sampling device intended to be positioned in the region devoid of welding illustrated in Figure 12.

25 - **Figure 14** is a sectional view of a gas sampling device according to a second embodiment.

- **Figure 15** is a diagrammatic view of a leak detection device according to a first embodiment.

- **Figure 16** is a cross-sectional view of the detection hood of the leak detection device of Figure 15.

- **Figure 17** is a perspective view of a leaktightness seal according to a first embodiment.

- **Figure 18** is a diagrammatic view of an alternative form of a leak detection device in which the detection hood is equipped with a clamping system.

5 - **Figure 19** is a perspective view of a leaktightness seal according to a second embodiment.

- **Figure 20** diagrammatically illustrates the positioning of the detection hood facing a portion of a welding bead ensuring the leaktightness between two adjacent corrugated metal sheets of a membrane.

10 - **Figure 21** is a diagrammatic view of a leak detection device according to a second embodiment.

Detailed description of embodiments

The method for testing leaktightness which will be described subsequently is more particularly targeted at testing the leaktightness of a membrane of a leaktight
15 and thermally insulating membrane tank. By way of example, such membrane tanks are described in particular in Patent Applications WO14057221 and FR2691520, which respectively relate to the Mark V and Mark III technologies.

Membrane tanks exhibit a plurality of walls which exhibit a multilayer structure, such as represented in Figure 1. Each wall 1 comprises, from the outside
20 towards the inside of the tank, a secondary thermally insulating barrier 2 comprising secondary insulating panels 3 anchored to a carrier structure 4, a secondary membrane 5 resting against the secondary thermally insulating barrier 2, a primary thermally insulating barrier 6 comprising primary insulating panels 7 resting against the secondary membrane 2 and anchored to the carrier structure 4 or to the secondary
25 insulating panels 3 and a primary membrane 8 which rests against the primary thermally insulating barrier 6 and which is intended to be in contact with the liquefied gas present in the tank.

The tank exhibits a polyhedral general shape. According to the embodiment illustrated in Figure 2, the tank exhibits a front wall 9 and a rear wall, not represented,
30 which are here of octagonal form. The tank also comprises a ceiling wall 10, a bottom wall 11 and side walls 11, 12, 13, 14, 15, 16, 17 which extend along the longitudinal direction of the tank between the front wall 9 and the rear wall.

The secondary thermally insulating barriers 2 of the tank walls are in communication with one another so as to form, between the carrier structure 4 and the secondary membrane 5, a secondary thermally insulating space, which is leaktight. Likewise, the primary thermally insulating barriers 6 of the tank walls are in communication with one another so as to form, between the secondary membrane 5 and the primary membrane 8, a primary thermally insulating space, which is leaktight.

At least one of the primary membrane 8 and secondary membrane 5 comprises a plurality of metal sheets which are welded to one another. The leaktightness test method which will be described subsequently is more particularly targeted at testing the leaktightness of the welds which make it possible to couple the metal sheets to one another. According to one embodiment, the membrane to be tested exhibits corrugations which allow it to be bent out of shape under the effects of the thermal and mechanical stresses generated by the fluid stored in the tank. In order to do this, as represented, for example, in Figure 20, each metal sheet comprises two series of corrugations perpendicular to one another.

The leaktightness test method comprises three stages which will each be described in detail subsequently, namely:

- the diffusion of a tracer gas into a thermally insulating space covered by the membrane 5, 8, the leaktightness of which it is desired to test;
- the monitoring of the diffusion of the tracer gas into the thermally insulating space; and
- the checking of the leaktightness of the welds of the membrane 5, 8.

Diffusion of a tracer gas

The tracer gas is injected into the thermally insulating space which is covered by the membrane 5, 8, the leaktightness of which it is desired to check. When it is desired to check the leaktightness of the secondary membrane 5, the tracer gas is injected into the secondary thermally insulating space. In this case, the leaktightness test method is carried out before the primary thermally insulating barrier 7 and the primary membrane 8 are installed. When it is desired to check the leaktightness of the primary membrane 8, the tracer gas is injected into the primary thermally insulating space.

The tracer gas is, for example, chosen from helium, ammonia and a mixture of nitrogen and molecular hydrogen. According to one embodiment, the tracer gas is injected in the form of a mixture additionally comprising air and/or nitrogen.

Advantageously, in order to facilitate the diffusion of the tracer gas into the thermally insulating space, the latter is placed beforehand under negative pressure until a set negative pressure of between -40 kPa and -80 kPa, for example of the order of -60 kPa, is reached.

In order to do this, the thermally insulating space is connected to a vacuum pump, not represented, capable of sucking in the gas present in the thermally insulating space and of expelling it outside the said thermally insulating space. In addition, the thermally insulating space is equipped with a or with one or more pressure sensors capable of delivering a signal representative of the pressure prevailing in the thermally insulating space and which make it possible to ensure control of the vacuum pump.

Subsequently, when the pressure prevailing in the thermally insulating space has reached the set negative pressure, the tracer gas is injected into the thermally insulating space until the pressure inside the said thermally insulating space is slightly greater than atmospheric pressure, for example between 1 and 6 kPa relative and advantageously of the order of 2 kPa relative.

Figure 2 diagrammatically illustrates a leaktight and thermally insulating tank and also a system for injection of the tracer gas into a thermally insulating space.

The injection system comprises a plurality of pipes 18 which are, on the one hand, connected to a source of tracer gas, not illustrated, and, on the other hand, connected to devices for injection of the tracer gas 19 making a passage for injection of the tracer gas through the membrane 5, 8, the leaktightness of which has to be tested. More particularly, the devices for injection of the tracer gas 19 make passages for tracer gas through the membrane of the bottom wall 11. Such an arrangement is particularly advantageous as the tracer gas exhibits a lower vapour density than that of air, so that there is a tendency to rise in the thermally insulating space. Consequently, the injection of the tracer gas via the bottom, through the membrane 5, 8 to be tested of the bottom wall 11, makes it possible to ensure a rapid and homogeneous diffusion of the tracer gas into the thermally insulating space.

In the embodiment represented in Figure 2, the bottom wall 11 is equipped with at least four devices for injection of the tracer gas 19 which are evenly distributed over the surface of the bottom wall 11. The bottom wall 11 exhibits a rectangular shape and can thus be divided into four regions of equal surface area by its two axes of symmetry x and y. Each of the four devices for injection of the tracer gas 19 is positioned in one of the four abovementioned regions. In the specific embodiment illustrated, each device for injection of the tracer gas 19 is positioned close to the centre of its respective region. In a specific embodiment, each of the four devices for injection of the tracer gas is positioned at a distance $\frac{1}{4} L$ from the adjacent longitudinal edge and at a distance $\frac{1}{4} B$ from the adjacent transverse edge, with L the longitudinal dimension of the bottom wall 11 and B the transverse dimension of the bottom wall 11.

Figures 3 to 5 illustrate in detail a device for injection of the tracer gas 19 through a corrugated primary membrane, of Mark III or Mark V type, for example. The corrugations of the primary membrane 8 in this instance project in the direction of the inside of the tank. Each device for injection of the tracer gas 19 exhibits a hole 20, illustrated in Figure 3, made through the primary membrane 8. The hole 20 is made in a flat region 21 of the primary membrane, delimited by two corrugations extending along the longitudinal direction of the bottom wall 11 and by two corrugations extending along the transverse direction of the bottom wall 11. The orifice is advantageously placed at the centre of the said flat region 21.

As represented in Figure 4, a coupling member 22 is welded in leaktight fashion to the primary membrane 8 at the periphery of the hole 20. The coupling member 22 comprises a tubular coupling 23 intended to be coupled to a pipe 18 connected to a source of tracer gas. In order to do this, the tubular coupling 23 comprises a threaded end intended to receive a complementary coupling associated with a pipe 18 connected to the source of tracer gas. The tubular coupling 23 is welded in leaktight fashion to a metal ring 24 which is itself welded in leaktight fashion to the primary membrane 8 all around the hole 20, so as to ensure the leaktightness of the device for injection of tracer gas 19.

According to one embodiment, as represented in Figure 5, a lifting tool 25 is used in order to detach the primary membrane 8 from the primary insulating panel 7 on which it rests, in the region for injection of the tracer gas. This facilitates the diffusion of the tracer gas into the primary thermally insulating space. The lifting tool

25 exhibits an L general shape and comprises a handle 26 and a hook 27 which is positioned at the end of the handle 26 and extends perpendicularly to the said handle 26. The lifting tool 25 is introduced through the coupling member 22 and is positioned so that the hook 27 is inserted between the primary membrane 8 and the surface of
 5 the primary insulating panel 7 supporting the said primary membrane 8. This makes it possible to detach the said primary membrane 8 in order to promote the diffusion of the tracer gas.

The lifting tool 25 is put in place before the pipe 18 connected to the source of tracer gas is coupled to the coupling member 22. In addition, advantageously, the
 10 lifting tool is held in position when the coupling member 22 is coupled to a pipe 18 connected to the source of tracer gas and when tracer gas is injected into the primary thermally insulating space through the device for injection of the tracer gas 19.

Figure 6 represents a device for injection of tracer gas 19 made through a corrugated secondary membrane 5, of Mark V type, for example. This corrugated
 15 secondary membrane 5 differs in particular from the primary membrane 8 represented in Figures 3 to 5 in that the corrugations project towards the outside of the tank, whereas they projected in the direction of the inside of the tank in Figures 3 to 5. As in the preceding embodiment, each device for injection of the tracer gas 19 is positioned in a flat region 21 of the membrane delimited by two corrugations extending
 20 along the longitudinal direction of the bottom wall 11 and by two corrugations extending along the transverse direction of the bottom wall 11. Furthermore, each device for injection of the tracer gas 19 exhibits a structure identical to that represented and described in connection with Figures 3 to 5. In addition, a lifting tool 25 can also be used in order to detach the secondary membrane 5 from the secondary
 25 insulating panel 3 on which it rests, in the region for injection of the tracer gas.

Figures 7 to 11 illustrate devices for injection of the tracer gas 28 according to another embodiment. Although exhibiting a more complex structure than those represented and described in connection with Figures 2 to 5, these devices for injection of the tracer gas are advantageous in that they facilitate the diffusion of the
 30 tracer gas and are thus particularly suitable for the injection of a tracer gas exhibiting a high viscosity, such as ammonia, for example.

In this embodiment, each device for injection of the tracer gas 28 exhibits a hole 29, represented in Figures 7 and 8, made in the primary membrane 8. In the

embodiment represented, the hole 29 is made through a metal plate 30 which is attached to a primary insulating panel 7 of the primary thermally insulating barrier 6 and to which corrugated metal sheets of the primary membrane 8 are welded in leaktight fashion. As represented in Figure 9, a coupling member 31 intended to be
 5 coupled to a pipe 18 connected to a source of tracer gas is welded in leaktight fashion to the periphery of the hole 29. Furthermore, the hole 29 is coupled in leaktight fashion to a multiroute distribution member 32 which is positioned in a housing made in the primary insulating panel 7 and which is capable of conveying the tracer gas from the opening 29 towards one or more injection pipes 33. By way of example, in Figure 9,
 10 the multiroute distribution member 32 is coupled to two aligned injection pipes 33.

As represented in Figures 10 and 11, each injection pipe 33 is attached to one or more primary insulating panels 7 of the primary thermally insulating barrier 6 and extends inside one of the corrugations of the primary membrane 8 projecting in the direction of the inside of the tank. Each injection pipe 33 comprises one or two
 15 drill holes 34 at each intersection between, on the one hand, the corrugation in which said injection pipe 33 is housed and, on the other hand, a corrugation perpendicular to this corrugation. The drill holes 34 are oriented in the direction of a corrugation perpendicular to the corrugation in which the injection pipe 33 is housed and thus make it possible to convey the tracer gas towards the series of corrugations
 20 perpendicular to the corrugation traversed. Thus, the tracer gas is diffused into the primary thermally insulating space through the networks of corrugations of the primary membrane 8.

In Figure 7, the arrangement of the injection pipes 33 in the primary thermally insulating barrier of the bottom wall 11 according to one embodiment is observed. The
 25 arrows represent the direction of injection of the tracer gas through the drill holes 34 made in the injection pipes. One or more injection pipes 33 are positioned along each of the edges of the bottom wall 11 and comprise, at each intersection between two corrugations, at least one drill hole directed towards the adjacent tank wall. These injection pipes 33 thus make it possible to cause the tracer gas to diffuse from the
 30 bottom wall 11 in the direction of the other tank walls.

Furthermore, in the embodiment represented, the thermally insulating barrier of the bottom wall 11 comprises one or two series of injection pipes 33 which are each positioned in a corrugation of the primary membrane of the bottom wall 11 which is oriented along the longitudinal direction of the tank and is positioned in a median

region of the bottom wall 11. The abovementioned injection pipes 33 comprise two drill holes 34 oriented along two opposite directions, at each intersection between the corrugation in which the said injection pipe 33 is housed and a corrugation perpendicular to this corrugation.

5 Monitoring of the diffusion of the tracer gas

Advantageously, when the tracer gas has diffused through the thermally insulating space, the diffusion of the tracer gas into the thermally insulating space is monitored.

10 In order to do this, the gas present in the thermally insulating space into which the tracer gas has been injected is sampled by means of a plurality of gas sampling devices installed through the membrane covering the said thermally insulating space. Each sampling device is connected to an analytical apparatus, such as a mass spectrometer, which makes it possible to check the presence and the concentration of the tracer gas in the corresponding region of the thermally insulating space.

15 Advantageously, at least one gas sampling device is positioned on each of the tank walls 9, 10, 11, 12, 13, 14, 15, 16. The gas sampling devices are advantageously positioned close to the corner regions between the tank walls as these regions are the most difficult for the tracer gas to access due to the specific structures of the thermally insulating barriers in these regions.

20 In connection with Figures 12 and 13, a gas sampling device is described which makes it possible to sample the gas in the primary thermally insulating space through a primary membrane 8.

25 As represented in Figure 12, the primary membrane 8 exhibits, at the junction between two adjacent metal sheets 38, 39, a region devoid of welding 40. The gas sampling device 35 comprises a cover plate 41, represented in Figure 13, which is combined with the primary membrane 8 using a leaktight mastic bead 42. The mastic bead 42 surrounds the region devoid of welding 40. The cover plate 41 exhibits a hole 43 and supports a tubular coupling 44 which is welded in leaktight fashion at the periphery of the hole 43. The tubular coupling 44 is intended to be coupled to a pipe
30 connected to the analytical apparatus.

In connection with Figures 14 and 15, a gas sampling device 45 is described which makes it possible to sample the gas in the secondary thermally insulating space

through a secondary membrane 5. Such a gas sampling device 45 is positioned in a region of the secondary membrane 5 intended to be traversed by an anchoring member which passes through the secondary membrane 5 and ensures the attaching of the primary insulating panels 7 of the primary thermally insulating barrier 6 to the secondary thermally insulating barrier 2. The sampling device 45 comprises a coupling member 46 which exhibits a joining piece 47 intended to be coupled in leaktight fashion to a pipe connected to the analytical apparatus. The coupling member also exhibits a threaded lower rod 48 which is screwed into a threaded boring 49 intended to subsequently receive, during the installation of the primary thermally insulating barrier 6, an anchoring member of a primary insulating panel 7 of the primary thermally insulating barrier 6. The threaded boring 49 is, for example, made in a metal plate 50 which is attached to a secondary insulating panel 3. Furthermore, the coupling member 45 exhibits an annular leaktightness seal 52 which is threaded onto the lower rod 48 and a shoulder 51 which is configured in order to flatten the annular leaktightness seal 52 against the lower face of the secondary membrane 5 so as to ensure the leaktightness of the gas sampling device 45. The coupling member 46 also exhibits a drill hole 53 which emerges, on the one hand, at the joining piece 47 and, on the other hand, in the secondary thermally insulating space.

When the concentration of tracer gas reaches a minimum concentration threshold for each sampling region, the stage of checking the leaktightness of the welds can then be carried out.

In addition, according to one embodiment, the concentration of tracer gas in the sampling regions is regularly monitored during the checking of the leaktightness of the welds in order to determine if the concentration of tracer gas falls below the minimum concentration threshold. In such a case, tracer gas can again be injected into the thermally insulating space in order for the concentration of tracer gas to again become greater than the said minimum concentration threshold.

Checking of the leaktightness of the welds

In order to confirm the leaktightness of the welding beads of a membrane 5, 8, a leak detection device 54, as represented in Figure 15, is used.

The leak detection device 54 comprises a detection hood 55 which is intended to be positioned against the internal face of the membrane 5, 8, facing a portion of the welding bead to be tested.

The detection hood 55 exhibits an elongated shape and exhibits a length of between 0.5 and 3 m, for example of the order of 1 m.

As represented in Figure 16, the detection hood 55 comprises a rigid central core 59 and a flexible leaktightness seal 60 which are attached to one another and
 5 which are arranged in order to define, with the membrane 5, 8 to be tested, a leaktight detection chamber 61 positioned facing the portion of the welding bead 62 to be tested.

Returning to Figure 15, it is observed that the leak detection device 54 also comprises an analytical apparatus 56 which is coupled to the detection chamber 61
 10 and makes it possible to detect the tracer gas. If the analytical apparatus 56 detects tracer gas in an amount greater than a threshold, it may be concluded that there exists a leaktightness defect of the portion of the welding bead 62 tested. According to one embodiment, the analytical apparatus 56 is a mass spectrometer.

The leak detection device 54 also comprises a vacuum pump 57 which is
 15 combined with the said analytical apparatus 56. The vacuum pump 57 is coupled, on the one hand, to the detection chamber of the detection hood 55, so as to make it possible to produce a negative pressure in the detection chamber, and, on the other hand, to the analytical apparatus 56, so as to convey the gas present in the detection chamber 61 towards the analytical apparatus 56.

20 The vacuum pump 57 is connected to the detection hood 55 via a pipe 58 which is preferably flexible. The pipe 58 is coupled to a channel which is made in the central core 59 and emerges in the detection chamber 61.

As represented in Figures 16 and 17, the leaktightness seal 60 comprises a casing 63 which matches the shape of the central core 59 and a peripheral
 25 leaktightness lip 64 which extends the casing 63 downwards. The casing exhibits a bottom which covers the upper surface of the central core 59 and a peripheral wall 74 which matches the periphery of the central core 59. The bottom exhibits at least one hole, not represented, to which the pipe 58, connected to the vacuum pump 57, is coupled in leaktight fashion.

30 The peripheral leaktightness lip 64 is bent towards the outside of the detection hood 55 and is thus configured in order to be flexed and to be flattened against the membrane 5, 8 when the leaktight chamber 61 is placed under negative

pressure. In other words, the peripheral leaktightness lip 64 exhibits a section exhibiting an L general shape.

The portion bent towards the outside of the peripheral leaktightness lip 64 exhibits a width of the order of 15 to 40 mm. The peripheral leaktightness lip 64 is shaped in order to fit the geometry of the membrane 5, 8 along the welding bead to be tested. Consequently, in Figure 17, the peripheral leaktightness lip 64 comprises indentations 65 exhibiting a shape corresponding to that of the corrugations of the membrane 5, 8 which the detection hood 55 is intended to straddle when it is in position against the portion of the welding bead 62 to be tested.

10 The leaktightness seal 60 is advantageously made of an elastomer material exhibiting a Shore A hardness of between 20 and 50. The leaktightness seal is, for example, made of polyurethane elastomer, of silicone, of nitrile or of Viton®.

In an embodiment illustrated diagrammatically in Figure 18, the detection hood 55 is additionally equipped with a clamping system 66 capable of flattening the peripheral leaktightness lip 64 against the membrane 8 to be tested, so as to guarantee the leaktightness of the detection chamber 61. The clamping system 66 in this instance comprises a clamp 67 at each of the indentations 65 of the peripheral leaktightness lip 64. Each clamp 67 comprises two branches respectively positioned on either side of the indentation 65 and configured in order to apply an effort of clamping the peripheral leaktightness lip 64 against the membrane 8. Advantageously, the branches are configured in order to clamp the peripheral leaktightness lip 64 against the leaktightness membrane, close to the base of the corrugation.

Furthermore, in the embodiment represented, the clamping system 67 additionally comprises, at each of the longitudinal ends of the detection hood 55, a movable finger 68 which is configured in order to flatten one of the longitudinal ends of the peripheral leaktightness lip 64 against the membrane 8.

Figure 19 illustrates a leaktightness seal 60 according to an alternative embodiment. This leaktightness seal 60 is shaped in order to fit a membrane 5 in which the corrugations project towards the outside of the tank. Such a membrane is, for example, a secondary membrane 5 according to the Mark V technology, as illustrated in Figure 6. Consequently, the peripheral leaktightness lip 64 comprises

regions in projection 69 intended to be inserted inside the corrugations of the membrane 5.

The procedure for detecting a leaktightness defect of a welding bead is as follows.

5 In a first step, the detection hood 55 is positioned facing the portion of the welding bead 62 to be tested, as represented in Figure 20.

It is advisable to make sure that the detection hood 55 is suitably centred with respect to the welding bead 62 so that the two lateral parts of the bent portion of the peripheral leaktightness lip 64 are positioned on either side of the welding bead
10 62.

The vacuum pump 57 is then started up in order to place the detection chamber 61 under negative pressure and to promote the migration of the tracer gas through the defective regions of the welding bead 62.

As soon as the pressure inside the detection chamber 61 passes below a
15 pressure threshold P_t , a stream of gas is conveyed from the detection chamber 61 towards the analytical apparatus 56 and a rate of leakage ϕ of tracer gas is measured for a minimum time T_m . The rate of leakage ϕ is then compared with a threshold ϕ_t .

If the rate of leakage ϕ is less than the threshold ϕ_t , then it is concluded that the tested portion of the welding bead 62 does not exhibit a leaktightness defect. In
20 this case, the detection hood 55 is then positioned facing an adjacent portion of the welding bead 62, an overlap being provided between the two portions successively tested so as to guarantee that the leaktightness of the welding bead 62 has been tested over the entire length of the said welding bead 62.

On the other hand, if the rate of leakage ϕ is greater than or equal to the
25 threshold ϕ_t , then it is concluded that the tested portion of the welding bead 62 exhibits a leaktightness defect. Corrective welding measures are then carried out in order to correct the defect.

By way of example, for a concentration of helium in the thermally insulating space of the order of 20%, the pressure threshold below which the rate of leakage is
30 measured is between 50 and 1000 Pa absolute, for example of the order of 100 Pa absolute, the minimum duration of measurement of the rate of leakage is 5 seconds and the threshold ϕ_t is of the order of $1.0 \times 10^{-6} \text{ Pa.m}^3.\text{s}^{-1}$.

Figure 21 represents a leak detection device 54 according to another embodiment. This embodiment differs from the embodiment described above in that it additionally comprises a homogenization chamber 70 which is positioned between the detection chamber 61 and the analytical apparatus 56 and in that the detection
 5 hood 55 comprises a gas inlet 71.

The gas inlet 71 is equipped with a valve which makes it possible to establish or to interrupt a flow of ambient air towards the detection chamber 61. The homogenization chamber 70 is coupled to one end of the detection chamber 61, while the gas inlet 71 is coupled to the opposite end of the detection chamber 61.

10 The mode of operation of the leak detection device is as follows.

When the detection hood 55 is positioned facing the portion of the welding bead 62 to be tested, the valve of the gas inlet 71 is closed and the vacuum pump 57 is started up in order to place the detection chamber 61 under negative pressure. As soon as the pressure inside the detection chamber 61 passes below a pressure
 15 threshold P_t , the valve of the gas inlet 71 is opened and all of the gas present beforehand in the leaktight chamber is transferred towards the homogenization chamber 70. The homogenization chamber 70 exhibits a greater volume than that of the detection chamber 61 and comprises, for example, a piston system which makes it possible to precisely suck up all of the gas present in the detection chamber 61.

20 The gas present in the homogenization chamber 71 is subsequently transferred in the direction of the analytical apparatus 56 in order to determine a rate of leakage ϕ of tracer gas.

Such an embodiment is advantageous in that it makes it possible to reduce the diffusion time of the tracer gas inside the detection hood 55 and thus makes it
 25 possible to reduce the minimum measurement time. This is in particular advantageous when the time for the tracer gas to migrate from one end to another of the detection hood 55 is likely to be long because the detection hood 55 is very long and/or when the negative pressure prevailing inside the detection chamber 61 is insufficient.

Although the invention has been described in connection with several
 30 specific embodiments, it is very obvious that it is in no way limited thereto and that it comprises all the technical equivalents of the means described and also their combinations, if the latter come within the scope of the invention.

The use of the verb "to comprise" or "to include" and of its conjugated forms does not exclude the presence of other elements or of other stages than those stated in a claim.

In the claims, any reference sign in brackets should not be interpreted as a
5 limitation of the claim.

CLAIMS

1. Method for injection of a tracer gas into a thermally insulating space of a leaktight and thermally insulating tank, the leaktight and thermally insulating tank exhibiting a polyhedral shape defined by a plurality of tank walls (9, 10, 11, 12, 13, 14, 15, 16, 17) which are attached to a carrier structure (4), each of the tank walls comprising a thermally insulating barrier (2, 6) comprising insulating elements (3, 7) which are anchored to the carrier structure (4) and a membrane (5, 8) resting against the insulating elements (3, 7) of the thermally insulating barrier (2, 6), the thermally insulating barriers (2, 6) of each tank wall communicating with the thermally insulating barrier (2, 6) of the other tank wall (9, 10, 11, 12, 13, 14, 15, 16, 17) one another and together defining the thermally insulating space; the said plurality of tank walls including a bottom wall (11), the said injection method comprising injecting a tracer gas into the thermally insulating space by means of at least one device for injection of tracer gas (19, 28) which passes through the membrane (5, 8) of the bottom wall (11) and which emerges in the thermally insulating barrier (2, 6) of the bottom wall (11), the tracer gas being introduced and transported from the inner space of the vessel to said thermally insulating space and wherein, prior to the stage of injection of the tracer gas by means of at least one device for injection of tracer gas (19), the membrane (5, 8) is detached from the thermally insulating barrier (2, 6) in the region of the device for injection of the tracer gas (19) by introducing a lifting tool (25) through a hole (20) made in the membrane (5, 8) and by positioning said lifting tool (25) so as to be interposed between the membrane (5, 8) and the thermally insulating barrier (2, 6) of the bottom wall (11).

2. Method according to Claim 1, in which the tracer gas is chosen from helium, ammonia and a mixture of nitrogen and molecular hydrogen.

3. Method according to Claim 1 or 2, in which the bottom wall (11) exhibits a rectangular shape exhibiting two axes of symmetry, the said two axes of symmetry delimiting four regions of the bottom wall, and in which the tracer gas is injected by means of at least four devices for injection of tracer gas (19, 28) which each pass through the membrane (5, 8) of the bottom wall (11) and which emerge in the thermally insulating barrier of the bottom wall (11), each of the four regions of the

bottom wall being equipped with one of the four devices for injection of tracer gas (19, 28).

4. Method according to any one of Claims 1 to 3, in which the or each device for injection of tracer gas (19, 28) comprises a coupling member (22, 31) which is welded in leaktight fashion to the membrane (5, 8) of the bottom wall (11) at the periphery of a hole (20, 29) which is made in the said membrane (5, 8), the coupling member (22, 31) being combined with a pipe (18) connected to a source of tracer gas.

5. Method according to Claim 4, in which, the lifting tool (25) is introduced through the coupling member (22) and the hole (20) made in the membrane (5, 8) and is positioned so that a hook (27) of the said lifting tool (25) is inserted between the membrane (5, 8) and the thermally insulating barrier (2, 6) of the bottom wall (11).

6. Method for testing the leaktightness of a membrane of a leaktight and thermally insulating tank, the leaktight and thermally insulating tank exhibiting a polyhedral shape defined by a plurality of tank walls (9, 10, 11, 12, 13, 14, 15, 16, 17) which are attached to a carrier structure (4), each of the tank walls comprising a thermally insulating barrier (2, 6) comprising insulating elements (3, 7) which are anchored to the carrier structure (4) and a membrane (5, 8) resting against the insulating elements (3, 7) of the thermally insulating barrier (2, 6), the thermally insulating barriers (2, 6) of the tank walls communicating with one another and together defining a thermally insulating space; the said plurality of tank walls including a bottom wall (11), the method for testing the leaktightness comprising:

- carrying out the method for injection of a tracer gas according to any one of Claims 1 to 5 in order to inject the tracer gas into the thermally insulating space;
- providing a leak detection device (54) comprising a detection hood (55), configured in order to define, with the membrane (5, 8), a leaktight detection chamber (61), and a vacuum pump (57) which is, on the one hand, coupled to the leaktight detection chamber (61) and, on the other hand, to an analytical apparatus (56);
- positioning the detection hood (55) against a face of the membrane (5, 8) opposite the thermally insulating space, the detection hood (55) being positioned so that the leaktight detection chamber (61) is positioned facing a portion of a welding bead (62) to be tested;

- placing the detection chamber (61) under negative pressure using the vacuum pump (57);

- conveying the gas phase present in the detection chamber (61) towards the analytical apparatus (56); and

5 - analysing, using the analytical apparatus (56), the said gas phase and delivering a variable representative of a concentration of tracer gas in the said gas phase.

7. Method for testing the leaktightness of a membrane according to Claim 6, in which the detection hood (55) comprises a central core (59) and a
10 leaktightness seal (60) which are attached to one another and are arranged in order to define, with the membrane (5, 8), the detection chamber (61).

8. Method for testing the leaktightness of a membrane according to Claim 7, in which the leaktightness seal (60) comprises a casing (63) attached to the central core (59) and a peripheral leaktightness lip (64) which extends the casing (63).

15 9. Method for testing the leaktightness of a membrane according to Claim 8, in which the peripheral leaktightness lip (64) is bent towards the outside of the detection hood (55) and is configured in order to be flexed and to be flattened against the membrane (5, 8) when the detection chamber (61) is placed under negative pressure.

20 10. Method for testing the leaktightness of a membrane according to claim 8 or 9, in which the portion of the welding bead (62) is traversed by at least one corrugation of the membrane and in which the peripheral leaktightness lip (64) is shaped in order to fit the geometry of the said corrugation.

11. Method for testing the leaktightness of a membrane according to
25 Claim 10, in which the at least one corrugation of the membrane (8) projects towards the inside of the tank and in which the peripheral leaktightness lip (64) comprises at least two indentations (65) exhibiting a shape corresponding to that of the corrugation, the detection hood (55) being positioned against the membrane (8) so that the indentations (65) straddle the corrugation.

30 12. Method for testing the leaktightness of a membrane according to Claim 10, in which the at least one corrugation of the membrane (5, 8) projects towards the outside of the tank and in which the peripheral leaktightness lip (64)

comprises at least two regions in projection (69) exhibiting a shape corresponding to that of the corrugation, the detection hood (55) being positioned against the membrane (5) so that the regions in projection are inserted into the corrugation.

13. Method for testing the leaktightness of a membrane according to any one of Claims 8 to 12, in which the detection hood (55) is equipped with a clamping system (66) capable of flattening the peripheral leaktightness lip (64) against the membrane (8) and in which, prior to placing the detection chamber (61) under negative pressure, the clamping system (66) is actuated so as to guarantee the leaktightness of the detection chamber (61).

14. Method for testing the leaktightness of a membrane according to any one of Claims 7 to 13, in which the leaktightness seal (60) is made of an elastomer material exhibiting a Shore A hardness of between 20 and 50.

15. Method for testing the leaktightness of a membrane according to any one of Claims 6 to 14, in which the detection chamber (61) is placed under negative pressure down to an absolute pressure value of between 50 and 1000 Pa.

16. Method for testing the leaktightness of a membrane according to any one of Claims 6 to 15, in which the leak detection device additionally comprises a homogenization chamber (70) which is positioned between the detection chamber (61) and the analytical apparatus (56), the detection hood (55) comprising a gas inlet (71) equipped with a valve, the homogenization chamber (70) and the gas inlet (71) being coupled to two opposite ends of the detection chamber (61), the valve of the gas inlet (71) being closed during the placing of the detection chamber (61) under negative pressure, the stage of conveying the gas phase present in the detection chamber (61) comprising:

- opening the valve of the gas inlet (71) and conveying the gas phase present in the detection chamber (61) towards the homogenization chamber (70); and
- conveying the gas phase from the homogenization chamber (70) towards the analytical apparatus (56).