



(51) International Patent Classification:
C01B 7/20 (2006.01) **C25B 1/24** (2006.01)

(21) International Application Number:
PCT/EP2011/065836

(22) International Filing Date:
13 September 2011 (13.09.2011)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
10177216.8 16 September 2010 (16.09.2010) EP

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(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— with international search report (Art. 21(3))

(54) Title: FLUORINE GAS PLANT WITH SEISMIC PROTECTION

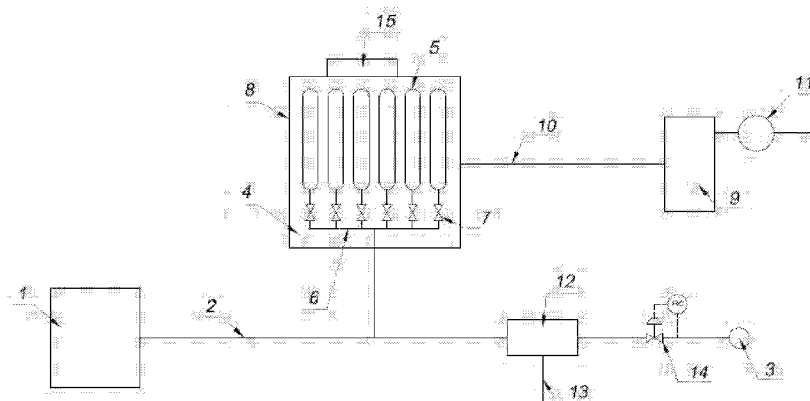


Fig 1

(57) Abstract: A fluorine gas plant comprising a fluorine generating unit (1) connected to a fluorine supply line (2) having a point of use of fluorine (3) is disclosed which comprises a seismometer. The seismometer preferably is an accelerometer and detects earthquakes. It generates a signal which can trigger the shutdown of predetermined processes running in the plant.

Fluorine gas plant with seismic protection

The invention which claims priority to European patent application N° 10177216.8 filed September 16, 2010 the whole content of this application being incorporated herein by reference for all purposes concerns a fluorine gas plant with protection against damages which may be caused by earthquakes.

5 Fluorine gas such as molecular fluorine (F_2) and mixtures thereof are useful, for example as cleaning gas for process chambers in semiconductor manufacturing processes or as an etching gas for the manufacture of semiconductors, photovoltaic devices or flat panel displays.

Document WO 2006/067364-A1 discloses in a method of delivering high
10 purity fluorine to a processing system, wherein an on-site fluorine generator supplies high purity fluorine to a fixed storage unit, from which the high purity fluorine is supplied to the processing system.

On-site fluorine generating plants (" F_2 gas plants") often comprise tanks for HF storage, electrolytic cells for producing raw F_2 gas and H_2 as side
15 product, means for the purification of the raw F_2 gas to obtain pure F_2 , means including buffer tanks for the delivery of the F_2 to the point of use, scrubbers for the abatement of F_2 and HF in gases ventilated from the ambient air in the F_2 gas plant, scrubbers for the abatement of F_2 considered as being outside the specification, emergency scrubbers to abate F_2 and HF in case of leakages,
20 scrubbers to remove HF from H_2 which is also formed as a product, thermo-regulated water-based circuits which heat the electrolytic cells before the electrolysis reaction is started, and which cool the cells during the electrolysis reaction, and which cool the raw F_2 gas leaving the cells. The HF is stored in HF tanks which are pressurized to convey HF to the cells via valves which are open
25 when the F_2 generator produces F_2 . Evaporators provide heat to the HF to introduce it into the cells in evaporated form. The F_2 produced is purified, i.a. by passing it through heated NaF absorber towers. The plant usually comprises means for analyzing the raw and the purified F_2 gas, e.g. FT-IR and UV spectrometers, electrical rectifiers to provide electric current to the electrolytic
30 cells, transformers, utilities and amenities like a laboratory, a control board room wherein process data can be controlled on one or more control boards, and rest rooms for the personnel, and if desired, an emergency power supply, e.g. a diesel

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generator. The produced F_2 is pressurized before being conveyed to the customer.

Elemental F_2 , HF, and gases comprising F_2 or HF are, of course, potentially hazardous compounds. Consequently, precautions are taken in F_2 gas plants to prevent hazards. Housings are well ventilated, waste gas is passed through scrubbers, gas detectors are distributed in the plant, smoke detectors and fire extinguishing installations are present, and emergency push buttons are distributed in the plant for a manual shutdown if needed. Many parts essential for operating the plant are redundant, for example, pumps and scrubbers.

There is still a need for improvements in respect of the safe operation of fluorine gas plants. The present invention provides an improved fluorine gas plant and an improved process for manufacturing pure F_2 in a safer manner.

The fluorine gas plant according to the present invention comprises an F_2 generation unit (1) connected to a fluorine gas supply system (2) having a point of use of fluorine gas (3), and a fluorine gas storage unit (4) connectable to said supply system, wherein said fluorine gas plant comprises at least one seismometer (15). The reference signs refer to the attached drawing in figure 1. If desired, the parts of the plant can be assembled in the form of skids.

Short explanation of the drawing

Figure 1 shows a fluorine gas plant according to the invention. The plant comprises a fluorine storage unit (4) with multiple storage containers (5) for F_2 which is delivered to a point of use (3). A seismometer (15) is attached to the fluorine storage unit (5).

Detailed description of the invention

The seismometer should be resistant to aggressive media and comprises preferably a respective housing. It can be bolted, for example, to parts of the fluorine gas plant, or to the ground. Preferably, at least 2 seismometers are foreseen for redundancy.

The plant preferably comprises a shutdown system. The shutdown system shuts down predetermined processes.

The seismometer generates a signal in the event of detecting an earthquake. The signal is at least one signal selected from the group consisting of an optical signal, an acoustic signal, and an electric signal sent to a receiver, e.g. a receiver integrated into a control board. Preferably, the seismometers generates an electric signal sent either to the respective units of the plant for shutdown, or to a control board which actuates the automatic shutdown switch shutting down at

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least one of the processes running in the fluorine gas plant. The control board may be organized such that different processes are allocated to a single shutdown switch or each process may be allocated to a separate shutdown switch.

Preferably, the signal of the seismometer is sent to the control board which
5 in turn automatically actuates the shutdown switch or switches. The invention will now be explained for the alternative wherein one shutdown switch stops several running processes. The shutdown switch shuts down predetermined processes performed in the plant, e.g. the electrolysis process, the delivery of fluorine to the point of use, or even all processes performed in the F₂ production
10 including scrubbing, purification, and analysis.

The connection between seismometer and automatic shutdown switch may be in the form of a cable through which a digital or analogous electric signal can be sent. Alternatively, seismometer and the shutdown switch may be connected via electromagnetic waves sent from the seismometer by a transmitter and
15 received a receiver. It is preferred that the seismometer is connected to a control board which includes the shutdown system.

The seismometer detects seismic activity, especially earthquakes. If an earthquake is detected, the seismometer sends an electronic signal to the automatic shutdown switch (or switches), preferably comprised in a control
20 board. The shutdown switch stops automatically predetermined running processes of the fluorine gas plant. Preferably, the switch interrupts the electric current passing through the rectifiers to the electrolytic cells and thus stops the F₂ generating process. It is preferred that warning signals are initiated by the seismometer signal, e.g. acoustic signals, e.g. specific sounds or announcements,
25 or optic signals, like warning lights. The seismometer may also send a signal to the facility using the fluorine gas produced indicating that F₂ delivery will be interrupted. "Fluorine gas" is understood to denote in particular molecular fluorine (F₂) and mixtures thereof, in particular with inert gases. Inert gases can be selected for example from N₂O, oxygen, argon and nitrogen. A preferred
30 fluorine gas consists or consists essentially of F₂.

The seismometer may also trigger other actions. For example, valves of storage tanks comprising HF, fluorine gas or F₂ may be shut so that no gas is further distributed through lines and pipes, or electric heating may be switched off which is applied to heat water or cooling liquid which keeps the temperature
35 of the electrolyte salt in the electrolysis cells at a temperature in the range between about 80 and 100 °C. Valves may be closed to isolate lines or pipes to

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prevent the intrusion of air or moisture. The capacity of fans ventilating the air of the plant through scrubbers may be increased. Inerting gas may be blown into specific parts of the fluorine gas plant. The gas or liquid in pipes or lines may be forced to pass through the scrubbers or emergency scrubbers, for example, by passing N₂ through them. This measure will also be helpful to prevent air or moisture of intruding into the lines or pipes which improves the later restart of the plant. Precautionary, a diesel generator can be started to safeguard supply of electric power.

Preferably, the seismometer is an accelerometer.

The advantage of using a seismometer, especially an accelerometer, is that it is much more sensitive than humans in the detection of developing earthquakes. Due to the early detection of the earthquakes, the plant can be shut down in a safe or at least less hasty manner, and additional safety precautions may be performed by the personnel present in the plant including precautions to protect themselves.

Seismometers suitable for application in the F₂ gas plant are generally known. Accelerometers are especially suitable as seismometers because they can measure seismic activity very well. Accelerometers with very differing working principles are known. For example, accelerometers are suitable which are based on the principle of resistivity (potentiometric or extensimetric), of inductivity, electromagnetism, electrostatics, piezo resistance, piezoelectricity or optoelectronic according to the Doppler principle are known. These accelerometers detect accelerations ranging from 10⁻² ms⁻² (some even from 10⁻⁹ msec⁻²) to 10² msec⁻² (some even up to 10⁶ msec⁻²) and frequencies of >0 to 10² Hz (some even up to 10⁴ Hz).

A specific device which may be applied in the F₂ gas plant of the present invention as described in US-A 5,742,235. In this device, a mass is suspended on the end of a flat spring mounted on a base which is fastened to elements of, or objects within a structure in contact with the earth. The motion of the mass is decoupled from that of the base. When the base experiences the upward accelerations and displacements characteristic of the initial shock waves of a major earthquake the inertia of the mass exerts a relative downward force on the spring closing electrical contacts allowing current to flow from a battery through a light. The spectral response of the assembly can be readily tuned to any desired acceleration and displacement thresholds by adjusting the inertial mass,

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spring and damping forces of the assembly and the distance between the contacts.

The invention will now be described in view of a preferred embodiment wherein fluorine gas including F_2 is stored in a plurality of hollow bodies. In a preferred embodiment, the fluorine gas plant comprises stored F_2 to provide a back up to the fluorine generator. A stable and economic supply of required quantities of fluorine gas to a point of use, while minimizing safety risks related to the presence of F_2 is especially possible if the fluorine gas plant comprises a F_2 generating unit (1) connected to a fluorine gas supply system (2) having a point of use of fluorine gas (3), and a permanent fluorine gas storage unit (4) connectable to said supply system, wherein said fluorine gas storage unit comprises a plurality of hollow bodies (5).

“Permanent fluorine gas storage unit” is understood to denote in particular a fluorine gas storage unit which is integrated into the fluorine plant. Preferably, the permanent fluorine gas storage unit is designed to contain more than 90 wt % more preferably more than 95 wt %, most preferably about 100 wt % of the fluorine gas relative to the total weight of fluorine gas stored in the plant. Preferably, F_2 is stored in the form of a gas.

“Fluorine gas supply system” is understood to denote in particular an element which can contain fluorine gas and which is able to convey fluorine gas from the F_2 generating unit to the point of use. Possible components of a fluorine gas supply system include but are not limited to supply lines, compressors, mixers and buffer tanks.

“Connectable” is understood to denote in particular that the permanent fluorine gas storage unit is equipped to be able to be connected to a component of the fluorine gas supply system. Suitable equipment for connecting the fluorine storage unit connected to a component of the fluorine gas supply system includes a manifold (6) connected to each hollow body (5) of the fluorine gas storage unit through a line and preferably having a shut-off valve (7) in each line allowing to individually isolate each hollow body and said manifold is further connected to a component of the fluorine gas supply system.

The fluorine gas storage unit preferably comprises from 4 to 25 hollow bodies, more preferably from 5 to 8 hollow bodies. The hollow bodies are preferably of substantially identical shape and dimensions. Cylindrically shaped hollow bodies are preferred. Each hollow body preferably contains maximally about 6 to 10 kg, preferably about 8 kg, of F_2 .

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In a preferred embodiment of the fluorine gas plant according to the invention, the fluorine gas storage unit is comprised in an enclosed space (8). The enclosed space generally comprises a fluorine sensor capable to trigger connection of the enclosed space to a fluorine destruction system (9). Suitably,
5 the enclosed space is connected to the fluorine destruction system through a suction line (10) connected to a pump (11) which is operable to pump gas from the enclosed space to the fluorine destruction system.

In a further embodiment, the fluorine gas plant according to the invention further comprises a mixer (12), preferably a static mixer, said mixer being
10 preferably capable to receive fluorine from the fluorine generating unit (1) and to receive inert gas, such as preferably argon and/or nitrogen, from an inert gas supply line (13).

The invention also concerns a method for supply of fluorine gas comprising use of the fluorine gas plant according to the invention to supply
15 fluorine gas to a point of use.

Fig. 1 shows an illustrative preferred fluorine plant of the invention.

F₂ generating unit (1) is connected to fluorine gas supply system (2) which comprises a fluorine gas supply line connected to point of use (3), which is connected to a semiconductor manufacturing plant. In an optional embodiment
20 shown as dotted lines, the fluorine gas supply line is connected to mixer (12) which is further connected to inert gas supply line (13) and a further fluorine gas supply line connects the mixer (12) via valve (14) to the point of use (3). The fluorine gas supply system (2) is further connected to fluorine gas storage unit (4) through manifold (6) connected to each hollow body (5) of the fluorine
25 gas storage unit through a line and preferably having a shut-off valve (7). The fluorine gas storage unit is contained in enclosed space (8) which comprises a fluorine sensor capable to trigger connection of the enclosed space to the fluorine destruction system (9). The enclosed space is connected to the fluorine destruction system through a suction line (10) connected to a pump (11) which is
30 operable to pump gas from the enclosed space to the fluorine destruction system. A seismometer (accelerometer) (15) is located on the housing of the storage unit (8). It could as well be located anywhere in the plant or even in close proximity. It detects seismic vibrations and provides a respective signal to a control board which, i.a., automatically shuts off any hollow body (5) which is in
35 fluid contact with lines and pipes providing fluorine gas to the point of use.

Preferably, also the emergency scrubber is put to “stand by” so that in case of the detection of hazardous gas (HF or F₂) the ambient atmosphere containing the hazardous gas can be ventilated through the emergency scrubber for abatement. Generally, the emergency scrubber is capable to destroy fluorine gas, especially F₂, contained in 1 or 2 hollow bodies, preferably to destroy fluorine gas, especially F₂, contained in about 1 hollow body.

Thus, the preferred embodiment which provides an F₂ back up in the form of hollow bodies each of which contains only a fraction of the total F₂ back up of the plant, coupled with a seismometer, for example an accelerometer, improves the safety of the fluorine gas plant. The seismometer allows a quick reaction to a developing earthquake, and the provision of hollow bodies containing only a fraction of fluorine gas if compared to a tank which contains the total amount of back up fluorine gas. This is especially the case if an emergency scrubber is present with the capacity to abate the F₂ escaping one or more of the hollow bodies.

The invention also provides a method for producing fluorine gas in a fluorine gas plant comprising a fluorine generation unit (1) connected to a fluorine supply system (2) having a point of use of fluorine gas (3), and a fluorine gas storage unit (4) connectable to said supply system, wherein said fluorine gas plant comprises at least one seismometer, in which method a current is passed through a molten KF/HF composition which is electrolyzed thereby to form F₂ and H₂, the resulting raw F₂ is purified and delivered, optionally after mixing with inert gas, to the point of use and/or to a fluorine gas storage unit, and wherein, if the seismometer detects vibrations originating from an earthquake, a signal is generated and causes the shut down one or more processes performed in the fluorine gas plant.

Preferably, the electrolysis process is shut down.

Preferably, also the fluorine gas delivery to the point of use is shut down.

Preferably, the signal closes a valve to shut off the fluorine gas storage unit (4).

It is preferred that the generated signal is sent to a control board which initiates the shutdown of the process or processes.

The fluorine gas plant according to the invention and the method according to the invention allow for stable, economic and especially safe fluorine gas supply, even in regions where earthquakes occur. In particular, the seismometer,

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the fluorine gas storage system and optional fluorine destruction system are highly efficient, allowing for high safety of the plant.

Should the disclosure of any patents, patent applications, and publications which are incorporated herein by reference conflict with the description of the present application to the extent that it may render a term unclear, the present description shall take precedence.

Example

A fluorine plant in accordance with the basic scheme of figure 1 (without mixer and inert gas supply) comprises a seismometer. In the plant, about 415 kg/day of F_2 is produced in the F_2 generating unit (1) through HF electrolysis in molten $KF \cdot 2HF$ electrolyte. The F_2 is conveyed through the fluorine gas supply system (2) to point of use (3) where F_2 is supplied to a flat panel display manufacturing plant using F_2 for chamber cleaning. The F_2 is supplied on demand to the point of use.

The fluorine pressure in the fluorine supply system (2) and in the F_2 storage unit (4) is maintained at 3.5 barg (about 55 psig) by adapting the F_2 production in the F_2 generating unit. The F_2 storage unit (4) comprises 6 identical cylindrically shaped containers having each an internal volume of 1.3 m^3 . The fluorine pressure at the point of use (3) is further reduced at 1.5 barg (about 24 psig) by a pressure control loop (14).

In normal operation, the shut-off valves (7) are open and the pressure difference between the F_2 storage unit (4) and the point of use (3) provides a buffer allowing a smooth control of the delivered fluorine gas flow, even for variable consumption patterns at the point of use (3) or during interruption of the production of the F_2 generating unit.

If the seismometer (15) detects accelerations characteristic for an earthquake (or a heavy explosion close to the plant), it sends a signal which may be analogous or digital to a control board in the plant. The control board, i.e., cuts off the current to the electrolytic cells and the fluorine supply from that unit to the supply system (2). It also cuts off any supply of F_2 stored in the F_2 storage unit (4). Further, pressurization of HF feed tanks, HF evaporation, HF supply valves to the electrolytic cells, the heaters for the NaF absorption towers and the valve delivering the F_2 or F_2 /inert gas mixture to the customer is shut off.

Any fluorine containing gas from the enclosed space is pumped through suction line (10) to the fluorine destruction system (9) which is a scrubber

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containing an aqueous KOH/potassium or sodium thiosulphate mixture. The scrubber is able to treat 15 kg F₂.

C L A I M S

1. A fluorine gas plant comprising a fluorine generation unit (1) connected to a fluorine supply system (2) having a point of use of fluorine gas (3), and a fluorine gas storage unit (4) connectable to said supply system,
5 wherein said fluorine gas plant comprises at least one seismometer.
2. The plant of claim 1 wherein the seismometer is an accelerometer.
3. The plant of claims 1 or 2 comprising a shut down system.
4. The plant of claims 1, 2 or 3 wherein the fluorine gas storage unit comprises a plurality of hollow bodies (5).
- 10 5. The plant according to claim 4, wherein the fluorine storage unit comprises from 5 to 8 hollow bodies.
6. The plant according to anyone of claims 1 to 5 wherein the seismometer is connected to a control board and sends a signal to the control board.
- 15 7. The plant according to anyone of claims 1 to 6 further comprising an emergency scrubber capable of abating the F₂ content of at least one hollow body (5).
8. The plant according to anyone of claims 3 to 7 wherein the shutdown system is connected with the power supply for the F₂ generating unit.
- 20 9. The plant according to anyone of claims 3 to 8 wherein the shutdown system is connected with valves capable of shutting down the fluorine gas storage unit (4).
10. A method for producing F₂ gas in a fluorine gas plant comprising a fluorine generation unit (1) connected to a fluorine gas supply system (2) having
25 a point of use of fluorine gas (3), and a fluorine gas storage unit (4) connectable to said supply system, wherein said fluorine gas plant comprises at least one seismometer, in which method a current is passed through a molten KF/HF composition which is electrolyzed thereby to form F₂ and H₂, the resulting raw F₂ is purified and delivered to the point of use and/or to a fluorine gas

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storage unit, and wherein, if the seismometer detects vibrations originating from an earthquake, a signal is generated and causes the shut down one or more processes performed in the fluorine gas plant.

11. The method of claim 10 wherein the electrolysis process is shut down.

5 12. The method of claims 10 or 11 wherein the fluorine gas delivery to the point of use is shut down.

13. The method of anyone of claims 10 to 12 wherein the signal closes a valve to shut off the fluorine gas storage unit (4).

10 14. The method of anyone of claims 10 to 13 wherein the generated signal is sent to a control board which initiates the shutdown of the process or processes.

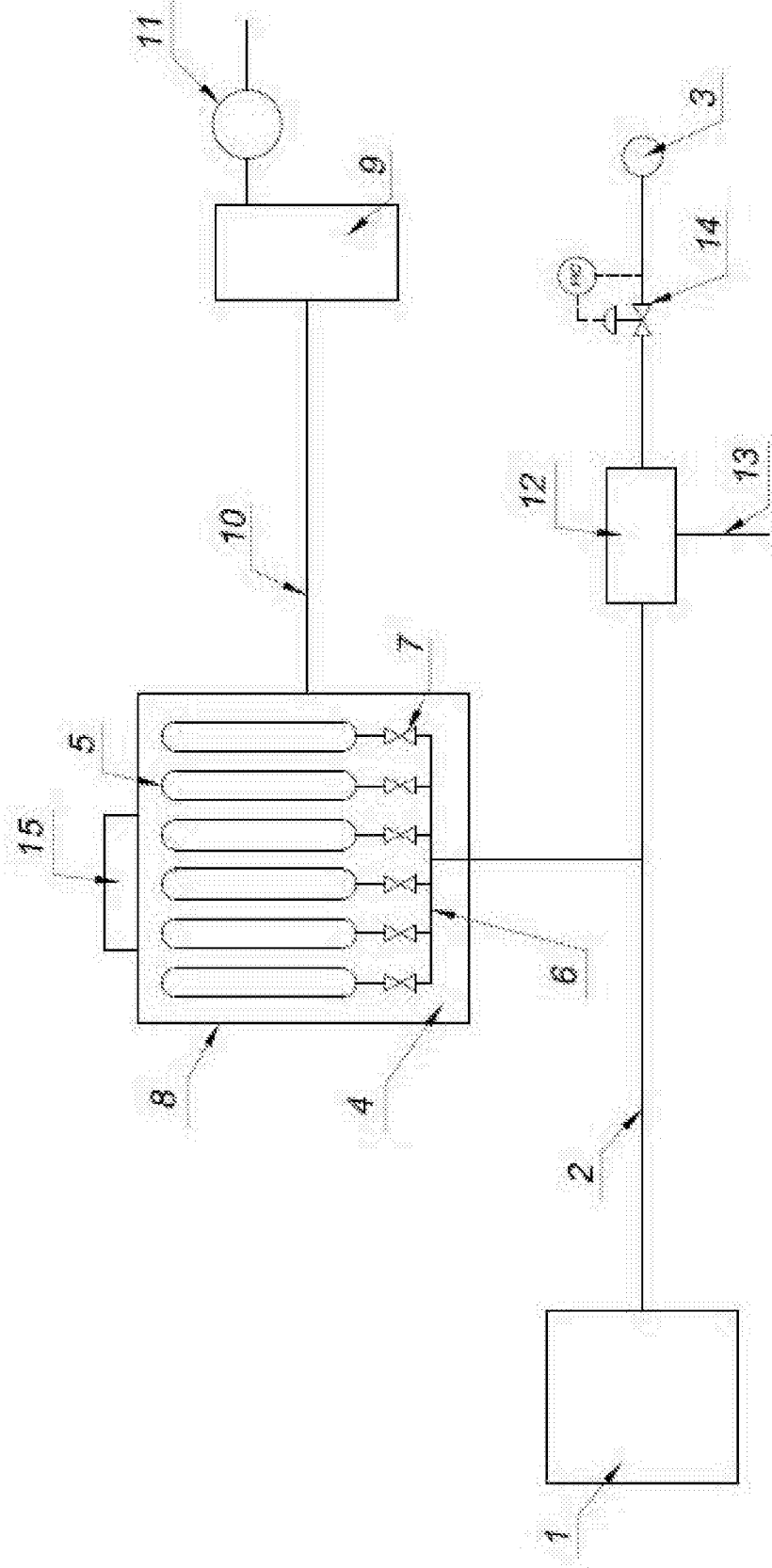


Fig 1

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2011/065836

A. CLASSIFICATION OF SUBJECT MATTER
INV. C01B7/20 C25B1/24
ADD.

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C01B C25B F17C F17D G01V G08B

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

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

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier document but published on or after the international filing date
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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
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Date of the actual completion of the international search

13 October 2011

Date of mailing of the international search report

24/10/2011

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INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2011/065836

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

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