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(71) **Applicant: INTERNATIONAL ENGINE INTELLEC-
TUAL PROPERTY COMPANY, LLC** [US/US]; 2701
Navistar Drive, Lisle, Illinois 60532 (US).

(72) **Inventors: LACK, Adam C.**; 8733 Willow Blvd., #4D,
Willow Springs, Illinois 60480 (US). **SINGH, Navtej**; 912
W. Nichols Road, Arlington Heights, Illinois 60004 (US).
VENKATESH, Prasanna Nagabushan; 1324 South Fin-
ley Road, Apt # 3G, Lombard, Illinois 60148 (US). **ROEP-
KE, Randall R.**; 632 N. Racine Ave., #2N, Chicago,
Illinois 60642 (US). **MILLER, Michael James**; 600 W.
Larkdale Lane, Mt. Prospect, Illinois 60056 (US).

(74) **Agent: BACH, Mark C.;** 2701 Navistar Road, Lisle, Illinois 60532 (US).

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— *as to the identity of the inventor (Rule 4.17(i))*

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(54) Title: AMMONIA ESTIMATION METHOD

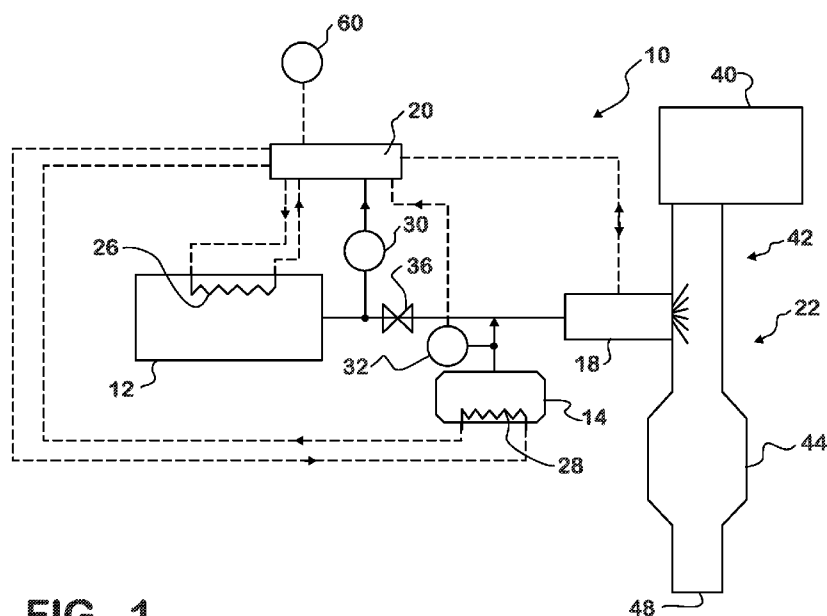


FIG. 1

(57) Abstract: A method for determining the degree of saturation of a solid ammonia storage material in a storage unit includes activating a heater to release ammonia from the storage material. The method monitors the pressure of the storage unit while the heater is active to determine a rate of pressure increase in the storage unit. The method estimates the degree of saturation of the ammonia storage medium in response to the rate of pressure increase. According to some embodiments, determining the decay rate may include measuring the time required for the pressure of the storage unit to rise from a first pressure threshold to a second pressure threshold. The method may further include sensing ambient temperature and compensating the estimated degree of saturation based on ambient temperature.



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- *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*
- *as to the applicant's entitlement to claim the priority of the earlier application (Rule 4.17(iii))*
- Published:**
- *with international search report (Art. 21(3))*

AMMONIA ESTIMATION METHOD

BACKGROUND

[0001] Selective catalytic reduction (SCR) is commonly used to remove NO_x (*i.e.*, oxides of nitrogen) from the exhaust gas produced by internal engines, such as diesel or other lean burn (gasoline) engines. In such systems, NO_x is continuously removed from the exhaust gas by injection of a reductant into the exhaust gas prior to entering an SCR catalyst capable of achieving a high conversion of NO_x.

[0002] Ammonia is often used as the reductant in SCR systems. The ammonia is introduced into the exhaust gas by controlled injection either of gaseous ammonia, aqueous ammonia or indirectly as urea dissolved in water. The SCR catalyst positioned in the exhaust gas stream causes a reaction between NO_x present in the exhaust gas and a NO_x reducing agent (*e.g.*, ammonia) to reduce/convert the NO_x into nitrogen and water.

[0003] In many applications, such as SCR systems for vehicles, for example, the storage of ammonia in the form of a pressurized liquid in a vessel may be too hazardous and a storage method involving absorption in a solid may circumvent the safety hazard of anhydrous liquid ammonia. For example, metal ammine salts are ammonia absorbing materials, which can be used as solid storage media for ammonia, which in turn, for example, may be used as the reductant in SCR to reduce NO_x emissions from internal combustion engines in vehicles, *see e.g.*, U.S. Patent No. 8,088,201 and WO 1999/01205. The ammonia can be released from the ammine salts through thermal desorption, *e.g.*, by external heating of a storage container, *see e.g., id.* and U.S. Patent App. Pub. No. 2010/0086467. The ammonia is released from an either adsorptive or absorptive solid

storage medium, among others $\text{Sr}(\text{NH}_3)_8\text{Cl}_2$ or $\text{Ca}(\text{NH}_3)\text{Cl}_2$ in granular form, in a storage container and temporarily stored as a gas in a buffer volume. The amount of ammonia to be supplied to a reaction volume in the vehicle's exhaust system is dosed under the control of an electronic controller according to the current operating state of the engine.

[0004] The ammonia is consumed during driving, and as a result, the storage medium is depleted over time. Eventually, the ammonia in a canister is depleted and must be recharged or replaced. Replacing the canisters too early is obviously undesirable, as it can result in increased cost and vehicle down time, for example. Conversely, if the user waits too long to recharge or replace the canister, the SCR system may stop functioning correctly, causing undesirable NOx emissions. Accordingly, it is desirable to be able to determine the fill level of the storage container and to alert the user when the canister is nearing depletion.

SUMMARY

[0005] Aspects and embodiments of the present technology described herein relate to one or more systems and methods for estimating the saturation level of a solid ammonia storage medium.

[0006] At least some embodiments of the present technology relate to a method for determining the degree of saturation of a reversible solid ammonia storage material in a storage unit. The storage unit is equipped with a heater to release ammonia. The method includes activating the heater and monitoring the pressure of the storage unit to determine a rate of pressure increase in the storage unit. The method then estimates the degree of saturation in response to the rate of pressure increase.

[0007] According to at least some embodiments, the method may determine the rate of pressure increase by measuring the time required for the pressure of the storage unit to increase from a first pressure threshold P1 to a second pressure threshold P2. The method then estimates the degree of saturation ammonia storage medium in response to the measured time. In some embodiments, the estimating step includes accessing a look-up table that correlates the measured time to a degree of saturation. Further, according to at least some embodiments, the method may further indicate a saturation level in response to the estimated saturation level. In some embodiments, the method may include sensing the ambient temperature and compensating the estimated saturation level in response to the ambient temperature.

[0008] One or more embodiments of the present technology relate to a system for determining the degree of saturation of a solid ammonia storage material in a storage unit. The storage unit includes a heater to release ammonia from the storage material. The system includes a pressure sensor and a controller. The pressure sensor senses the internal pressure of the storage unit and produces a pressure signal responsive thereto. The controller is configured to monitor the pressure signal, selectively activate the heater to release ammonia from the storage material, determine a rate of pressure increase in the storage unit, and determine the degree of saturation of the ammonia storage medium in response to the determined decay rate. In some embodiments, the controller determines the decay rate by measuring the time required for the pressure signal to drop from a first threshold to a second threshold. In some embodiments, the system further includes a temperature sensor configured to sense ambient temperature and produce an ambient temperature signal in response thereto. The controller may be configured to receive the

ambient temperature signal and compensate the estimated saturation level in response thereto.

BRIEF DESCRIPTION OF THE DRAWINGS

[0009] FIG. 1 is a schematic illustration of an exemplary ammonia storage and dosing system that can be used to implement at least one embodiment of the present technology.

[0010] FIG. 2 is an exemplary graph illustrating the relationship between ammonia saturation level and pressure.

[0011] FIG. 3 is an exemplary graph illustrating the relationship between the energy supplied to the heater and the rate of pressure increase in a storage unit.

[0012] FIG. 4 is a flow diagram of an exemplary method for estimating the saturation level of a solid ammonia storage medium according to at least one embodiment of the present technology.

DETAILED DESCRIPTION

[0013] Various examples of embodiments of the present technology will be described more fully hereinafter with reference to the accompanying drawings, in which such examples of embodiments are shown. Like reference numbers refer to like elements throughout. Other embodiments of the presently described technology may, however, be in many different forms and are not limited solely to the embodiments set forth herein. Rather, these embodiments are examples representative of the present technology. Rights based on this disclosure have the full scope indicated by the claims.

[0014] FIG. 1 is a schematic diagram illustrating an embodiment of an exemplary ammonia storage and dosing system 10. In the illustrated embodiment, the ammonia storage and dosing system 10 includes a main storage unit 12 and a start-up storage unit 14. In the example of FIG. 1, the main storage unit 12 is made up of a single storage container, which holds ammonia storage material. It will be appreciated, however, that the storage material of the main storage unit 12 can be held in more than one container. The start-up storage unit 14 can be relatively small compared to the main storage unit 12, to facilitate rapid start up. The number and the size of the other containers that make up the main storage unit 12 can vary in accordance with design and performance parameters, including, for example, the desired total ammonia amount reserves and the start-up time of the main storage unit. The coupling of several storage containers can be performed in a variety of different ways in combination with passive one-way valves, active valves, pressure sensors, pressure switches, etc. Additional details and considerations regarding the size and number of storage units are provided in U.S. Patent Application Pub. No. 2010/0086467 (the “467 Publication”), the disclosure of which is hereby incorporated in its entirety.

[0015] The storage units 12, 14 are fluidly connected to a dosing valve 18 by which ammonia from the storage units 12, 14 is dosed according to a demand to an ammonia consuming process or system 22, such as a selective catalytic reduction (SCR) system. An electronic control unit 20 controls operation of the dosing valve 18 to control delivery of ammonia from the storage and dosing system 10 to the consuming system 22

[0016] Pressure sensors 30, 32 monitor the pressure in the main and start-up storage units 12, 14, respectively and produce signals indicative of the sensed pressures. The ECU 20 is coupled to the pressure sensors 30, 32 for receipt of the pressure signals.

[0017] Heating units 26, 28, such as electrical heaters, are provided for heating the main storage unit 12 and the start-up storage unit 14, respectively. The heating units 26, 28 can be placed inside the main storage unit 12 and the start-up storage unit 14 containers, respectively

[0018] The ECU 20 is operable to control (*e.g.*, switch on or off, and/or regulate) the heating units 26, 28 independently from each other, *e.g.*, by controlling the power supplied to them. The ECU can be programmed to regulate operation of the start-up heating unit 28 to initially raise pressure P_S in the start-up storage unit 14 to its activation pressure. Once the activation pressure of the start-up storage unit 14 is reached, the start-up heating unit 28 is modulated on and off to maintain pressure P_S in the start-up storage unit 14 at or about its activation. When the start-up heating unit 28 is cycled off, the main heating unit 26 is cycled on to gradually raise the pressure P_M in the main storage unit 12 towards its activation pressure. During the start-up mode, the main and start-up heating units 26, 28 are modulated to maintain the start-up storage unit 14 around its activation pressure, while raising the pressure P_M in the main storage unit 12 during the times that the start-up heating unit 28 is off. Accordingly, the pressure in the start-up heating unit 28 can be quickly raised to the activation pressure so that the system can begin supplying reductant from the start-up storage unit 14. Once the pressure P_M in the main storage unit 12 reaches its activation pressure, the system/method transitions to the main mode where reductant is supplied from the main storage unit 12. During the main mode, the start-up

heating unit 28 can remain inactive, while the main heating unit 26 is modulated on and off to maintain the pressure in the main storage unit 12 at or about its activation pressure. Because the heating units 26, 28 are never active at the same time, the power requirements, *e.g.*, from a vehicle power system, can be reduced.

[0019] According to at least some embodiments, a valve can fluidly couple the main storage unit 12 and the start-up storage unit 14. In some embodiments, the valve can be a passive, one-way valve 36. The one-way valve 36 closes when the pressure downstream of it (*i.e.*, the pressure in the start-up storage unit 14) is higher than that upstream of it (*i.e.* lower than the pressure in the main storage unit 12), and opens when the upstream pressure becomes higher than the down-stream pressure. Accordingly, the one-way valve 36 allows the main storage unit 12 to resaturate the smaller (rapid) start-up storage unit 14 with ammonia, *e.g.*, in situations where the system 10 is powered off (because the pressure in the main storage unit 12 will be higher than that in the start-up storage unit 14, when the main storage unit 12 is more saturated), or where heating of the start-up storage unit 14 has ceased while the main storage unit 12 continues to be heated. This increases the likelihood that the smaller, start-up storage unit, is available to make a rapid start-up. At the same time, the one-way valve 36 prevents ammonia from being introduced into the main storage unit 12 from the smaller start-up storage unit 14 when the pressure in the latter is higher, particularly during start-up.

[0020] The ECU 20 is also configured to control the dosing valve 18, *e.g.*, by providing the dosing valve with a variable dosing target value (for example, the dosing target value prescribes a certain degree of opening of the dosing valve). In the embodiment illustrated in FIG. 1, the ammonia storage and dosing system 10 is used to

supply reductant (*i.e.*, ammonia) for selective catalytic reduction (SCR) of NO_x in the exhaust emitted by an internal combustion engine 40. The ECU 20 (or another controller) controls delivery of ammonia from the storage and dosing system 10 and into an exhaust system 42 through the dosing valve 18. The dosing valve 18 may be positioned in the exhaust system 42 upstream from a catalyst 44. As the ammonia is injected into the exhaust system 42, it mixes with the exhaust gas and this mixture flows through the catalyst 44. The catalyst 44 causes a reaction between NO_x present in the exhaust gas and a NO_x reducing agent (*e.g.*, ammonia) to convert the NO_x into nitrogen and water, which then passes out of a tailpipe 48 and into the environment. While the system 10 has been described in the context of SCR for engine exhaust, it will be appreciated that the system could be used to supply ammonia in other applications, such as ammonia used as an energy carrier for a fuel cell or ammonia used as a reactant or additive in a chemical reaction, as described in greater detail in the aforementioned 467 Publication.

[0021] Certain aspects of the present technology relate to a system for determining the degree of saturation of a solid ammonia storage material in a storage unit, such as the main storage unit 12 and/or the auxiliary storage unit 14. For illustration purposes, the system will be described in connection with the main storage unit 12. In order to determine the ammonia saturation level of the storage unit 12, the ECU 20 may initially activate the heating unit 26 to release ammonia from the solid ammonia storage material. As will be appreciated, this step can occur during normal operation ammonia storage and dosing system 10, such as upon power-up of the system 10, or may be performed as a part of a separate process for estimating the saturation level of the main storage unit 12.

[0022] The ECU 20 monitors the pressure in the main storage unit 12 by monitoring the output of the pressure sensor 30. The ECU 20 monitors the pressure signal while the heating unit is activated (either continuously or modulated) to determine a decay a rate of pressure increase of the storage unit 12. The ECU 20 is configured to determine the degree of saturation of the ammonia storage medium in response to the determined rate of pressure increase. In this regard, the greater the saturation level (fill level) of the storage unit, the quicker the pressure will rise to the desired dosing pressure. This concept is illustrated in FIG. 2, which illustrates varying rates of pressure increase as a function of saturation level. As can be seen, the pressure rises more quickly to the dosing pressure (1.5 bar in the example) from a full storage unit than from a storage unit that is only partially full. Accordingly, the rate of pressure increase may be used to estimate the saturation level of the storage unit 12.

[0023] Furthermore, as shown in FIG. 3, for a given fill level, the rate of pressure increase may vary as a function of the amount of power delivered to the heater. In particular, increasing the power level will increase the rate at which ammonia is released from the storage medium (and, accordingly, the rate of pressure increase in the storage unit). Accordingly, the ECU 20 may compensate the estimated saturation level as a function of the power delivered to the heater. This strategy may also be used to account for the effects of a power modulation strategy where full power is not applied to the heater unit 26.

[0024] In addition, the system may include a temperature sensor 60 configured to sense the ambient temperature and produce an ambient temperature signal in response thereto. The ECU 20 may be configured to monitor the ambient temperature signal and

compensate the estimated saturation level in response thereto. In particular, as ambient temperature decreases, the time required to reach the desired dosing pressure will increase.

[0025] According to at least some embodiments, the ECU 20 may determine the rate of pressure increase by measuring the time required for the pressure signal to rise from a first pressure threshold P1 to a second pressure threshold P2. In some embodiments, the ECU 20 may determine saturation level by accessing a look-up table that correlates the measured time to a degree of saturation.

[0026] In some embodiments, the ECU 20 may be configured to provide an indication of the saturation level of the storage unit to a user. For example, the method may include controlling a display that provides an output indicative of the estimated saturation level to the user. Alternatively or additionally, the method may provide an alert when the estimated saturation level drops below a predetermined value, such as 10% of the maximum. The alert can take various forms, such as an audible and/or visual alert.

[0027] FIG. 4 is a flow chart illustrating at least one embodiment of a method 400 for estimating the saturation level of a solid ammonia storage medium according to at least one embodiment of the present technology. The method may be used, for example, to determine the saturation level of the main storage unit 12 and/or the start-up storage unit 14. For illustration purposes, the method will be described in connection with the main storage unit 12.

[0028] The method begins in step 405. Control is then passed to the step 410, where the method activates the heating unit 26 to release ammonia from the storage medium. The method continues to heat the main storage unit 12 until its pressure storage

unit reaches a predetermined pressure. In this regard, the method continues to loop through step 415 until the pressure of the main storage unit is at or above the predetermined pressure. In some embodiments the predetermined pressure may correspond to a desired dosing pressure. Once the predetermined pressure is obtained, the controller may modulate the heater to maintain the pressure in the storage unit at or near the predetermined pressure.

[0029] Once the predetermined pressure is reached, control is passed to step 420 where the method 400 determines the rate of pressure increase of the storage unit 12. According to at least some embodiments, the method 400 may determine the rate of pressure increase by measuring the time required for the pressure signal to rise from a first pressure threshold P1 to a second pressure threshold P2. In some embodiments, the second pressure threshold P2 may correspond to the predetermined pressure, *e.g.*, dosing pressure, of step 415.

[0030] Next, control is passed to step 425 where the method 400 determines the saturation level of the storage unit 12 based on the rate of pressure increase from step 420. According to some embodiments, the method 400 may further account for ambient temperature in estimating the saturation level. In some embodiments, the method 400 may determine saturation level by accessing a look-up table that correlates the rate of pressure rise (or measured time) to a degree of saturation. Further, in some embodiments, the look-up table may use ambient temperature as an input.

[0031] In some embodiments, the method 400 may further include the step 430 of providing an indication of the saturation level of the storage unit to a user. For example, the method 400 may include controlling a display that provides an output indicative of the

estimated saturation level to the user. Alternatively or additionally, the method 400 may provide an alert when the estimated saturation level drops below a predetermined value, such as 10% of the maximum. The alert can take various forms, such as an audible and/or visual alert.

[0032] In some embodiments, the method may be used to determine the saturation level following periods when the SCR system has been active, such as when the vehicle is turned off.

[0033] While this disclosure has been described as having exemplary embodiments, this application is intended to cover any variations, uses, or adaptations using the general principles set forth herein. It is envisioned that those skilled in the art may devise various modifications and equivalents without departing from the spirit and scope of the disclosure as recited in the following claims. Further, this application is intended to cover such departures from the present disclosure as come within the known or customary practice within the art to which it pertains.

CLAIMS

1. A method for determining the degree of saturation of a reversible solid ammonia storage material in a storage unit, the storage unit being equipped with a heater to release ammonia, the method comprising:

monitoring the pressure of the storage unit;

activating the heater;

measuring the time required for the pressure of the storage unit to rise from a first pressure threshold to a second pressure threshold; and

estimating the degree of saturation of the ammonia storage medium in response to the measured time.

2. A method as set forth in claim 1, wherein the step of estimating includes accessing a look-up table that correlates the measured time to a degree of saturation.

3. A method as set forth in claim 1, further comprising providing an indication of saturation level to a user in response to the estimated saturation level.

4. A method as set forth in claim 1, further comprising sensing ambient temperature and wherein the step of estimating the degree of saturation of the ammonia storage medium further includes compensating for ambient temperature.

5. A method for determining the degree of saturation of a solid ammonia storage material in a storage unit, the storage unit being equipped with a heater to release ammonia, the method comprising:

monitoring the pressure of the storage unit;

activating the heater to release ammonia from the solid ammonia storage material;

determining the rate of pressure increase of the storage unit while the heater is activated; and

estimating the degree of saturation of the ammonia storage medium in response to the rate of pressure increase.

6. A method as set forth in claim 5, further comprising sensing ambient temperature and wherein the estimating step comprising estimating the degree of saturation of the ammonia storage medium in response to the ambient temperature and the rate of pressure increase.

7. A method as set forth in claim 6, wherein the step of determining a rate of pressure increase further comprises measuring the time required for the pressure of the storage unit to rise from a first pressure threshold to a second pressure threshold.

8. A system for determining the degree of saturation of a solid ammonia storage material in a storage unit, the storage unit being equipped with a heater to release ammonia, the system comprising:

a sensor configured to sense the internal pressure of the storage unit and produce a pressure signal responsive thereto; and

a controller configured to monitor the pressure signal, selectively activate the heater to release ammonia from the storage material, determine a rate of pressure increase of the storage unit, and determine the degree of saturation of the ammonia storage medium in response to the determined rate of pressure increase

9. A system as set forth in claim 8, further comprising a temperature sensor configured to sense ambient temperature and produce a temperature signal in response thereto; and wherein the controller is configured to monitor the ambient temperature signal and determine the degree of saturation of the ammonia storage medium in response to the ambient temperature signal and the determined rate of pressure increase.

10. A system as set forth in claim 8, wherein the controller determines the rate of pressure increase by measuring the time required for the pressure signal to rise from a first pressure threshold to a second pressure threshold.

11. A system as set forth in claim 8, further comprising a valve connected to the storage unit for controlling the release of ammonia from the storage unit and wherein the controller is further configured to control actuation of the valve.

12. A system as set forth in claim 11, wherein the pressure sensor is interconnected between the storage unit and the valve.

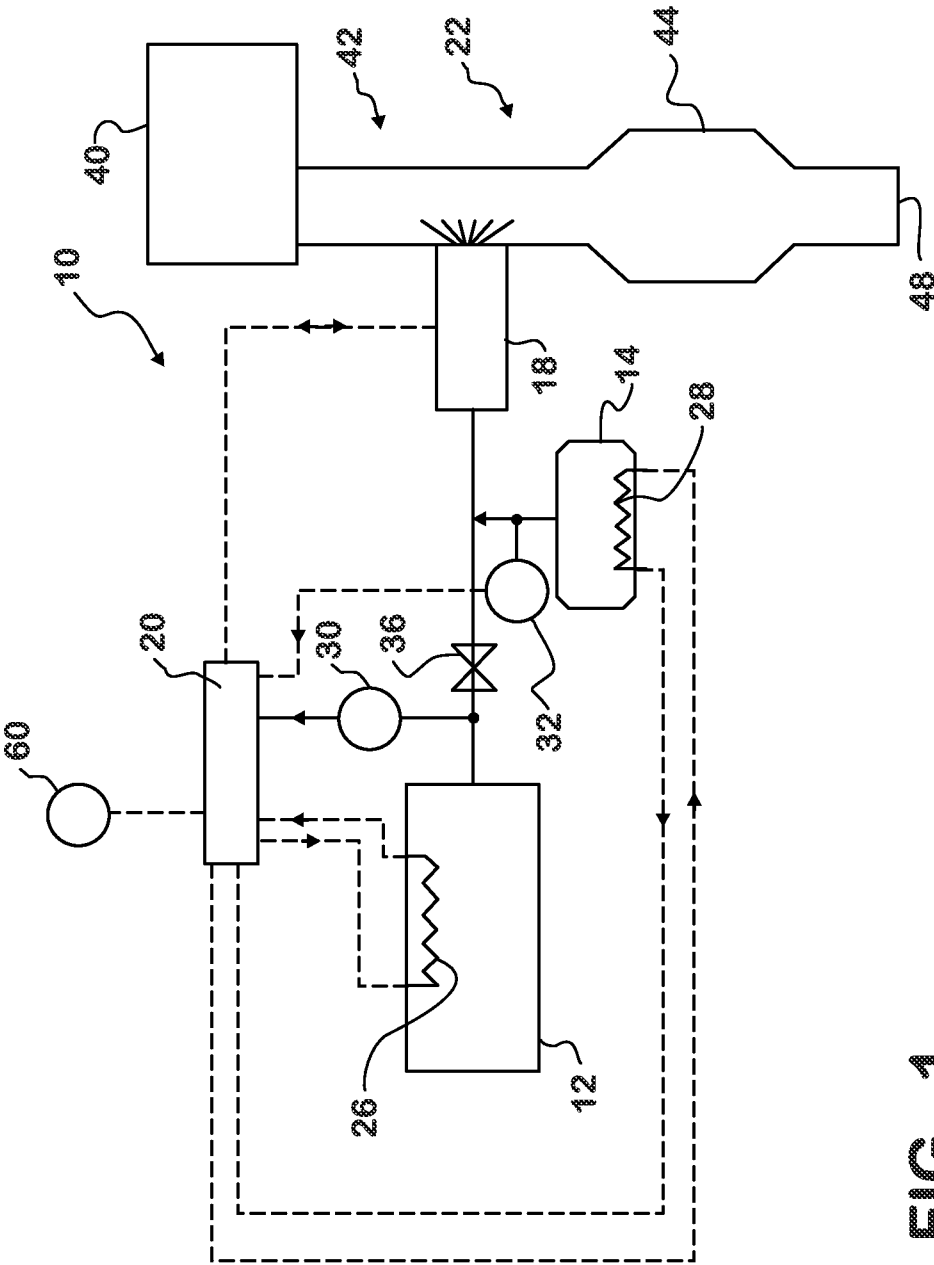


FIG. 1

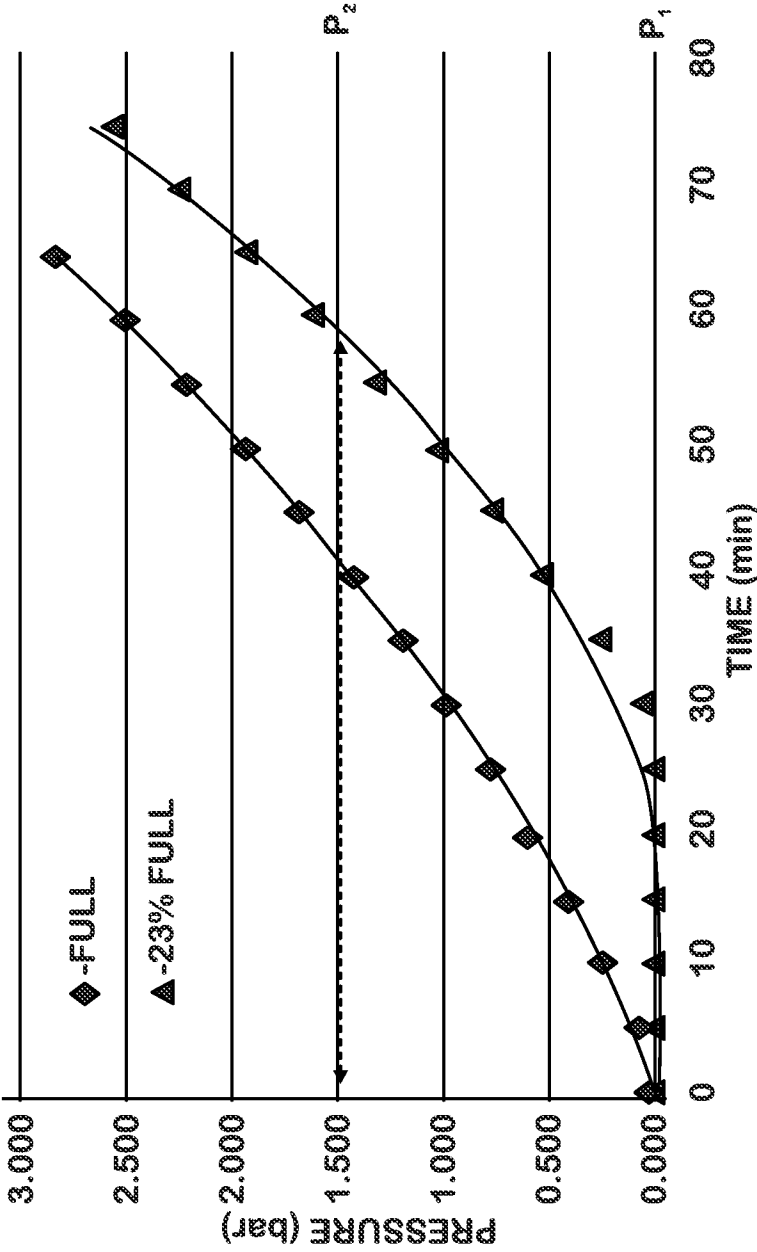
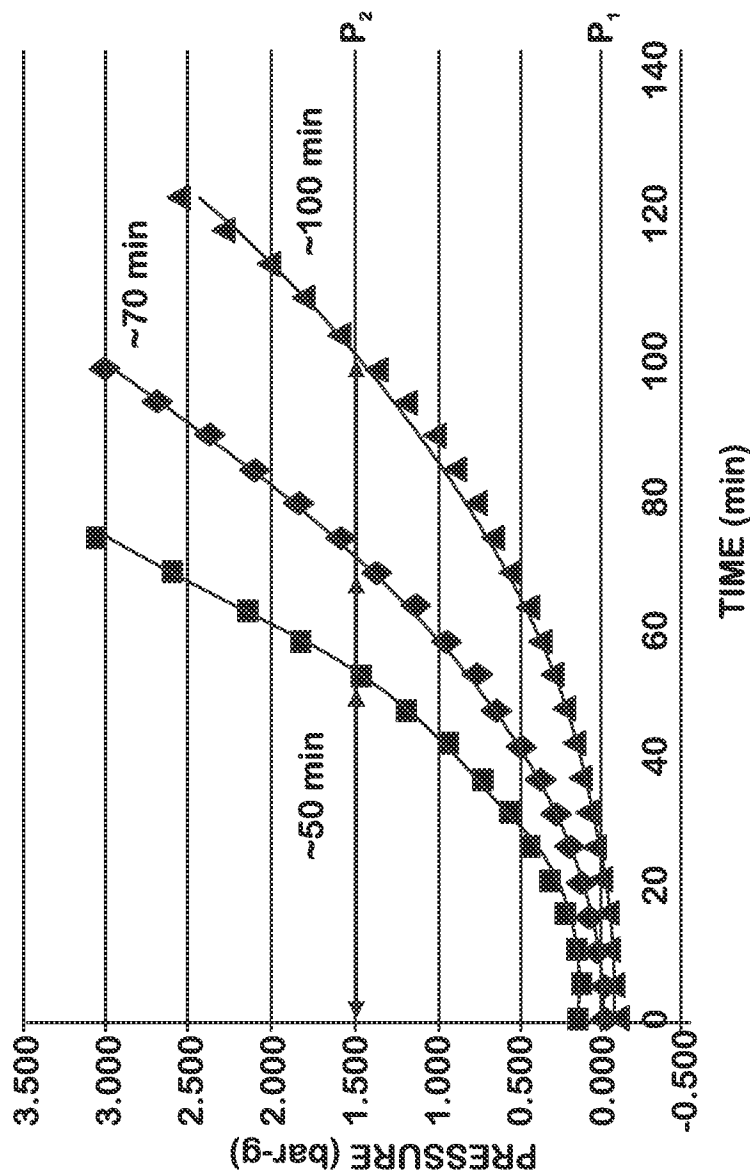


FIG. 2



▲ - 432 WATTS (OVERNIGHT SOAK AT -10°F)
◆ - 540 WATTS (OVERNIGHT SOAK AT -10°F)
■ - 648 WATTS (OVERNIGHT SOAK AT -10°F)

FIG. 3

3/4

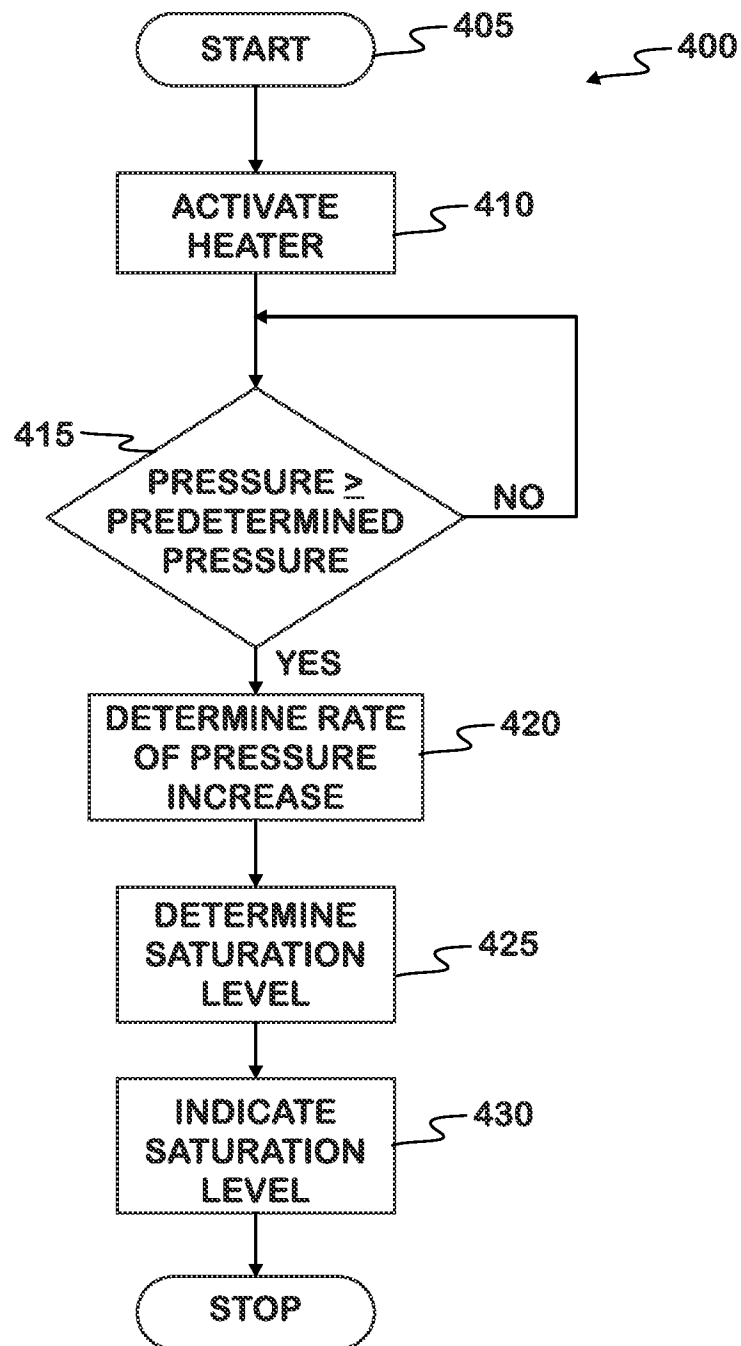


FIG. 4

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2013/042828

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - F01N 3/035 (2013.01)

USPC - 60/295

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC(8) - F01N 3/035 (2013.01)

USPC - 60/295, 300; 96/108; 123/568.21; 423/213.2, 239.1

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
CPC - B01D 2251/2062; C01C 1/006; F01N 3/208 (2013.01)

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

PatBase, Google Patents

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 2 361 883 A1 (JOHANNESSEN et al) 31 August 2011 (31.08.2011) entire document	1-12
A	US 2012/0072135 A1 (QUAADE) 22 March 2012 (22.03.2012) entire document	1-12
A	US 2012/0011830 A1 (GRIFFIN et al) 19 January 2012 (19.01.2012) entire document	1-12
A	US 2010/0047638 A1 (JOHANNESSEN) 25 February 2010 (25.02.2010) entire document	1-12

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

* Special categories of cited documents:

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"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

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"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

"&" document member of the same patent family

Date of the actual completion of the international search

10 October 2013

Date of mailing of the international search report

16 OCT 2013

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