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(54) Title: HYDROGEN GENERATOR CARTRIDGE

(57) Abstract: A fuel cartridge (10) for a hydrogen generating system includes a cartridge containing a substantially anhydrous chemical hydride reactant. A plurality of liquid conduits (21) extends into the cartridge housing (11) and each of the liquid conduits has a length and a plurality of liquid distribution apertures (32) formed along the length. The liquid distribution apertures (32) have a diameter of less than approximately 1 mm. The chemical hydride reactant forms a bed and the liquid conduits extend along at least half of a length of the bed.

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## HYDROGEN GENERATOR CARTRIDGE

The application claims the benefit under 35 U.S.C. §119(e) of U.S. Provisional Applications Serial Nos. 60/627,257 filed 11/12/04, 60/632,460 filed 12/02/04, 60/655,373 filed 02/23/05, 60/683,024 filed 05/20/05, and 60/688,456 filed 06/08/05, each of which is incorporated by reference herein in its entirety.

### Field of Invention

The present invention relates to apparatuses and methods for producing hydrogen. The embodiments described herein relate to apparatuses and methods for releasing hydrogen from chemical hydrides.

### Brief Description of Drawings

Figures 1A and 1B are perspective views of two embodiments of the hydrogen fuel cartridge of the present invention.

Figure 2 is another view of the fuel cartridge similar to that seen in Figure 1, but further including an acid reservoir.

Figures 3A and 3B are perspective views of the front and rear of the front end cap of the fuel cartridge seen in Figure 1.

Figures 4A and 4B illustrate the acid reservoir employed in one embodiment of the present invention.

Figure 5 illustrates a spirally wrapped chemical hydride pouch utilized in one embodiment of the present invention.

Figure 6 illustrates one embodiment of the chemical hydride pouch.

Figure 7 illustrates the placement of the chemical hydride in another embodiment of the present invention.

Figure 8 illustrates one embodiment of the cartridge receiver plate.

Figure 9 illustrates one embodiment of the receiver assembly.

Figure 10 is a schematic diagram of one embodiment of the hydrogen generation system of the present invention.

Figure 11 is a state diagram illustrating the operation of one embodiment of the hydrogen generation system of the present invention.

Figure 12 is a schematic diagram illustrating the use of liquid control valves to direct liquid into the injection tubes.

Figure 13 is a partial schematic diagram illustrating modifications made to Figure 10 when an acid reservoir is employed.

Figure 14 is a perspective view of a further embodiment of the hydrogen fuel cartridge of the present invention.

#### Detailed Description of Invention

Figure 1A illustrates one embodiment of a hydrogen fuel cartridge which may be employed in the hydrogen generation system of the present invention. Fuel cartridge 10 may include a tubular body or housing 11, which in this embodiment ranges from 1 to 3 inches in diameter and 4 to 8 inches in length. Naturally, housing 11 is not limited to any particular cross-sectional shape or any particular dimensions. In one preferred embodiment, housing 11 is formed of a material such as aluminum which has sufficient strength, is comparatively light, and has good heat transfer characteristics. However, many substitute materials will be readily apparent to those skilled in the art, including steel, stainless steel, copper, carbon fiber epoxy composites, fiberglass epoxy composites, PEEK, polysulfone derivatives, polypropylene, PVC, or other suitable materials. Figure 1A's embodiment also illustrates a rear end cap 12 having a handle 13 allowing fuel cartridge 10 to be easily positioned and locked into place with other components of the overall hydrogen generation system as will be described below.

The other end of housing 11 opposite rear end cap 12 will comprise a front end cap 15 which is more clearly seen in Figures 2 and 3. This embodiment of front end cap 15 will include a plurality of liquid reactant (e.g., water) inlet ports 21a-21d, a hydrogen gas outlet port 20, alignment structures 24 and a threaded center aperture 23. In certain embodiments described herein, the liquid reactant may be water or another aqueous reactant (e.g., a water based acid solution). For simplicity, liquid reactant inlet ports 21 may be referred to as water inlet ports 21 when describing certain embodiments. In the embodiment of Figures 1-3, alignment structures 24 are circular apertures which engage a cartridge receiver or in one embodiment, the receiver plate 40 as seen in Figure 8 (discussed below). In this embodiment, alignment structures 24 are non-symmetrically positioned around center aperture 23 to ensure end cap 15 can only engage receiver plate 40 in one orientation. Of course, other embodiments could include different numbers of water inlet ports 21 and hydrogen outlet ports 20, along with different types of alignment structures. In the illustrated embodiment, front end cap 15 will include a groove 25, which will allow end cap 15 to engage and be locked into

housing 11 by way of the front end of housing 11 being crimped into the groove 25 (or by any other method forming a substantially gas tight seal).

As best seen in Figure 3B, the rear side of front end cap 15 will include a support assemblage 17 formed of structural elements having tube connectors 26 which secure liquid reactant injection tubes 30 (see Figure 1A) to front end cap 15 and provide continuous passages between injection tubes 30 and ports 21. In one embodiment, injection tubes 30 are fixed to end cap 15 in a manner which substantially prevents tubes 30 from moving relative to end cap 15 along an axis parallel to the length of cartridge housing 11. Another support assemblage 17 will provide space for a check valve cavity 22a (the rear side of hydrogen outlet port 20) to hold an integrated check valve 22 allowing gas flow only in the outward direction, i.e., from the interior to the exterior of cartridge 10. In the preferred embodiment, the end cap is made of molded plastic, e.g., Delrin<sup>®</sup>, but one skilled in the art will see that end cap 15 could be made from a variety of materials, including aluminum, steel, stainless steel, copper, other plastics or composite materials. In the illustrated embodiment, the check valve may be a silicone duckbill type valve manufactured by Vernay Laboratories, Inc. of Yellow Springs, OH. As suggested in Figure 1A, fluid injection tubes 30 will extend into the interior of cartridge housing 11 which holds a solid reactant (as explained in more detail below). In one embodiment, injection tubes 30 may extend into housing 11 at least half housing 11's length, although in other embodiments the injection tubes 30 may extend less than half the housing's length.

In one preferred embodiment, water injection tubes 30 will have an inside diameter ranging from about 0.5 to 5.0 mm with the inside diameter more preferably being about 1 mm. The injection tubes may be made of aluminum, brass, or other metal, PTFE, Nylon<sup>®</sup>, Delrin<sup>®</sup>, or other high temperature polymers. As suggested in Figure 1A, a series of liquid distribution apertures 32 will be formed along the length of water injection tubes 30. In one embodiment, the liquid distribution apertures 32 will have a diameter which is no greater than about 1/5 the inside diameter of water injection tubes 30, and more preferably no greater than about 1/10 such inside diameter (e.g., on the order of about 50 to 1000 microns). Liquid distribution apertures 32 may be spaced in any manner to promote uniform liquid distribution, such as for example, every half inch on opposing sides of injection tubes 30. In certain embodiments, liquid distribution apertures may be spaced more closely along the length of tubes 30 as the tubes extend away from front end cap 15 in order to compensate for head loss in the fluid as it travels down the length of injection tube 30. Alternatively, the aperture size could be increased as the apertures become further from front end cap 15 in order to compensate for head loss.

A further embodiment seen in Figure 1B would eliminate the multiple apertures 32 along the length of the tubes and have only one injection aperture 32 at the open end of the tubes (although this single aperture could be elsewhere along the length of the tubes). The length of the different tubes 30 could vary from the endcap 15 in order to distribute liquid reactant throughout the housing. The above described alternative embodiments along with other unspecified alternative embodiments may be employed to distribute liquid reactant at different locations within housing 11.

As suggested above, one embodiment of fuel cartridge 10 will contain a solid reactant such as an anhydrous chemical hydride. In certain embodiments, a chemical hydride may be considered a reducing compound containing a metal and hydrogen that generates hydrogen gas when it reacts with water or other oxidizing agents. Various examples of chemical hydrides are disclosed in US application serial no. 10/459,991 filed June 11, 2003 which is incorporated by reference herein. Nonlimiting examples of chemical hydrides may include sodium borohydride, lithium borohydride, lithium aluminum hydride, lithium hydride, sodium hydride, and calcium hydride. In the embodiment seen in Figure 5, the chemical hydride reactant will be enclosed within a fabric pouch 31. As used herein, "fabric" includes not only textile materials, but also includes paper based porous materials typically used for filtration purposes. One embodiment of the fabric will be a porous material which can maintain structural integrity at temperatures ranging from about -20 °C to about 200 °C, a pH ranging from about 4 to about 14, and which exhibits at least some wicking potential. Preferred fabrics may include woven Nylon, Rayon polyester blend (for example Pellon 30 manufactured by Pellon Consumer Products of Tucker, GA) or porous filter paper such as Paper 602 provided by National Filter Media Corporation, Salt Lake City, UT. The wicking potential may be measured in distance water wicks divided by time. Illustrative examples include paper 603 - 6" in 60 min., Pellon 30 - 1.8" in 60 min., Nylon - 1.2" in 60 min., coffee filter paper - 2.3" in 60 min. In certain embodiments, a wicking potential of between 0.1 and 10 inches a minute may be employed; other embodiments could be between 0.5 and 3 inches a minute. In one embodiment, the thickness of the fabric or paper could be from about 3 mil to 12 mil, while the pore size of the fabric or paper could be between at least 1 micron and about 50 microns. Naturally, the present invention may include wicking potentials, fabric thicknesses, and pore sizes different from those listed above.

In the embodiment of Figure 5, the fabric pouch 31 is comparatively thin having a substantially greater area than thickness. Pouch 31 may be formed in any conventional manner. For example, viewing Figure 6, it can be seen how two rectangular sheets of fabric material 33a and 33b may be sealed along three edges (for example by stitching 34) and segmented into 1 to 2 inch sections 36 (also by stitching) to leave open ends 35. The series of sections 36 thus formed are filled

with a fine grain chemical hydride (described below) and sealed along the fourth edge by stitching closed open ends 35. An illustrative thickness of such a pouch 31 (i.e., the thickness of sections 36 when unrolled and charged with a chemical hydride) may be approximately  $\frac{1}{4}$  of an inch in one embodiment and its unrolled dimensions could be approximately 5.75 inches by 20 inches. Then the pouch 31 is rolled to a diameter sufficiently small to be inserted into tubular housing 11 as suggested in Figure 5 (the front end cap 15 has been removed for purposes of clarity). The water injection tubes 30 are then carefully inserted between overlapping layers of the rolled pouch 31. Figures 1 and 3A illustrate how in one embodiment, water ports 21 (and thus injection tubes 30) may be arranged in a nonuniform configuration such as a spiral pattern. For example, water port 21a in Figure 3A is closest to the center point of end cap 15. Then water port 21b is radially spaced further from the center point, with water port 21c spaced even further and water port 21d spaced still further. In this manner, water injection tubes 30 may follow the spiral pattern of rolled pouch 31. However, other embodiments could utilize different orientations of pouch 31 or could use a series of smaller pouches 31 as opposed to the continuous pouch 31 seen in Figure 6 and could use any arrangement of injection tubes 30. In one embodiment, the components within cartridge 10 will have a temperature stability between at least about  $-20\text{ }^{\circ}\text{C}$  to at least about  $200\text{ }^{\circ}\text{C}$  and a corrosion stability/resistance at pHs ranging from about 4 to about 14.

An alternate embodiment of cartridge 10 is seen in Figure 7. In this embodiment, the chemical hydride material 38 is not positioned within a pouch, but is placed directly within the interior space of cartridge housing 11 (seen with end cap 15 removed) and water injection tubes 30 will extend into the bed of chemical hydride material 38. However, in this embodiment water injection tubes 30 will be covered with a thin sleeve 37 of fabric such as a woven refractory material which in one embodiment is a 10 mil thick fabric sold under the tradename Siltex<sup>®</sup> manufactured by Mid Mountain Materials of Mercer Island, WA. Naturally end cap 15 seen in Figures 1-3 would be modified to have two liquid injection ports 21 to match the two injection tubes 30 seen in Figure 7. Although not explicitly shown in the drawings, when chemical hydride material 38 is placed directly in cartridge housing 11, it may also be advantageous to place a 0.125 inch thick layer of a porous carbon fiber felt material between the hydrogen outlet and hydride material in order to filter the exiting hydrogen gas.

A still further embodiment of cartridge 10 is seen in Figure 14. In this embodiment the end cap 15 is similar to that described above. However, a single injection tube 30 extends into housing 11 toward the bottom of a bed of chemical hydride material. The injection tube 30 will have an aperture at the end of the tube. The cartridge 10 may be oriented vertically such that water injected

into the hydride bed will react first with the hydride material at the bottom of the bed. As additional water is injected, the water will rise and activate hydride material along the length of cartridge 10. In other variations, the cartridge 10 may be in non-vertical orientations. The term "bed" of chemical hydride material may mean a mass of loose hydride material placed directly in the cartridge 10 (e.g., Figure 7) or the hydride material being in a pouch (e.g., Figure 5) which is inserted in cartridge 10 or any other manner of (or container for) positioning the hydride material in cartridge 10.

As suggested above, Figure 8 illustrates one embodiment of receiver plate 40. Receiver plate 40 will include a receiver body 41 which has various structures formed thereon and within for mating with end cap 15 of cartridge 10. Within the receiver body 41 are alignment structures or guide pins 45 which will mate with alignment structures 24 on end cap 15. Additionally, receiver plate 40 includes a plurality of port connectors 44 which will mate with hydrogen and water ports 20 and 21 on end cap 15. Indentations 49 around port connectors 44 will accommodate sealing devices such as O-rings or gaskets. A port connector 44 will communicate with each hydrogen passage 42 and water passages 43 which is formed through receiver body 41. It will be understood that external water and hydrogen lines (see schematic in Figure 10) will connect (directly or indirectly depending on the embodiment) to hydrogen passage 42 and water passages 43 and thereby provide fluid communication through receiver plate 40 to the hydrogen port 20 and water ports 21 of cartridge 10. Figure 8 illustrates how one embodiment of receiver plate 40 will include internal water passages 46 and various inlets and outlets 54 for passages 46. Figure 9 illustrates how the openings in water passages 46 will communicate with electronic control valves 77 and hose fittings 53. In the embodiment shown, hose fittings 53 are 90 degree, 1/8 inch I.D. hose barb fittings supplied by Clippard Instruments Laboratory, Inc. of Cincinnati, OH and control valves 77 are x-valves available from Parker Hannifan Corporation located in Cleveland HO.

In one embodiment, a plurality of hoses (not shown) will connect the plurality of water passages 46 (via hose fittings 53) in the receiver plate 40 to water passages 43, likewise equipped with hose fittings. In other embodiments, the passages 46 may connect directly to passages 43 through the internal volume of receiver plate 40, but forming long internal passages within receiver plate 40 adds substantial manufacturing complexity. The layout of fluid pathways and control valves in the receiver block of Figures 8 and 9 is described schematically in Figure 12. A fluid pump 78 will direct fluid toward a series of three control valves 77a, 77b, and 77c through hose 50. Control valves 57 will have two orientations, an unactivated or normal open (NO) position and an activated or normal closed position (NC). It can be seen that control valves 77a, 77b, and 77c are connected in series by fluid pathway 58 (which would be the combination of hoses and passages if embodied in

the receiver plate 40 of Figures 8 and 9). Control valves 77a and 77b will direct fluid to pathway 58 in the NO position and to their respective fluid injection tubes 30 when energized to the NC position by the associated control circuitry. Control valve 77c is connected somewhat differently since in the NO position, fluid is directed to injection tube 30d and in the NC position fluid is directed to injection tube 30c. The operation of control valves 77a to 77c in selectively directing fluid to different injection tubes will be readily apparent. To direct fluid to injection tube 30d, all valves 77a to 77c are left in the NO position; for injection tube 30c, valve 77c is moved to the NC position; for injection tube 30b, valve 77a will be in the NO position and valve 77b in the NC position; and for injection tube 30a, valve 77a will be in the NC position. In this manner, the path of fluid flow will pass through control valves 77 and allow these valves to control fluid delivered to injection tubes 30.

The receiver plate 40 seen in Figure 8 will connect to end cap 15 (see Figure 3A) by way of a holding assembly, one example of which is a bolt or other threaded member passing through aperture 48 in receiver plate 40 and engaging the threaded inner surface of center aperture 23 on end cap 15 in order to pull end cap 15 firmly against receiver plate 40 and form seals between hydrogen/water ports 20/21 and port connectors 44. Figure 9 shows the complete receiver assembly 28 including support plate 56 and connecting rods 51 engaging rod apertures 50 in receiver plate 40. In the embodiment shown, one rod aperture 50 will be elongated and the others round to assist in orienting the rods 51 for insertion into receiver plate 40. The cartridge 10 slides through the opening in support plate 56 and in between the connecting rods 51 and seals against the receiver plate 40 as described previously with the threaded end of knob 52 extending through aperture 48. A mounting arm 57 will extend from support plate 56 and rest against cartridge 10. A cartridge temperature sensor described herein will be attached to mounting arm 57. In the embodiment of Figure 9, the electronic valves 77 are shown connected to the receiver plate 40. Slot 47 in the receiver plate 40 shown in Figure 8 firmly holds the cartridge sense switch 82 as shown in Figure 9. In one embodiment, cartridge sense switch 82 is ZM series microswitch with lever manufactured by Honeywell International, Inc. of Morristown, NJ and will detect when cartridge 10 is in direct or near contact with sense switch 82.

In one embodiment of the present invention, the chemical hydride reactant utilized in the fuel cartridge may be a dry, powdered form of sodium borohydride ( $\text{NaBH}_4$ ) mixed with an activating agent. The  $\text{NaBH}_4$  is particularly suitable for use in the pouch 31 seen in Figure 5 and in one embodiment; the  $\text{NaBH}_4$  will have a grain size ranging from about mesh 10 to about mesh 1000. It is preferred that the activating agent be a powdered solid when mixed with  $\text{NaBH}_4$ , since solids tend to react very slowly with each other. However, in alternative embodiments, the activating agent could



also be mixed into an organic/oil solvent. In addition, the activating agent in certain embodiments is preferably water soluble to increase its effectiveness, since the greater its solubility, the greater its potential to activate the water/ $\text{NaBH}_4$  reaction.

One effective activating material is magnesium chloride ( $\text{MgCl}_2$ ), since it is relatively lightweight, low cost, and strongly activating. Other potential activating agents are other salts of Group IIA (alkaline earth metals) or Group VIIB (halides), such as  $\text{BeF}_2$ ,  $\text{BeCl}_2$ ,  $\text{BeBr}_2$ ,  $\text{BeI}_2$ ,  $\text{MgF}_2$ ,  $\text{MgBr}_2$ ,  $\text{MgI}_2$ ,  $\text{CaF}_2$ ,  $\text{CaCl}_2$ ,  $\text{CaBr}_2$ , and  $\text{CaI}_2$ . The fluorides and chlorides are preferred because they have a lower molecular weight. However, some of these salts may be less preferred depending on their degree of solubility in water or if they are considered toxic (e.g., beryllium compounds).

Activating agents may also include other water soluble salts such as Group IA (alkali metals) salts including  $\text{LiF}$ ,  $\text{LiCl}$ ,  $\text{LiBr}$ ,  $\text{LiI}$ ,  $\text{NaF}$ ,  $\text{NaCl}$ ,  $\text{NaBr}$ ,  $\text{NaI}$ ,  $\text{KF}$ ,  $\text{KCl}$ ,  $\text{KBr}$ , and  $\text{KI}$ . Group IA and Group IIA hydroxides may be less preferred, since they make basic solutions in water and thus reduce the reaction rate. Group IA and Group IIA oxides may also be less preferred since they tend to be more stable and thus not as reactive. However, Group IA and Group IIA sulfides and selenides, such as  $\text{Li}_2\text{S}$ ,  $\text{Li}_2\text{Se}$ , may be better activating agents if they are sufficiently water soluble. In one preferred embodiment, the activating agents will be from the group of  $\text{MgCl}_2$ ,  $\text{BeCl}_2$ ,  $\text{CuCl}_2$ ,  $\text{LiCl}$ ,  $\text{NaCl}$ , or  $\text{KCl}$ . However, any of the above activating agents (or others not listed) could possibly be employed given the proper design and use conditions. In certain embodiments, the activating agent will have a grain size ranging from about mesh 10 to about mesh 1000.

In one preferred embodiment, the quantity of activating agent mixed with the chemical hydride will be from about 25 to about 65 weight percent and more preferably about 50 to about 60 weight percent. In one embodiment, the quantity of activating agent is 55 weight percent. In the embodiment where the solid reactant is 55 weight percent  $\text{MgCl}_2$ , approximately 0.8 gm of water will be required to fully react each gm of solid reactant. One consideration in optimizing the amount of activating agent is determining the minimum amount of the material which gives the desired hydrogen generation rate and results in complete reaction/utilization of the  $\text{NaBH}_4$ . For a 55 weight%  $\text{MgCl}_2/\text{NaBH}_4$  mixture, the energy density is 3116 Whr/kg. For comparison, the energy density of a 20 weight%  $\text{NaBH}_4/\text{H}_2\text{O}$  mixture (i.e.,  $\text{NaBH}_4$  dissolved in water) is 1066 Whr/kg, while the energy density for pure  $\text{NaBH}_4$  is 7101 Whr/kg.

An alternative activating agent may be an anhydrous or powdered acids such as boric acid ( $\text{H}_3\text{BO}_3$ ), oxalic acid, tartaric acid, etc. Such anhydrous acids can be mixed with the  $\text{NaBH}_4$  without reaction, but when water is added, the anhydrous acid dissolves and thus causes a reaction. Weak or relatively insoluble anhydrous acids such as boric acid when mixed with  $\text{NaBH}_4$  produce hydrogen

in the presence of water at a relatively low rate, and thus are less preferred. Strong acids such as oxalic acid are very soluble in water and generate substantial hydrogen when mixed with  $\text{NaBH}_4$ . However, this mixture is difficult to controllable and is also less preferred. However, intermediate strength acids, such as tartaric acid or benzoic acid are more favorable. In one preferred embodiment, the strength ( $K_a$ ) of the dry acid will range from about  $1 \times 10^{-4}$  to about  $1 \times 10^{-11}$ . In certain embodiments, the powdered acid will have a grain size ranging from about mesh 10 to about mesh 1000. In one preferred embodiment, the quantity of tartaric acid mixed with  $\text{NaBH}_4$  will be from about 5 to about 50 weight percent and more preferably about 8 to about 12 weight percent. In this embodiment, approximately 0.8 gm of water will be required to fully react each gm of solid reactant.

As a further alternative, an inexpensive, water-insoluble catalyst may be mixed with the  $\text{NaBH}_4$ . The catalyst can act to accelerate the water/ $\text{NaBH}_4$  reaction as water is injected. Such metal catalyst could include Co, Ni, Cu, Pt, Pd, Fe, Ru, Mn, and Cr. Typically, the metal catalyst will be in a powder form (e.g., particles less than 25  $\mu\text{m}$ ) and will be added to the chemical hydride in an amount of about 25 weight percent to about 65 weight percent. In this embodiment, approximately 0.8 gm of water will be required to fully react each gram of solid reactant.

A still further alternative to mixing an anhydrous activating agent with the  $\text{NaBH}_4$  may be to mix the water soluble activating agent in with the water before it is injected into the cartridge containing a bed of anhydrous  $\text{NaBH}_4$  or other metal hydride. This has the advantage that an aqueous substance such as hydrochloric acid (HCl) may be used. In this embodiment, the activating material is held in separate container or reservoir 60 such as seen in Figure 2. This container may be attached to the cartridge housing 11 but could be detached in other embodiments. Figure 2 illustrates reservoir 60 connected to housing 11 by way of strap 61. In one embodiment, strap 61 will be formed of aluminum, stainless steel, or composite polymer material in order to hold reservoir 60 in rigid orientation with housing 11. The liquid contents of reservoir 60 will be withdrawn through reservoir port 64 and be directed into liquid injection tubes 30 via liquid passages 43 in receiver plate 40. Although not actually shown in the drawings, it can be envisioned how one embodiment of receiver plate 40 may be modified such that it will have a port mating with reservoir port 64 and the contents of reservoir 60 will be directed to receiver plate 40 in the same manner as water described above. In one embodiment seen in Figures 4A and 4B, reservoir 60 includes a plunger 62 positioned therein. Plunger 62 will move toward port 64 as fluid is removed from reservoir 60. Vent hole 63 prevents a vacuum from forming behind plunger 62 and resisting its movement toward port 64. The plunger is moved forward by pump 78 (see Figure 13) applying suction to port 64.

Returning to the example acid listed above, concentrated HCl acid (38 weight%) could be mixed into the water to give a concentration of 28 weight%. When this solution is controllably injected into the NaBH<sub>4</sub>, it reacts readily to generate hydrogen. At this acid concentration (28 weight%), approximately 0.4 gm of acid solution will be required to fully react each gm of NaBH<sub>4</sub>. It is possible to control the reaction rate by controlling the concentration of acid in the water in addition to the acid solution injection rate. It is also possible to mix water soluble salts, such as MgCl<sub>2</sub> into the water and then inject the mixture into the bed of NaBH<sub>4</sub>. Nor is the acid containing liquid necessarily limited to aqueous solutions. In one preferred embodiment, the aqueous solution injected into the bed of NaBH<sub>4</sub> will have a pH ranging from about 1 to about 6 and more preferably a pH of about 2. In addition to HCl, other suitable acids could include (but are not limited to) nitric acid, sulfuric acid, acetic acid, hydrofluoric acid, hydrobromic acid, carbonic acid, etc., or mixtures thereof.

Although NaBH<sub>4</sub> is mainly discussed above, alternative chemical hydrides may include (but are not limited to) lithium borohydride, lithium aluminum hydride, lithium hydride, sodium hydride, and calcium hydride. In certain embodiments, these latter chemical hydrides need not be combined with a powdered activating agent as described above and may be activated with water alone. In one preferred embodiment, these latter chemical hydrides will be utilized in a cartridge such as seen in Figure 7.

Fuel cartridges such as those described above will typically be employed in a hydrogen generation system. One embodiment of such a hydrogen generation system is shown schematically in Figure 10. This hydrogen generation system 1 will generally comprise a fuel cartridge 10 connected to receiver plate 40 with a liquid reactant (e.g., water) line 79 supplying water to fuel cartridge 10. A water pump 78 controlled by control system 75 will provide a carefully metered amount of water to fuel cartridge 10. In one preferred embodiment, control system 75 consists of a micro-processor and related control circuitry such as a PIC microcontroller 16F877A. Control system 75 will also operate cooling fans 81, switching valves 77, and transfer valve 84. Moreover, control system 75 will receive data on system parameters from temperature sensor 80, cartridge sensor 82, and hydrogen pressure sensor 89. In one embodiment, temperature sensor 80 is mounted against the external skin of aluminum housing 11. To prevent hydrogen from escaping from the system when the cartridge is removed, a check valve 87 is incorporated into the hydrogen line between the receiver plate and the hydrogen trap 83.

Hydrogen gas exiting cartridge 10 will flow through a check valve 87 and a hydrogen filter/water trap 83 before being directed to a fuel cell or other device to which hydrogen is to be

supplied. Filter/water trap 83 serves the dual purpose of filtering particulate out of the hydrogen and also removing excess moisture from the hydrogen gas. A water condenser/reservoir 85 will collect water from any moist air returned from the fuel cell or other hydrogen consuming device and will also store water collected from water trap 83 and transferred via transfer valve 84.

In operation, control system 75 will determine the volume of water to pump into fuel cartridge 10 based upon monitoring parameters such as the temperature of the chemical hydride (as indicated by temperature sensor 80) and the hydrogen pressure within the system as measured by pressure sensor 89. As hydrogen pressure drops below a predetermined level in system 1, water pump 78 will be activated to deliver water to fuel cartridge 10, thereby causing the chemical hydride in cartridge 10 to release additional hydrogen gas. In one preferred embodiment, switching valves 77 will be individually controlled by control system 75 as described above. This allows pump 78 to deliver water through only one water injection tube 30 at a time and to sequentially deliver water to each injection tube 30. This sequential method of delivering water may in some instances provide a more uniform distribution of water than if all water injection tubes were simply manifolded together without individual control of water flow to each injection tube 30. Likewise, the temperature sensor 80 monitoring the temperature of the chemical hydride will allow control system 75 to make decisions regarding whether fans 81 should be turned on to cool cartridge 10 or whether water should be limited to slow down the reaction rate of the chemical hydride. Hydrogen generation system 1 may also include the cartridge sensor 82 which will signal control system 75 as to whether a fuel cartridge 10 is presently installed in the system and will also provide control system 75 with information concerning when a spent cartridge has been removed and a new, fully charged cartridge installed.

As hydrogen gas flows through filter/water trap 83, excess moisture in the hydrogen gas will be removed and when a sufficient amount of water has accumulated, will be transferred via transfer valve 84 to water condenser/reservoir 85. Hydrogen gas exiting filter/water trap 83 will be directed through line 90 to the particular hydrogen consuming device, which for illustrative purposes will be considered a fuel cell in the present description. Typically, a regulator 88 will be positioned in line 90 to assure the fuel cell is supplied with hydrogen at a constant pressure. If the hydrogen consuming device produces water vapor as a by-product (as do fuel cells), the moist air will be directed via line 86 back to condenser 85 and the water recovered from the air. Likewise, water vapor in the hydrogen passing through purge line 91 (another characteristic feature of fuel cells) will be recovered in condenser 85.

When an embodiment having acid reservoir 60 is employed, a portion of the schematic seen in Figure 10 may be modified as suggested in Figure 13. In this embodiment, a metering valve 95 will be positioned between pump 78 and acid reservoir 60 while a metering valve 94 is positioned between pump 78 and water reservoir 85. In one embodiment, metering valves 94 and 95 may be stainless steel adjustable needle-type valves such as provided by Swagelock Company of Solon, OH. Using these types of valves, the amount of acid and water drawn upon each activation of pump 78 is preset.

In another embodiment of the present invention, the injection switching valves 77 seen in Figure 10 can be replaced with a manifold 95 (illustrated in the alternative with dashed lines in Figure 13). In this case, fluid from the injection pump 78 is split equally between the multiple injection tubes 30 which are connected in parallel. Parallel injection tubes are preferably very uniform with tight tolerances so that the pressure drop along the length of each injection tube is the same.

Figure 10 is a state diagram 100 illustrating the controller logic which could be utilized in one embodiment of the present invention. Beginning at an idle state (block 101), control system 75 monitors the cartridge temperature, the cartridge in/out status, the hydrogen demand status (e.g., does the fuel cell currently require hydrogen), and the pump/valves status. Presuming the use of a bi-color LED indicator (e.g., red/green), the idle state could be indicated by all LED illumination being off. When control system 75 is signaled that hydrogen is required, the system will enter state 1 (block 102). Control system 75 will signal pump 78 to inject a predetermined amount or "pulse" of water (e.g., one cycle of pump 78) toward switching valves 78. Control system 75 will determine which injection tube 30 received the last water pulse and then open or shut the appropriate valves 77 to ensure the current water pulse is directed to the desired injection tube 30. As alluded to above, the water pulses will typically be directed in sequence to the various injection tubes 30 to evenly distribute water throughout the hydride bed. Additionally, control system 75 will monitor the total number of pulses (i.e., total volume of water) injected into cartridge 10. Since the cartridge has a known amount of chemical hydride, the volume of water needed to completely react with that weight of chemical hydride may be calculated. By tracking the volume of water injected into cartridge 10, control system 75 may accurately estimate when all the chemical hydride in cartridge 10 has been reacted with water and thus when cartridge 10 should be considered exhausted or spent. An LED response (e.g., flashing red/green) may then indicate when the amount of unreacted chemical hydride in cartridge 10 is becoming low and the current cartridge 10 should be replaced.

State 2 (block 103) will monitor the increased pressure of hydrogen due to the injection of a water pulse. If the current hydrogen pressure is less than the desired hydrogen pressure (or the hydrogen pressure appears to be falling), the control system will return to state 1 and signal that pump 78 is to injection another pulse of water. If the cartridge temperature exceeds a predetermined value, the system will activate fans 81. If the temperature continues to increase above a higher predetermined value, then the system will enter state 3 (indicated by a red LED) which will cease injection of further water until the cartridge temperature returns to the specified operating range. State 7 (block 104) will monitor when water should be transferred from filter/trap 83 to reservoir 85 and accordingly activate transfer valve 84.

When the total volume of water injected into cartridge 10 is sufficient to react approximately the total weight of chemical hydride in cartridge 10, state 6 (block 106) will be entered indicating with a flashing red LED that cartridge 10 is exhausted and needs to be replaced. While the spent cartridge is removed, state 4 (block 108) will indicate with a red LED that no cartridge is present in the system. Once a new cartridge 10 is inserted into the system, state 5 (block 107) resets the cartridge life counter such that state 1 may once again begin counting pulses of water injected into the new cartridge 10 in order to monitor the remaining life of the new cartridge once the system resumes generation of hydrogen. Naturally, the state diagram of Figure 10 is simply one version of control system 75's operation and many alternate sequences of operation may be employed in other embodiments of the invention.

Although the present invention has been described in terms of selected specific embodiments, those skilled in the art will recognize many modifications and variations of the invention. All such modifications and variations should be considered within the scope of the following claims.

## I Claim:

1. A hydrogen generation system comprising:
  - a. at least one cartridge comprising at least one liquid inlet port and at least one gas outlet port and containing a substantially anhydrous hydride reactant;
  - b. at least one liquid conduit extending into said cartridge, said liquid conduit having at least one liquid distribution aperture formed thereon;
  - c. a cartridge receiver removably engaging said cartridge, said cartridge receiver comprising passages corresponding to said gas outlet port and said liquid inlet port on said cartridge; and
  - d. a liquid reactant injection control system operatively connected to said cartridge receiver, said control system reading at least one reaction parameter and adjusting an amount of liquid reactant injected from said distribution aperture based upon said at least one reactant parameter.
2. The hydrogen generation system according to claim 1, further comprising a plurality of distribution apertures formed along said liquid conduit, said liquid distribution apertures having a diameter between about 50 and about 1000 microns.
3. The hydrogen generation system according to claim 1, wherein said hydride reactant forms a bed and said liquid conduit extends along at least half of a length of said bed.
4. The hydrogen generation system according to claim 1, wherein said reaction parameter comprises a hydrogen pressure within said cartridge.
5. The hydrogen generation system according to claim 1, wherein said liquid reactant is an aqueous reactant.
6. The hydrogen generation system according to claim 5, wherein each of said liquid conduits has a separate control valve.
7. The hydrogen generation system according to claim 1, wherein said reaction parameter comprises a temperature of said cartridge and said control system activates a cooling system based upon said temperature.
8. The hydrogen generation system according to claim 7, wherein said cooling system comprises at least one fan generating an air flow across said cartridge.
9. The hydrogen generation system according to claim 1, wherein said hydride is enclosed in a fabric pouch having a substantially greater area than thickness.
10. The hydrogen generation system according to claim 9, wherein said fabric pouch is rolled within said cartridge and said liquid reactant conduit extends between folds of said fabric pouch.
11. The hydrogen generation system according to claim 1, wherein said fabric pouch comprises at least one chosen from woven, Pellon 30, Nylon sheet or porous paper.

12. The hydrogen generation system according to claim 11, wherein said fabric maintains a structural integrity at temperatures ranging from about -20 °C to about 200 °C, a pH ranging from about 4 to about 14, and which exhibits at least some wicking potential.
13. The hydrogen generation system according to claim 1, wherein said hydride reactant is lithium aluminum hydride and the amount of liquid reactant exiting a distribution aperture does not exceed about 50 uL per injection cycle and the cartridge temperature does not exceed 80 C.
14. The hydrogen generation system according to claim 1, further comprising an aqueous reactant having a pH of between about 1 and about 6.
15. The hydrogen generation system according to claim 1, wherein said liquid reactant is an aqueous acid solution.
16. The hydrogen generation system according to claim 1, further including an acid reservoir and a water reservoir, wherein acid and water are mixed prior to being injected from said distribution apertures.
17. The hydrogen generation system according to claim 1, wherein said cartridge receiver is a receiver plate having hydrogen and liquid inlet ports mating with said ports of said cartridge.
18. The hydrogen generation system according to claim 16, wherein a sealing device is positioned between said ports of said cartridge and said receiver plate; and a holding assembly brings said cartridge and said receiver plate together with sufficient force to form an effective seal.
19. The hydrogen generation system according to claim 17, wherein a receiver assembly comprises a support plate attached to said receiver plate by at least two support rods.
20. The hydrogen generation system according to claim 17, wherein said receiver plate includes at least one passage having a control valve connected thereto and said passage communicates with said liquid inlet port.
21. A fuel cartridge for a hydrogen generating system, said cartridge comprising:
  - a. a cartridge comprising an end cap and containing a bed of substantially anhydrous hydride reactant;
  - b. at least one liquid conduit fixed to said end cap and extending into said bed, said liquid conduit having at least one liquid distribution aperture formed thereon, said liquid distribution aperture having a diameter of between about 50 microns and about 1000 microns such that liquid reactant may pass in liquid form into said bed of hydride reactant;
  - c. wherein said end cap comprises a liquid inlet port communicating with said liquid conduit and a gas outlet port, both said liquid inlet port and said gas outlet port being adapted to seal with a cartridge receiver.



22. The fuel cartridge according to claim 21, wherein said hydride reactant comprises sodium borohydride and an anhydrous activating agent.
23. The fuel cartridge according to claim 22, wherein said hydride reactant comprises about 20 percent by weight to about 60 percent by weight of activating agent.
24. The fuel cartridge according to claim 23, wherein said activating agent comprises at least one salt of the group alkaline earth metals, alkali metals, or halides.
25. The fuel cartridge according to claim 24, wherein said activating agent is water soluble.
26. The fuel cartridge according to claim 22, wherein said activating agent is at least one chosen from  $\text{BeF}_2$ ,  $\text{BeCl}_2$ ,  $\text{BeBr}_2$ ,  $\text{BeI}_2$ ,  $\text{MgF}_2$ ,  $\text{MgBr}_2$ ,  $\text{MgCl}_2$ ,  $\text{MgI}_2$ ,  $\text{CaF}_2$ ,  $\text{CaCl}_2$ ,  $\text{CaBr}_2$ ,  $\text{CaI}_2$ ,  $\text{LiF}$ ,  $\text{LiCl}$ ,  $\text{LiBr}$ ,  $\text{LiI}$ ,  $\text{NaF}$ ,  $\text{NaCl}$ ,  $\text{NaBr}$ ,  $\text{NaI}$ ,  $\text{KF}$ ,  $\text{KCl}$ ,  $\text{KBr}$ ,  $\text{KI}$ ,  $\text{Li}_2\text{S}$ , or  $\text{Li}_2\text{Se}$ .
27. The fuel cartridge according to claim 24, wherein said activating agent is chosen from at least one of  $\text{MgCl}_2$ ,  $\text{BeCl}_2$ ,  $\text{CuCl}_2$ ,  $\text{LiCl}$ ,  $\text{NaCl}$ , or  $\text{KCl}$ .
28. The fuel cartridge according to claim 22, wherein said activating agent is chosen from at least one of  $\text{Cu}$ ,  $\text{Co}$ ,  $\text{Ni}$ ,  $\text{Pt}$ ,  $\text{Pd}$ ,  $\text{Fe}$ ,  $\text{Ru}$ ,  $\text{Mn}$ , or  $\text{Cr}$ .
29. The fuel cartridge according to claim 28, wherein said activating agent is at least one chosen from  $\text{Cu}$ ,  $\text{Ru}$ , or  $\text{Ni}$ .
30. The fuel cartridge according to claim 22, wherein said activating agent is an anhydrous acid having an acid constant  $K_a$  of between approximately  $10^{-4}$  to approximately  $10^{-11}$ .
31. The fuel cartridge according to claim 30, wherein said activating agent is at least one of tartaric acid or benzoic acid.
32. The fuel cartridge according to claim 21, wherein multiple liquid conduits extend into said cartridge.
33. The fuel cartridge according to claim 21, wherein said hydride is enclosed in a fabric pouch having a substantially greater area than thickness.
34. The fuel cartridge according to claim 33, wherein said fabric pouch is rolled within said cartridge and said water conduit extends between folds of said fabric pouch.
35. The fuel cartridge according to claim 33, wherein said fabric pouch comprises a woven Nylon material.
36. The fuel cartridge according to claim 21, wherein said hydride comprises lithium aluminum hydride and said liquid conduit is surrounded by a fabric sleeve.
37. The fuel cartridge according to claim 21, wherein said plurality of liquid conduits are spaced radially from a center point in a nonuniform configuration.

38. The fuel cartridge according to claim 37, wherein said nonuniform configuration is a spiral pattern.
39. The fuel cartridge according to claim 21, wherein a liquid acid containing reservoir is attached to said cartridge.
40. The fuel cartridge according to claim 21, further comprising at least one port allowing for the release of gas from said cartridge.
41. The fuel cartridge according to claim 21, further comprising a plurality of liquid conduits wherein at least two of said conduits are of different lengths.
42. The fuel cartridge of claim 21 wherein said liquid conduit further comprises a plurality of liquid distribution apertures.
43. The fuel cartridge of claim 21, further comprising a plurality of liquid conduits wherein said liquid distribution aperture one liquid conduit is at a different location than said liquid distribution aperture on at least one other conduit.
44. The fuel cartridge of claim 43, wherein said distribution aperture is formed by an open end of said conduit.
45. A fuel cartridge for a hydrogen generating system, said cartridge comprising:
- a. a cartridge comprising an end cap and containing a substantially anhydrous hydride reactant;
  - b. a plurality of liquid conduits fixed to said end cap and extending into said cartridge, each of said liquid conduits having a length and at least one liquid distribution aperture, said plurality of liquid conduits configured to distribute liquid reactant at different locations within said cartridge; and
  - c. wherein said hydride reactant forms a bed and said liquid conduits extend into said bed.

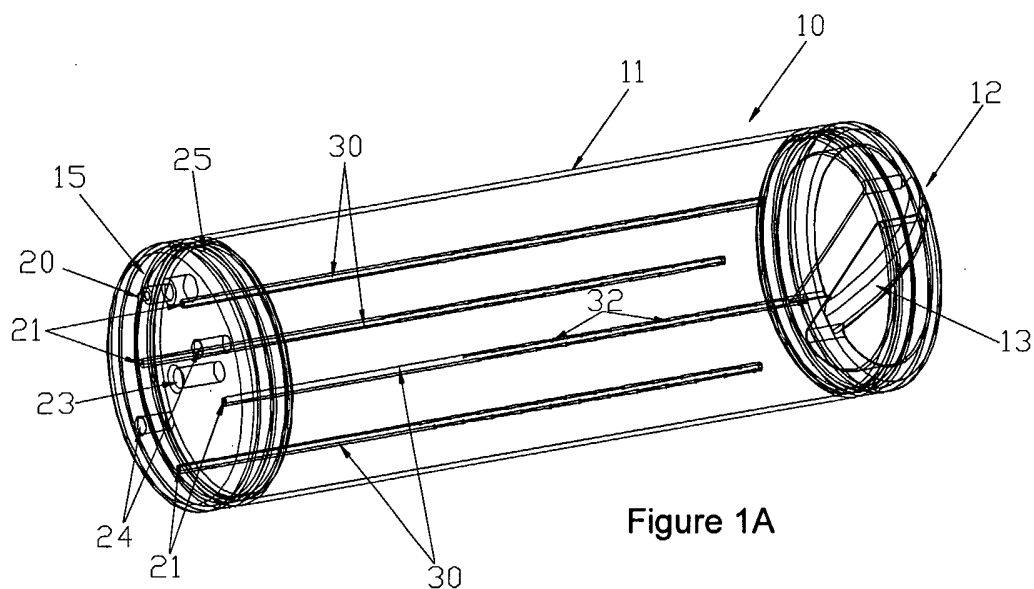


Figure 1A

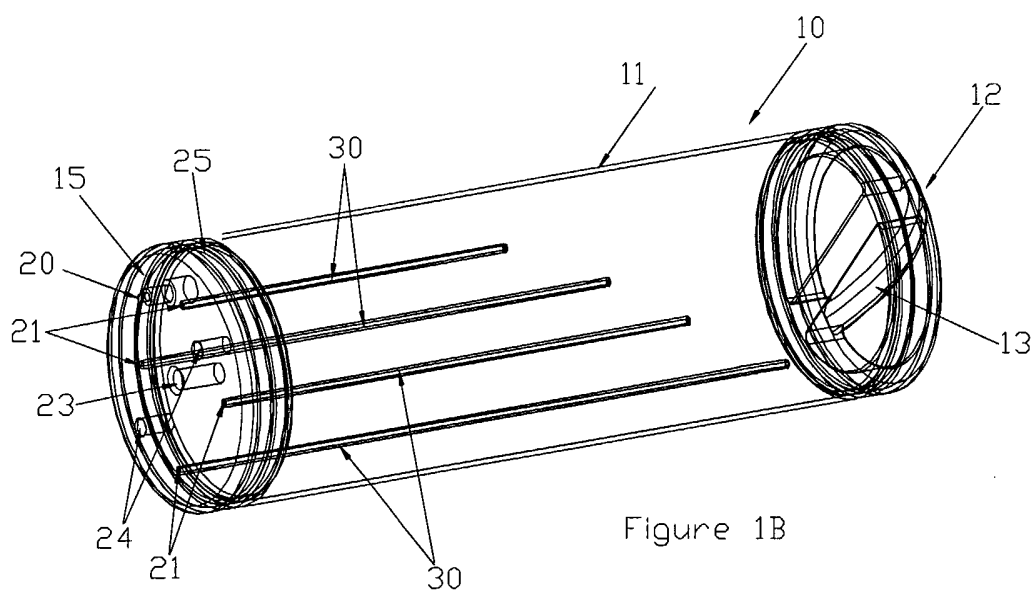
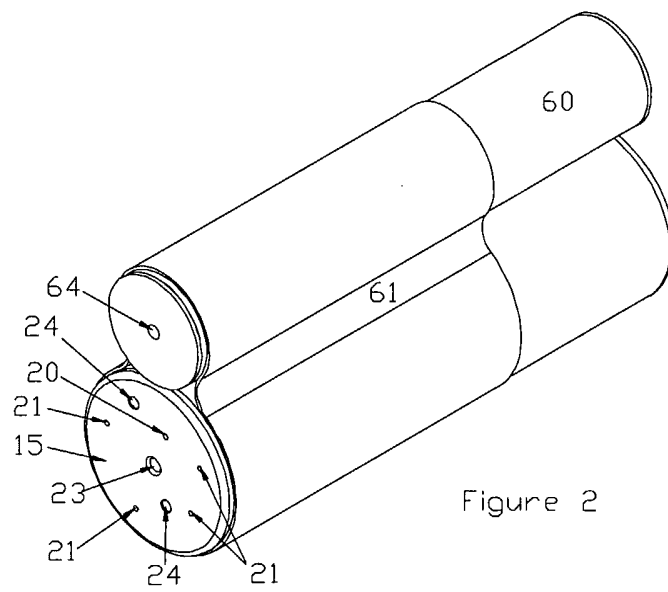


Figure 1B



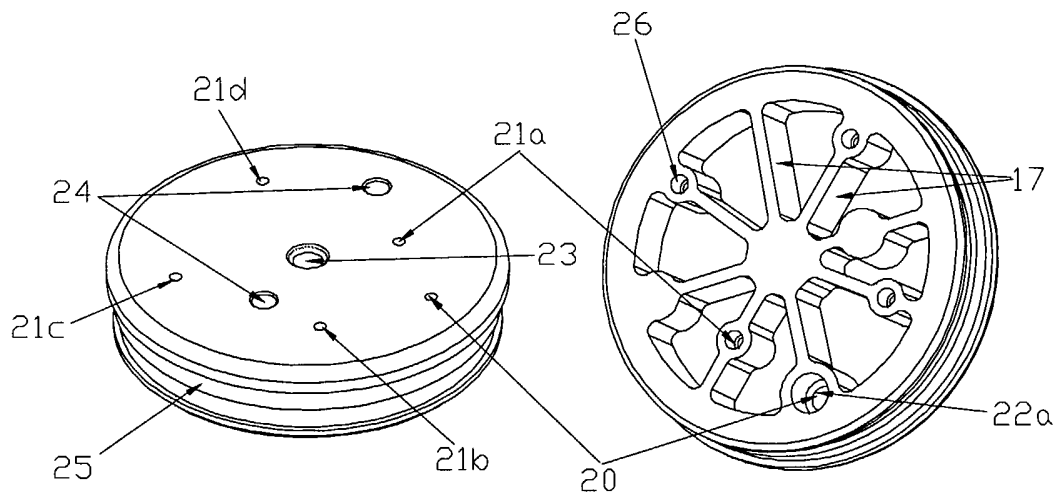


Figure 3A

Figure 3B

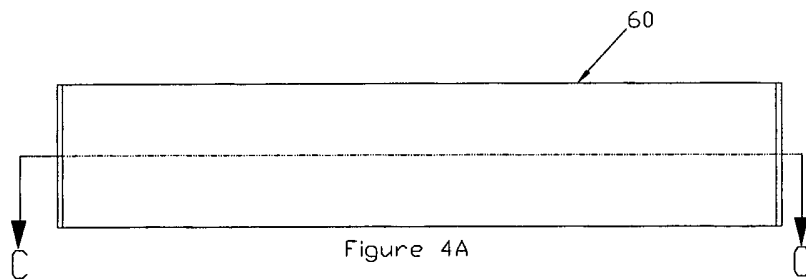


Figure 4A

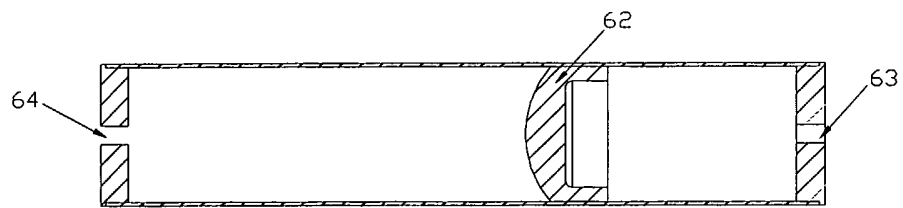


Figure 4B - Section CC

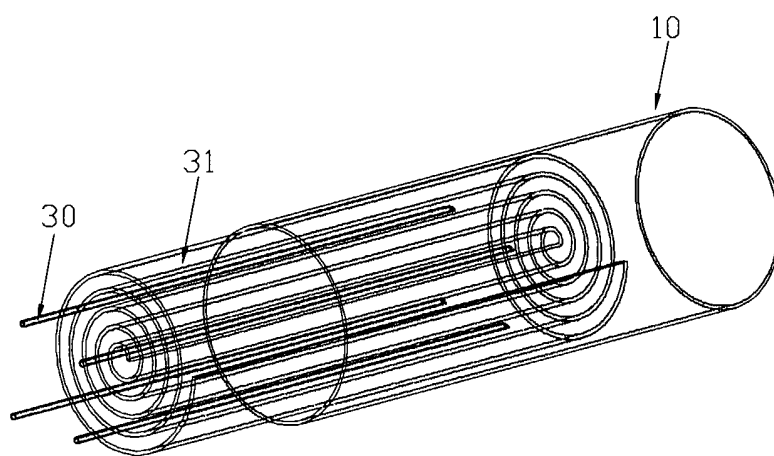


Figure 5

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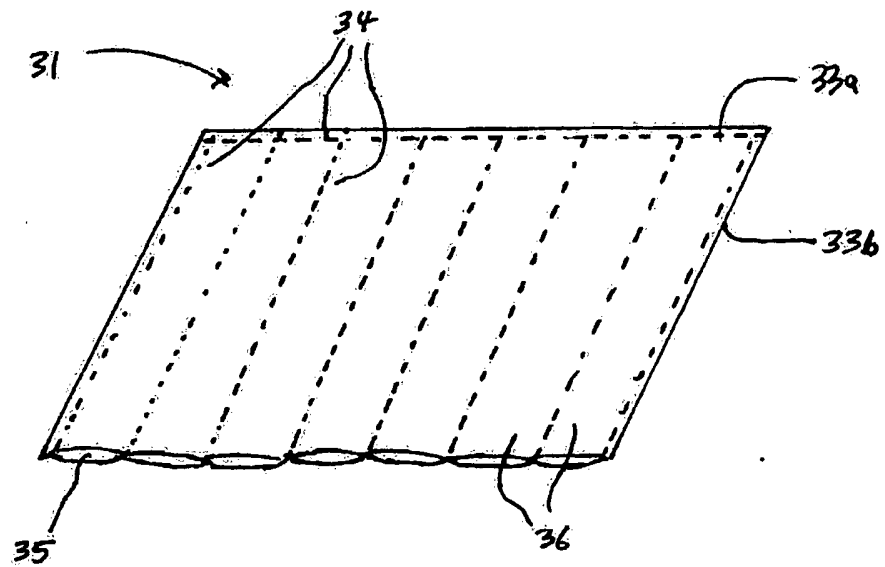


Figure 6

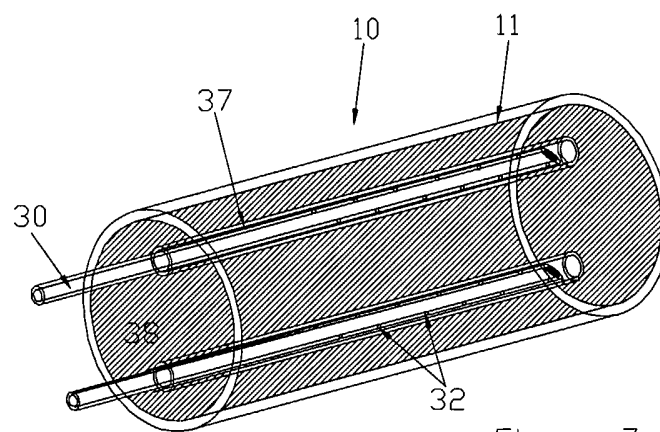


Figure 7

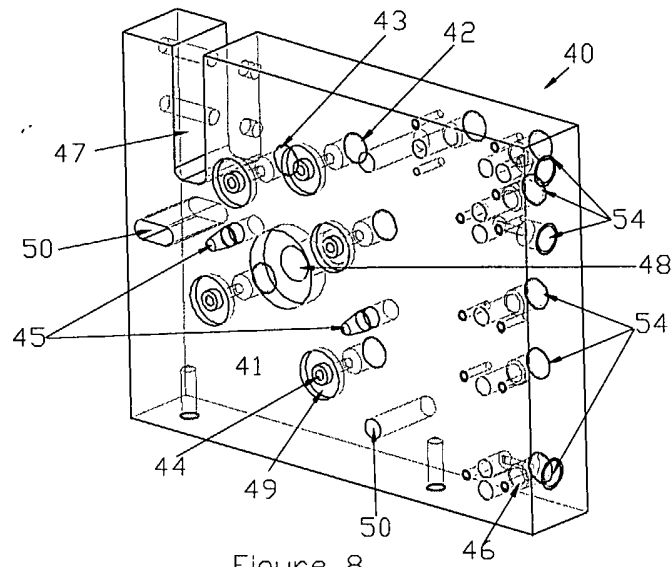


Figure 8



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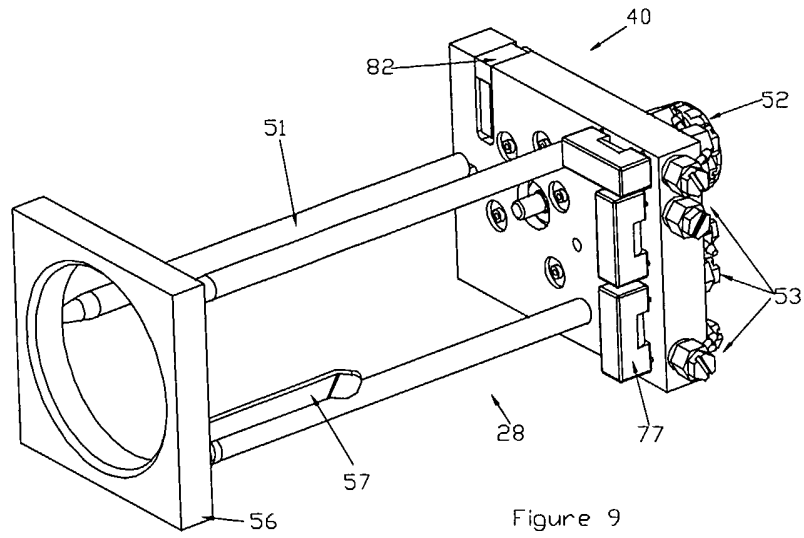


Figure 9

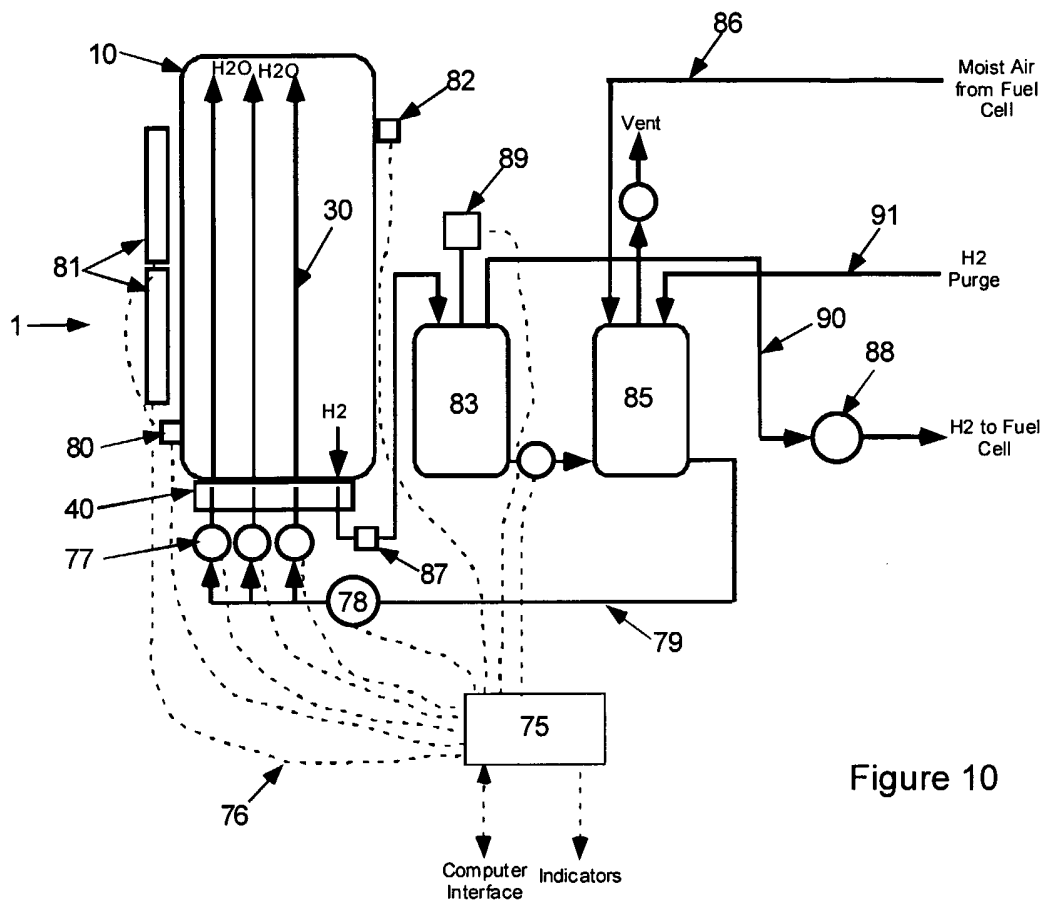


Figure 10

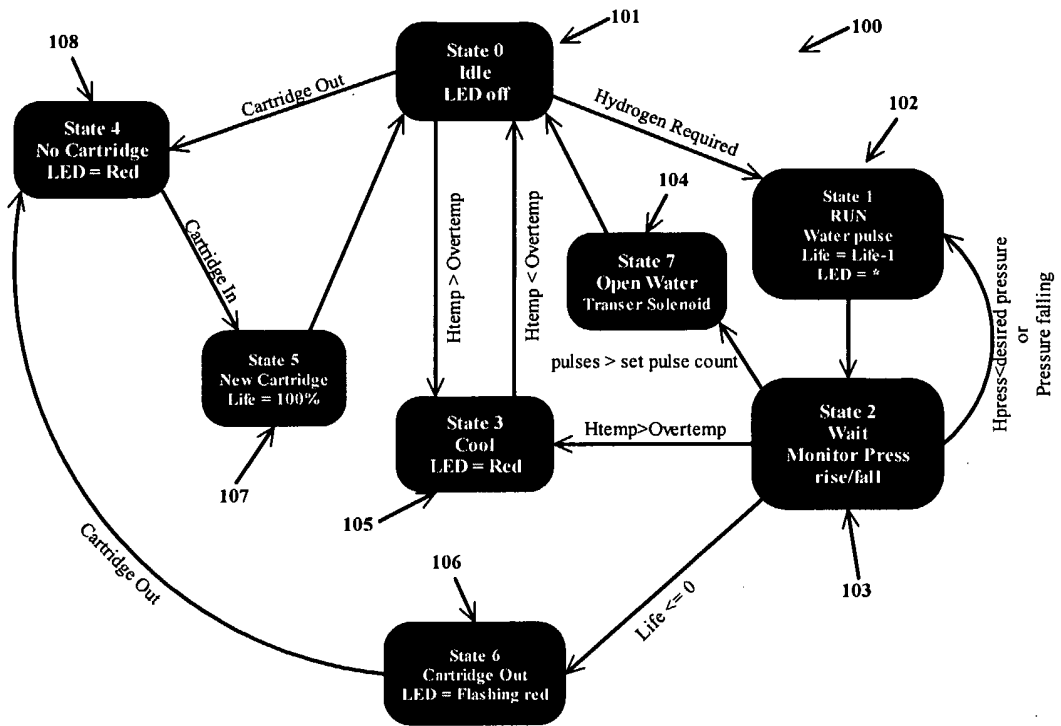


Figure 11

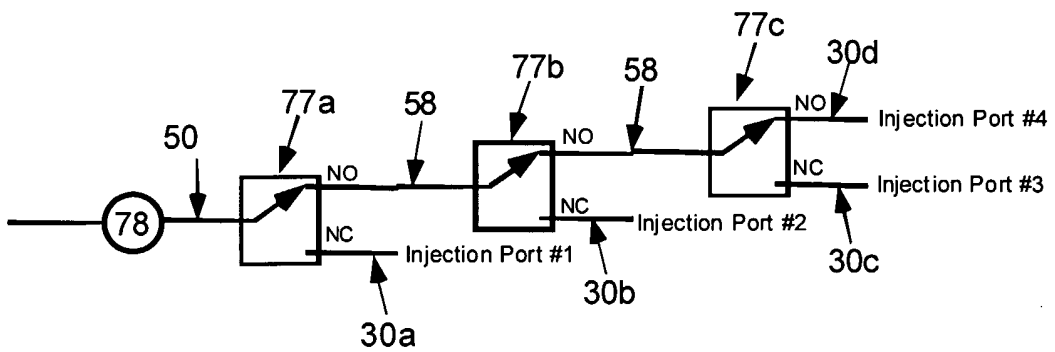


Figure 12

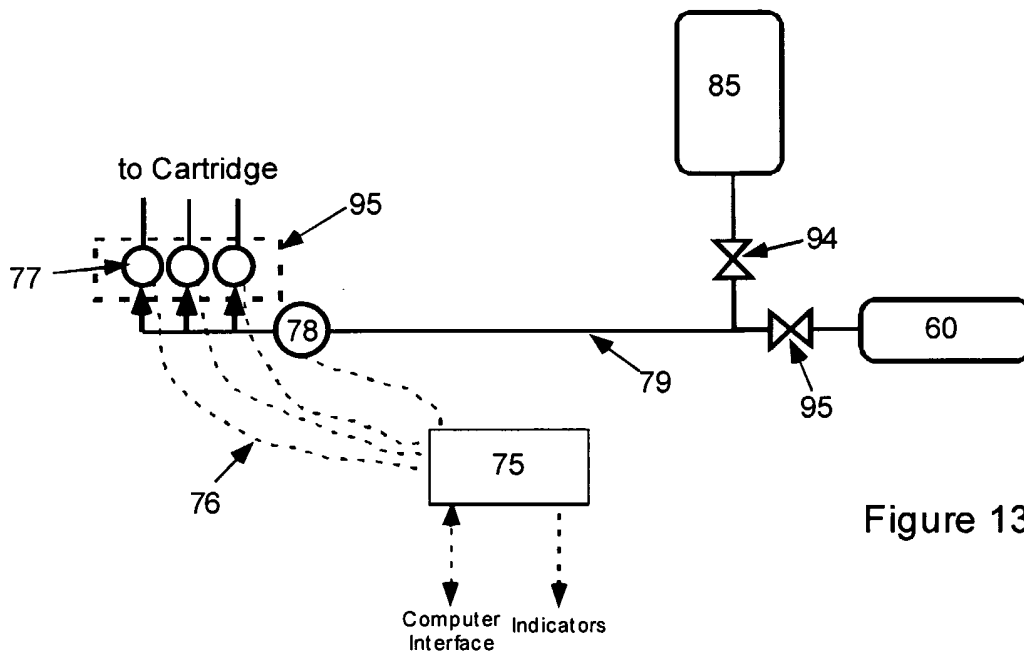


Figure 13

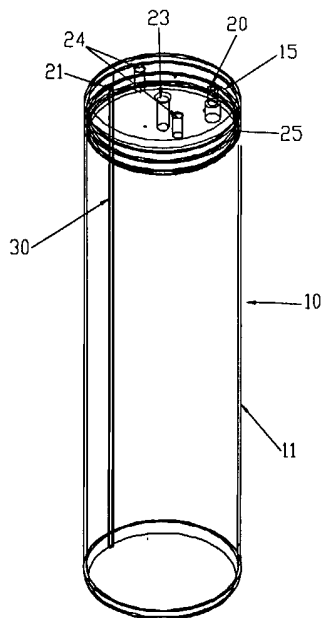


Figure 14

**INTERNATIONAL SEARCH REPORT**

International application No.  
PCT/US05/40975

**A. CLASSIFICATION OF SUBJECT MATTER**  
 IPC: B01J 7/00( 2006.01),7/02( 2006.01)  
 C10J 3/00( 2006.01);B01J 8/02( 2006.01)  
 USPC: 422/211,239;48/211,204,197R  
 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)  
 U.S. : 422/211, 239; 48/211,204,197R

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 practicable, search terms used)  
 Please See Continuation Sheet

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5,514,353 A (ADLHART et al) 07 May 1996 (07.05.1996), Column 2, lines 30-40, Claim 1 and Figure 5.	1,4 and 5
Y		2-3 and 6-45
Y	US 5,883,934 A (ADLHART) 10 November 1998 (10.11.1998), Abstract, Column 3, lines 31-57.	1-45
A	US 5,728,464 A (CHECKETTS) 17 March 1998 (17.03.1998).	1-45

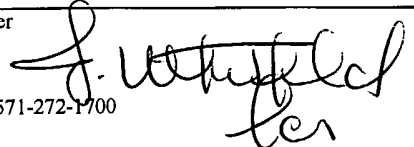
Further documents are listed in the continuation of Box C.  See patent family annex.

* Special categories of cited documents:	"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
"A" document defining the general state of the art which is not considered to be of particular relevance	"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
"E" earlier application or patent published on or after the international filing date	"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
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&" document member of the same patent family
"O" document referring to an oral disclosure, use, exhibition or other means	
"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search: 27 February 2006 (27.02.2006)  
 Date of mailing of the international search report: 14 MAR 2006

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**INTERNATIONAL SEARCH REPORT**

International application No.  
PCT/US05/40975

Continuation of B. FIELDS SEARCHED Item 3:  
USPAT, USPGPUB, DERWENT, JAPO, EPO  
search terms: cartridge, hydrogen generat\$5, hydride, hydrogen compact, fuel cell