

PEM Fuel Cell Integration With a Hydrogen Generator on a Bench

FINAL REPORT
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ABSTRACT

The main intent of this project was to bench test a next generation Proton Exchange Membrane (PEM) fuel cell system with a Hydrogen on Demand Fuel Source. This system will require the following elements to run: a fuel cell stack, a fuel source, a humidifier, a compressor and motor, an electronic controller, a cooling pump and motor(s), a pressure transducer, a hydrogen shut-off valve, a hydrogen purge valve(s), a tank for hydrogen waste storage, and a tank and apparatus for the hydrogen generation. A previous version of the fuel cell system has been demonstrated for numerous uses and its application need not be discussed here. Instead, the intent of this project was to demonstrate the improvements of the next generation of fuel cell system by bench-testing it and comparing it to the previous version.

BACKGROUND

In 1998 - 1999, the NJDOT successfully developed a mobile prototype fuel cell system fueled by compressed hydrogen in cylinders. The Proton Exchange Membrane Fuel Cell used in this prototype consisted of 64 cells, stainless steel rods, and stainless steel plates for the inner plates as well as the end caps. In addition, the system contained: a purge valve, a stack controller, a cooling pump and motor, a pressure transducer, an electronic controller, and a humidifier. These components were necessary to the operation of the fuel cell, but several of the components as well as the fuel cell provided problems that limited the output. For the next generation, a 11.7 kilowatt Proton Exchange Membrane (PEM) fuel cell was designed and built. Hydrogen fuel for the fuel cell is provided by a Hydrogen-On-Demand™ system using sodium borohydride as a storage medium for hydrogen; hydrogen gas is generated by the passage of a solution over a catalyst. For the next generation of fuel cell systems, many of the bugs like corrosion of the stainless steel parts of the system have been worked out of the original system. This research presents a comparison between the two systems demonstrating the advancements in the technology.

The complexity of a fuel cell and hydrogen system required that companies coordinate their expertise. This project utilized three companies: Millennium Cell, Inc., H Power, and Recon Industrial Controls Corporation. Millennium Cell was responsible for the technology behind the hydrogen delivery to the fuel cell. H Power Corporation was primarily responsible for the fuel cell and its operation. Recon was responsible for the fuel cell control and monitoring system.

PROJECT GOALS¹

Below please find the original project goals outlined in the original proposal to NJDOT.

- Ease of use, storage, and safety of chemical
- Gas quality as well as a brief discussion on pressure and temperature issues
- Matching generator output with fuel cell output
- Bench testing of next generation PEM fuel cell with a hydrogen generator.

- Discussion of potential refinements
- The report will also contain a brief discussion on the background description of hydrogen storage.

INTRODUCTION

Fuel Cells Background and Theory

“Fuel cells have no moving parts, are nonpolluting and quiet and produce water as the only byproduct.”² A Proton Exchange Membrane Fuel Cell (PEMFC) is a device that electrochemically combines hydrogen and oxygen to produce electricity and pure water. There are no other side-products or emissions produced. Pure oxygen is required for fuel cells used in outer space; but in most terrestrial applications the oxygen is supplied to the cathode by flowing air through the cell. For this project, pure hydrogen will be generated by a liquid hydride technology developed for this project. The hydrogen is consumed at the rate required by the electrical load. In this way a fuel cell is different from batteries. A battery must be recharged each time it uses the electrical charge that was stored in it, but a fuel cell will continue to generate electricity as long as hydrogen and oxygen are supplied to its cells³.

The first problem is that even though fuel cells are a highly efficient and clean method for converting hydrogen to electric power, they require refinement and further development. A sub-goal for this project was to make improvements in PEM fuel cell weight, volume, efficiency, reliability, and life expectancy. H-Power Corporation primarily undertook these tasks. Shown in Figure 1 is a test stand where fuel cell stacks are tested and developed.

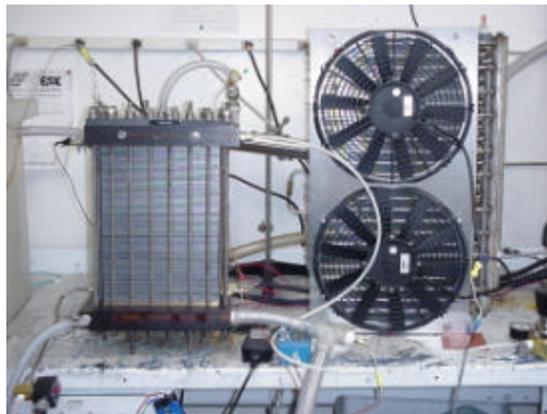


Figure 1 High-power fuel cell test stand at H-Power.

RESEARCH AND DEVELOPMENT

Next Generation Fuel Cell Development at H-Power Inc.

The fuel cell stacks are graphite stacks with plastic end caps, a design change that lowered fuel cell weight by nearly half over the previous years technology. For the previous generation, the fuel cell was steel intensive, components like the plates, end caps, and the tie rods were all steel. One concern in using the steel plates was that the humidification and the fuel cell cooling systems both used de-ionized water, which can corrode steel. To resolve this problem, the new stacks were produced using graphite plates. The graphite does not corrode or react with the de-ionized water. However graphite is generally more brittle, more expensive, and more variable in strength and thickness than the steel counterparts. The end caps of the fuel cells were also replaced with plastic. There are several reasons why this was done, the plastic caps are lighter and cheaper than steel. Also the plastic is easier to machine, the caps need to have the manifolds, tie rod holes, cooling loops, hydrogen feed line, and humidified air feed line machined into the cap. By using high strength plastic to withstand the torque of the tie rods the cost and weight of the end caps were significantly reduced in comparison to steel caps. The tie rods were replaced with titanium, this was done to reduce the weight of the stacks. Steel is approximately 75 percent heavier than titanium. Since titanium is stronger than steel, in theory the diameter of the tie rods could also have been reduced further reducing the weight but the plastic end caps had already been manufactured and purchased. The net weight savings per fuel cell stack was approximately 50 pounds. The result of the upgrades is that each fuel cell stack is now 10" X 12" X 24" and weighs only 75 pounds.

Based on actual use and lessons learned from previous generations, the fuel cells were built to improve overall electrical efficiency as well. The previous generation of fuel cell came equipped with a 64 cell, 4.2 kW fuel cell; whereas the one that was bench tested was outfitted with two (2) 100 cell 5.85 kW fuel cell stacks wired in series. The unregulated voltage varies from 120 to 200 VDC, because the voltage of the individual cells is proportional to the electrical load (0.6 to 1.0 V/Cell). The individual cells are "stacked" in series so the voltages add to provide the higher voltages needed by the other ancillary devices. A DC/DC converter regulates the variable voltage to a steady DC input to an inverter that transforms it to AC. The traction motor and the air compressor operate on AC; all other components operate on the regulated 12 and 24 VDC power.

In a fuel cell if the membranes become too wet, a condition referred to as 'flooding' occurs. The 'flooded' state will prohibit the reactant gases, mainly the oxygen from reaching the catalyst. As excess moisture accumulates in the stack the number of reactions decrease because it cannot perform hydrogen reduction. Consequently the stack will no longer be able to produce electricity efficiently⁴. The residual water from must be periodically purged from the cell, in the previous generation the fuel cell system used a four-channel flow manifold for the purge. It was believed that the stack had dead areas where water was not being fully purged or that the channels did not provide

a quick enough water removal system. Since flooding decreases the efficiency of the reactions within the stack this was considered a major concern. Therefore a sixteen-channel manifold was used for the fuel cells in this project. The hydrogen-side of the cells are pressurized with pure hydrogen and are periodically purged to remove any product water that may accumulate. The average flow rate of the hydrogen was 4.9 SCFM and both the air and hydrogen were pressurized to 6 psi. This has resulted in good laboratory results where the stack efficiency does not fluctuate as much as before.

The original ballpark figure of how much power would be produced by the fuel cells was estimated at 14kW. This figure was based on the fact that the individual cells can produce 0.6 to 1.0 V/cell, thus if each stack produces a maximum of 1.0 V/cell, has 100 cells, running at 90 amps, and there are two stacks the net power would be about 18,000 watts. If we further refine this design to account for the heat losses within the fuel cell of about 50 percent we find that the actual expected net output of the fuel cell should be about 9 kW. If the system were to really produce 9 kW this would be more than double the previous generation fuel cell system of 4.2kW. Thus for design purposes a conservative 10 kW was used in a rough design of the systems. The actual power output of the fuel cells was unknown until after the bench testing of the fuel cells. A summary load versus voltage and power graph is shown in Figure 2 and Figure 3. At 90 amps stack one is running at 64.4 V with an output of 5,797 watts. Stack two at 90 amps is running at 65.5 V and 5,895 watts. Therefore the estimated 14 kW fuel cell output is actually 11.7 kW based on the bench test results. Since the 9 kW output was really only a rough target this 2.7 kW increase was not unexpected.

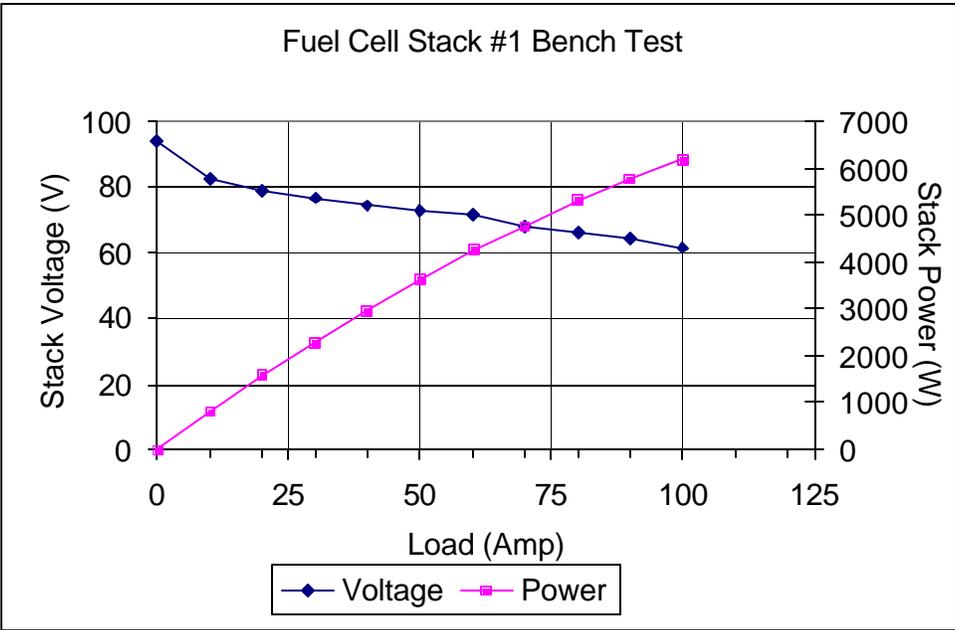


Figure 2 Bench test results of the fuel cell stack number two at H-Power.

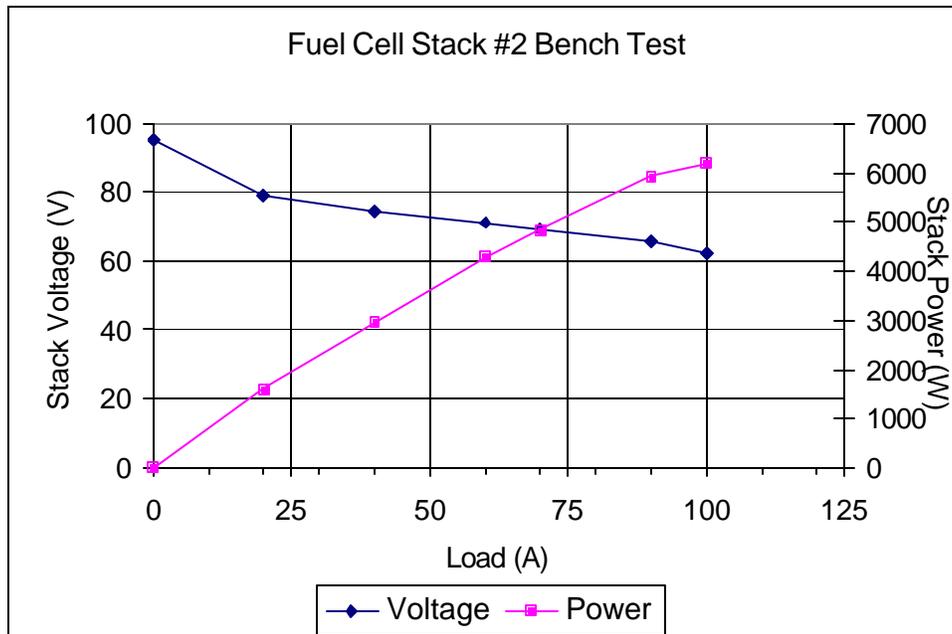


Figure 3 Bench test results of the fuel cell stack number two at H Power.

Two fuel cells are being used for this project both of which are larger than the one previously used. Instead of having true parallel system components, such as air blowers, controllers, humidifiers, and heat exchangers, they have been increased in size and the fuel cells linked in a series system. For example only one blower is used for both stacks. Also a new custom built fully automated controller monitors all fuel cell functions as well as optimizing flow rates and purge cycles thus increasing overall system efficiency. The control board is a critical component and is responsible for varying the speed of the air compressor as the load changes and for continuously performing safety checks of the voltage and temperature of the fuel cell stacks. Data is recorded for detailed analysis later.

Air Flow and Thermal Management of Fuel Cell Stacks

The byproducts of a PEM Fuel Cell are water, electricity, and heat. While the first two byproducts are desirable and not a problem, heat can be a problem with the fuel cell. For the PEM Fuel Cell, the operational temperature is approximately 60°C. On a bench the temperature must be regulated via a blower, which was used to cool the fuel cell. An improvement from the previous generation is the use of one blower to cool two fuel cell stacks. Within the old system, one blower would be used per stack.

The fuel cell is supplied with air to complete the reaction. This air is supplied by a air blower and a series of valves to regulate the pressure. This regenerative blower was borrowed from Becker, a German company. Figure 4 shows the diagram of the air flow. In fact, airflow rates of 30 CFM (~900 SLPM) can be obtained. Pressure can be as high as 7 psig at low flow rates.

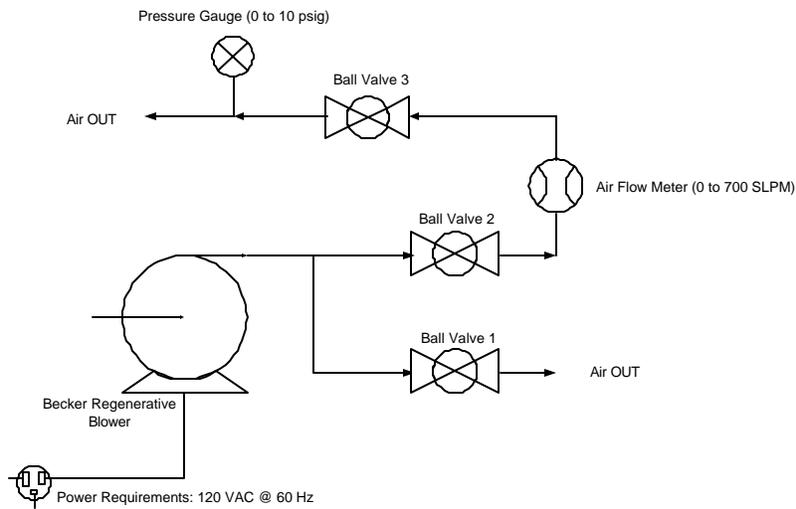


Figure 4 Air Flow Diagram

Ball valves were used in order to modulate both the airflow rate and the pressure. Flow rates and pressures were varied to delimit an operating range and to see the possibilities of this new blower. Results are illustrated in Figure 5. The airflow can be varied depending on the pressure. For instance, at 3 psig, the flow rate can be changed from 25 to 525 SLPM, which is a large range.

The power consumed by the blower is comparable and in the range of the one utilized in the first generation systems provided by Recon. Lower power consumption can be achieved by using larger pipes. According to the supplier, by using 1½" pipes instead of ¾" like in the experiment, an economy of 100 WAC on all the tested pressures is easily obtainable. A picture of the blower is shown in Figure 6. The air for each fuel cell will be humidified by a shell in tube membrane humidifier. The fuel cell stacks each require 14 scfm of air with the pressure drop across the fuel cell system being approximately 6 psi.

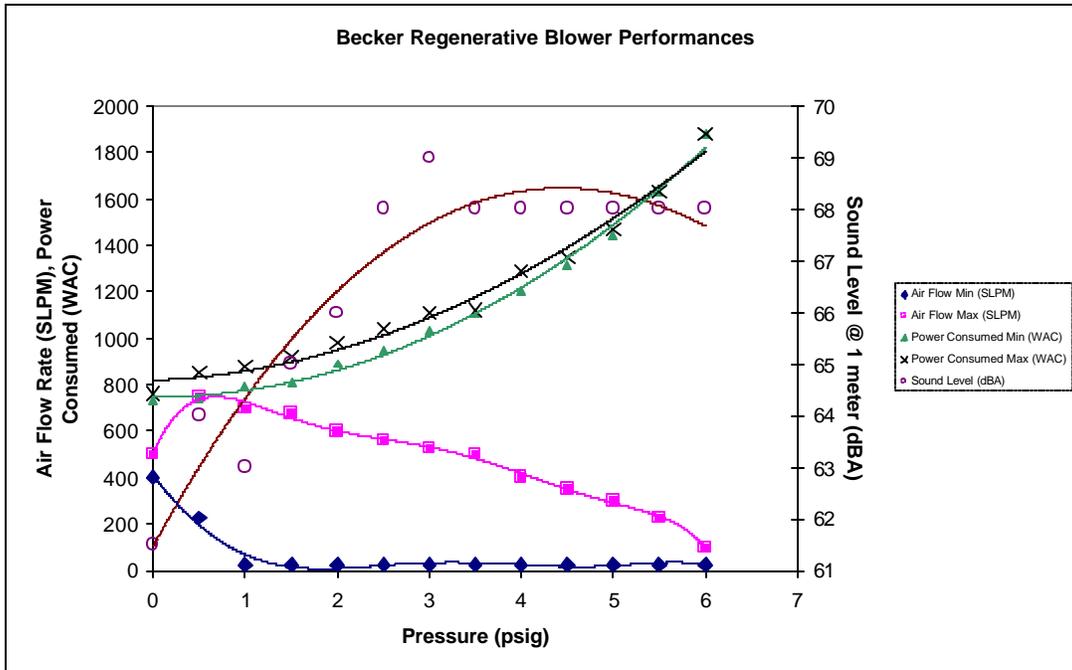


Figure 5. Results obtained on the Becker blower.



Figure 6. Closer look at the regenerative blower.

Advanced Fuel Cell / Hydrogen Generator Development

For the fuel cell, the cell will continue to provide energy output if there is a continuous supply of fuel input. The hydrogen fuel will be provided by a Hydrogen-On-Demand™ system using sodium borohydride as a storage medium for hydrogen. The hydrogen generation system could potentially be used in the heating, power generation, mobile communications, and many other stationary and mobile industry applications, as these are outside the scope of work, they shall not be discussed.

Hydrogen-generation holds great promise as a path to clean energy. "Hydrogen has the highest mass energy density of any fuel: 120 MJ/kg (LHV) 144 MJ/kg (HHV)¹" and has the potential to be produced by renewable fuels. When used as a fuel, virtually the only emission from the reaction is pure water. However, there are significant barriers to the introduction of hydrogen as an everyday fuel.

Hydrogen Storage Background and Theory

There are currently seven (7) primary ways to store hydrogen: Chemically Stored Hydrogen, Compressed Hydrogen, Metal Hydride Tanks, Liquid Hydrogen, Carbon Nanotubes, Glass Microspheres, and Liquid Carrier Storage. Chemically Stored Hydrogen works through a reaction which releases chemically stored hydrogen. Through storage of a compound that is hydrogen rich in its natural stable state, a reaction can be produced in which the hydrogen is produced and collected on demand. This process reduces or eliminates most of the storage issues related to the other six storage possibilities. Compressed high-pressure tanks are fairly common and the theory fairly straight forward. The tanks are filled with hydrogen gas at high pressure, thus making the tanks quite large and expensive. The Metal Hydrides work through absorption of the hydrogen, such that it can be released later. The release process occurs via heat either at room temperature or sometimes at temperatures in excess of 250°C. The biggest disadvantage of these Metal Hydride systems is that when releasing the hydrogen only pure hydrogen is released leaving behind any impurities to degrade storage potential. The Liquid Hydrogen requires large amounts of energy to obtain and maintain the -253°C temperature to keep the hydrogen in a liquid state. Plus safety becomes a significant factor when dealing with such cold temperature and tank integrity. Carbon Nanotubes utilize microscopic pores to store massive amounts of hydrogen. These Nanotubes work on a similar process to the Metal Hydrides without the disadvantages of the hydrides. However these Nanotubes are very costly and still in the research phase, manufacturing techniques still require development to go into commercial application. Glass Microspheres are similar to the Nanotubes, in which the spheres are heated then filled with hydrogen and the cooled thus trapping the hydrogen inside. Thus when the spheres are heated again the hydrogen can be released. These too are also still in research and development requiring development to go into commercialization. Liquid Carrier is just another name for fossil fuels such as gasoline storing the hydrogen. By reforming the fossil fuel the hydrogen is released, however much care must be taken to ensure the hydrogen purity and remove any carbon monoxide. Plus the energy to reform fossil fuels is high and the system inefficient.

Hydrogen-On-Demand Development at Millennium Cell, Inc.

Hydrogen gas has a very low volumetric energy density. Previous solutions to this problem, including high-pressure storage, storage using metal hydride adsorption⁵, and liquefaction, all have significant drawbacks. Researchers have spent considerable effort to identify a method to overcome the safety, weight, and volumetric limitations of these storage mechanisms for all uses.^{6,7}

The premise of the project required the use of a catalyst enhanced storage process. It was proposed that a sodium borohydride system be used as a medium for the storage, transportation, and generation of hydrogen gas, see Appendix 1 for Material Safety Data Sheet (MSDS) information. The stoichiometric hydrolysis reaction of sodium borohydride (NaBH_4) can generate 4 mols of H_2 gas per mol of NaBH_4 .⁸ Sodium borohydride powder is stable in dry air, but will undergo hydrolysis with acidic or neutral pH water to generate hydrogen gas. Sodium borohydride is incompatible chemically with heat, strong oxidizing agents, chemically active metals, and acids. See Appendix 2 – Commercially Available Sodium Borohydride Product Information for more information on sodium borohydride powders available from Rohm and Haas. The Rohm and Haas literature has summarized the properties, handling, and disposal of sodium borohydride powder. The powder form of sodium borohydride is considered flammable as the hydrogen generated from hydrolysis or thermal decomposition will ignite in the presence of free flame.

The most likely route of exposure to sodium borohydride powder is via skin contact, therefore lab protective gear is recommended including goggles and face shield, lab coat and apron, vent hood, etc. for an exact description and safety precautions of sodium borohydride in the powder form (please Appendix 1 - MSDS and Product Information¹³ for MSDS information). The acute dermal LD50 of sodium Borohydride is 4-8 g/kg¹⁰ (equivalent to 272 to 544 g for a 150 lb person). The current cost of sodium borohydride is \$40/kg however if it were to become more widely used for hydrogen production, its cost would reduce and it could become economically competitive with fossil fuels⁸.

Over time, non-stabilized solutions of sodium borohydride will decompose and off-gas hydrogen. This rapid reaction makes raw sodium borohydride an infeasible fuel solution, and continuous production of hydrogen gas is a safety issue. Alkaline solutions of sodium hydroxide are stable^{9 and 10} as the rate of the hydrolysis reaction is slowed with increasing pH. In fact, a solution of sodium borohydride and sodium hydroxide is commercially available for use in the paper industry and is stable for months. Concentrations as low as 1 percent sodium hydroxide are enough to prevent hydrolysis and to allow the use of aqueous sodium borohydride as a viable fuel solution. For use in the Hydrogen-On-Demand™ generator, a 20 percent by weight solution of sodium borohydride is stabilized by 1 percent by weight sodium hydroxide. Therefore the fuel composition by weight is 20 percent sodium borohydride (NaBH_4), one percent sodium

hydroxide stabilizer, and 79 percent water. As hydrogen generation only occurs in the presence of selected catalysts, hydrogen generation rates can be controlled, storage efficiencies of hydrogen are high, and hydrogen can be generated at a wide range of temperatures⁸. It should be noted that the solution is still corrosive and potentially hazardous, though nonflammable, and must be handled accordingly. At this concentration, the system should be capable of supplying between 120 to 150 L/min of hydrogen at about 100 psi. In Figure 7 it can be seen that there is an initial spike of about 150 L/min in hydrogen generation during the start-up of the generator. After about five minutes the reaction becomes more uniform and hydrogen production levels out at about 130L/min until the flow of sodium borohydride solution is ceased.

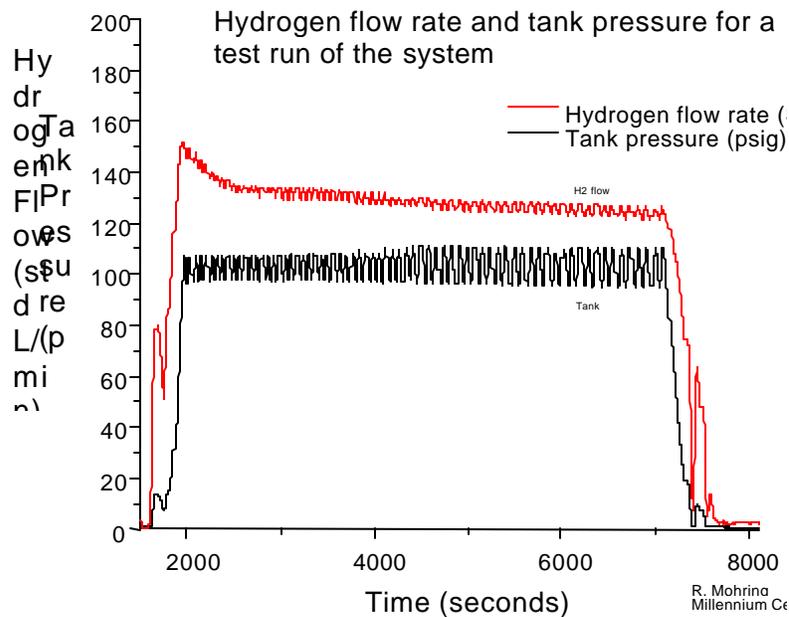


Figure 7 Initial hydrogen generation flow rate experiments utilizing the Hydrogen-On-Demand™ generator.

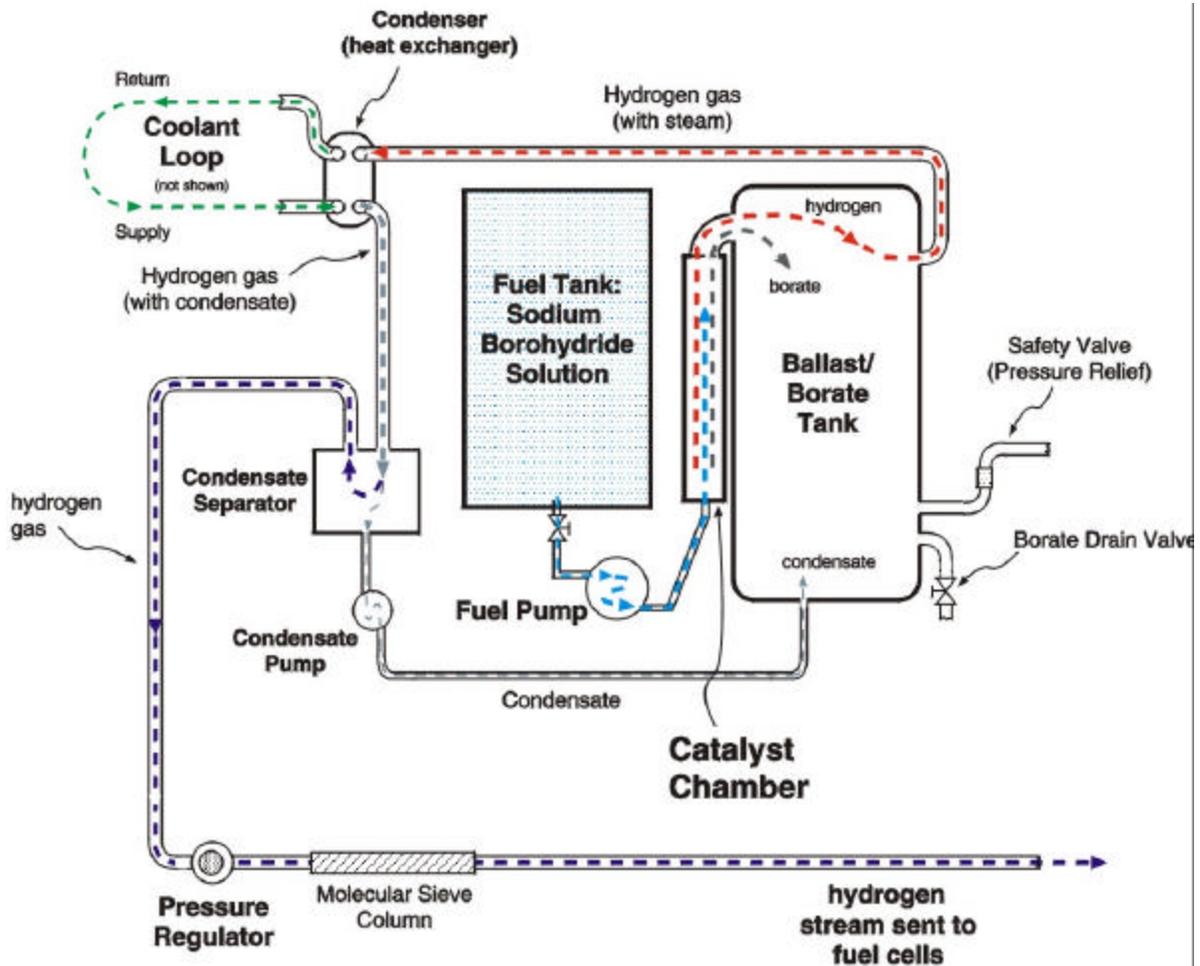


Figure 8 Millennium Cell, Inc. Hydrogen-On-Demand™ System showing pumps siphon the sodium borohydride from the fuel tank and deliver it into the reaction chamber followed by the coolant system utilizing heat exchanger to cool the hydrogen stream.

Millennium Cell, Inc. has evaluated several reactor designs in the course of development of their proprietary Hydrogen-On-Demand™ technology. The design used for this project maximizes hydrogen generation for this particular system. For a discussion of the reaction chamber, catalysts, and attempts to increase efficiency through re-design of the chamber please see the paper entitled "An ultrasafe hydrogen generator: aqueous, alkaline Borohydride solutions and Ru catalyst" published in the 2000 Journal of Power Sources.¹¹

The Hydrogen-On-Demand™ system is designed using stainless steel and plastics, which are resistant to the alkalinity of the fuel. The fuel solution can be stored in stainless steel, mild steel, or fiberglass vessels; with stainless steel (316 SS or 304 SS) recommended for piping, valves, pumps, etc. The fuel solution cannot be stored in vessels, which it may react with like aluminum.

In the Hydrogen-On-Demand™ system as shown in Figure 8, a solution of sodium borohydride in water is stored in a plastic fuel tank see Figure 9. Pumps siphon the sodium borohydride solution from the fuel tank to the reaction chamber. Millennium Cell's proprietary catalyst is contained within the reaction chamber; sodium borohydride solution flows over but does not disassociate the catalyst. As the solution flows over the catalyst, hydrolysis occurs and hydrogen gas is released; the byproducts of this reaction are water, heat, sodium borate, sodium hydroxide, and of course hydrogen. The spent fuel, sodium borate, as shown in Figure 10 and gaseous hydrogen from the reactor passes into a pressurized separation/buffer storage tank shown in Figure 9. This tank can hold +/- 32 gallons. The hydrogen and the water in the form of steam pass from the separation/buffer storage tank into the condenser. The steam is converted to liquid water and any residual sodium borate and sodium hydroxide are solidified as the water collects in the condensate reservoir where it can be removed.

In regards to freezing of the solution, all aqueous salt solutions exhibit the colligative property of freezing point depression. As the fuel solution is a solution of two inorganic salts – sodium hydroxide and sodium borohydride – both will have an effect on the freezing point of water. This effect is similar to the use of ethylene glycol as antifreeze. The fuel solution should not freeze at temperatures above -30°C .

In the condensate / sodium borate tank the spent fuel sodium borate is collected. This material must be drained and disposed of after each run of the generator. The drain valve on the bottom of the tank is opened and the material is gravity drained from the tank. Sodium borate is known to crystallize as demonstrated in Figure 10 and could potentially cause clogging issues within the system, though this has not been observed to date. After the tank is drained the entire system is flushed with clean warm water to remove the sodium borate spent fuel. Sodium borate is not considered to be toxic, and can be disposed of down the drain to the sewer in aqueous solutions in accordance with the MSDS and all federal, state and local environmental regulations.¹³ See Appendix 1 for the full MSDS information on the spent fuel sodium borate. There is currently research underway by other organizations into methods to recycle the sodium borate product back into sodium borohydride to make the generation system viable for the market.



Figure 9 Spent fuel condensate / sodium borate tank (left) and fuel tank (right).



Figure 10 Crystallized sodium borate byproduct of reaction.

After the hydrogen gas passes through the condenser and the condensate tank, it passes through a mist filter. Then the stream passes through two molecular sieves connected in series that helps ensure that no entrained sodium hydroxide that could poison the fuel cell membranes is carried into the cell. In general, high purity hydrogen is generated by hydrolysis of sodium borohydride. The Hydrogen-On-Demand™ system is a pressurized system. As the reaction chamber generates hydrogen the overall system pressure increases. After the hydrogen gas stream passes through the molecular sieves a regulator controls the pressure. The regulator maintains a system pressure of 100 psi, the pressurized hydrogen is then supplied to the fuel cell system at 7 to 9 psi.

Pressure and Thermal Management of Hydrogen-On-Demand System

A 12V diaphragm pump pushes sodium borohydride solution through a check valve/high pressure fluid release into the catalyst chamber. The check valve/high pressure fluid release valve will release fluid directly into the condensate / sodium borate tank in the event of a high-pressure condition in the liquid line.

The reaction of sodium borohydride and water produces heat when the materials are pumped over the catalyst. Heat is generated at the rate of 300 KJ/mol NaBH_4 - or, 7.8 KW. The majority of this heat is rejected via a heat exchanger coupled with a glycol/water-cooling loop to 2 fan-cooled radiators. The rest is radiated and convected from the catalyst chamber and condensate / sodium borate tank.

One obstruction encountered during the reaction chamber development was a thermal management problem. The system was running hotter than the anticipated design parameters; the hydrogen gas going to the fuel cells was gradually increasing in temperature during the reaction. Increasing the size of the heat exchanger and adding a second cooling fan solved this issue. Further evaluation, in particular field testing will be required to resolve the thermal management issues completely.

The Compressed Gas Association Guidelines for hydrogen were used for guidance in designing the system. The tank and its fittings are welded and pressure tested. The tank is steel. Piping and fittings in the system from the check valve onward are stainless steel with threaded fittings (with high-temp Teflon thread sealant). The operating temperature is 130°C to 170°C the tank temperature rating is 300°C.

The entire system is pressure tested overnight on hydrogen to 200 psi and monitored for any loss of pressure. The normal operating pressure is 100 psi with the pressure relief setting at 150 psi. In addition to this fluid pressure relief valve, there is a solenoid-operated pressure-relief valve and a backup mechanical pressure relief valve at the exit to the condensate / sodium borate tank. A short section of high-pressure stainless steel braided tubing on the hydrogen outlet provides strain relief to maintain the integrity of the hydrogen piping.

The net output of the fuel cells is 11.7 kW. Of that net voltage there are approximately 2 to 2.5 kW of system loads and efficiency losses. The boost converters are approximately 96 percent efficient therefore nearly 500 watts is lost in the converters. The compressor is one of the most significant loads in the fuel cell system at roughly 800 watts. One of the other most noteworthy loads is the cooling fans at about 500 watts. There are several additional system component loads like miscellaneous pumps and blowers among others that also draw down the net power estimated at another 300 watts. After taking into consideration all these loads the total usable power supplied to the motor is on the order of 9.6 kW. All of the main electrical systems were selected to reduce the power loss and loads as much as possible.

The fuel cell system consists of two stacks with an anticipated net out of 5.85 kW each, or 11.7 kW total at 90Amps and 120V to 130V. The differential between gross and net output is a result of losses and other drains such as various electrical systems for example pumps, fans, and blowers which are part of the fuel cell system. Each fuel cell stack generates power at 60-65 V DC nominal, and they are connected in series for 120-139 V DC output. The 120-volt power from the fuel cell system is converted to power in the range of 300 V DC to approximately 350 V DC .

It was expected that the two fuel cells would operate in series to produce about 14kW gross power however a majority of the system losses occur within the fuel cells, thus the projected design net output of the fuel cells was expected to be about 10kW. After the initial bench testing of the fuel cell stacks it was found that the stacks have a net output 11.7kW including the electrical loss. A comparison is shown in Table 1.

Table 1 Comparison Between Previous Generation and Next Generation Fuel Cell Systems.

	Previous Generation	Next Generation
Purge Valves	1	4
End Caps	Stainless Steel	Plastic
Rods holding stacks together	Stainless Steel	Titanium
Flow Channels	4	16
How Many Fuel Cells	1	2, in series
Total kilowatts	4.2 kW	11.7 kW
How Many Cells	64	100 per stack
Stack Controllers	1	1, running both stacks
Blowers	1	1 for both stacks
Hydrogen Source/Generation	Tank Storage	Sodium borohydride
Hydrogen Storage	12 tanks in backseat	1 tank in trunk

Fuel Cell System Design by Recon Industrial Controls Corporation

Recon designed and manufactured the fuel cell control and data acquisition system for testing a high power (> 1KW) PEM (Proton Exchange Membrane) fuel cell stack. The fuel cell that was tested consisted of cells, connected electrically in series, producing an open circuit voltage (no load) of 1V per cell and a full load voltage of 0.65V per cell at the rated operating current of 100A. The fuel cell stack used a humidified air stream and dry hydrogen. Temperature management was best achieved through water-cooling.

The Recon test system consisted of a Recon single stack controller, and a Recon Data Acquisition Module. The controller was connected to a PC through a serial port to monitor fuel cell operating parameters and to adjust operating parameters. The Data Acquisition Module, which was connected to the serial connection, also allowed continuous data storage. Software consisted of Recon Controller Interface Software, Data Acquisition Interface and Charting Software, and Recon Performance Analysis Software.

The controller consisted of a control circuit board including analog signal conditioning, 12-bit A/D conversion, an 8-bit microcontroller, and output drivers. This controller connected to peripheral control devices with solid-state relays. The controller continuously measures fuel cell stack current (0-140A), fuel cell stack voltage (0-1.1V per cell), fuel cell stack temperature (0-100C), and the cooling water temperature (0-100C). Current was measured using a resistive shunt and temperatures were measured with thermistor sensors. Other system parameters, such as battery voltage,

were also measured. Output devices controlled included a DC motor/compressor, water pump, cooling fans, hydrogen shutoff and purge valves, and a water-recycling pump.

One primary function of the controller is to provide the proper airflow to meet the stoichiometric oxygen needs based on fuel cell stack current. The controller provides a nonlinear control function to adjust the compressor motor voltage via PWM (Pulse Width Modulation) duty cycle regulation according to changes in fuel cell stack current. The period and duration of hydrogen purging is also controlled according to current to discharge water and any hydrogen impurities that collect on the cathodic (hydrogen) side. A control loop also runs based on fuel cell stack and cooling water temperature to control water pump and cooling fan speed to maintain proper operating conditions.

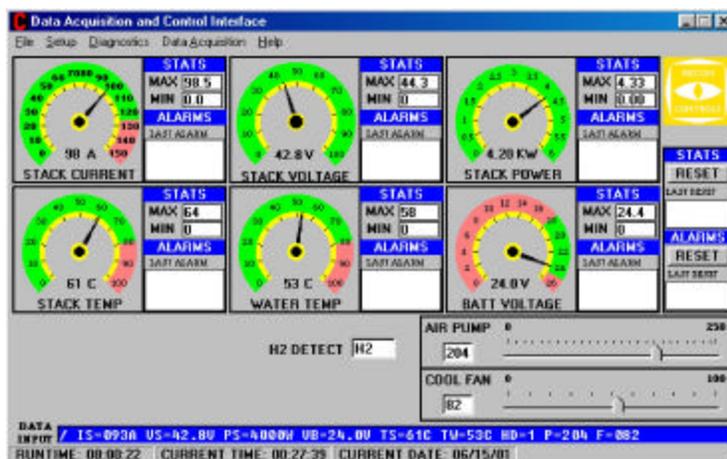


Figure 11. Interface Software for monitoring fuel Cell Stack Operating Variables.

The Recon Controller Interface software shown in Figure 11 provided a graphical operator interface to monitor fuel cell stack operating variables, to setup operating parameters to optimize performance, and to provide manual control of system devices for diagnostic purposes. Operating parameters also included operating variable limits to allow for automatic system shutdown. The software also included a strip chart recorder screen and file logging to the PC hard drive with time and date stamps.

The Recon Data Log Module shown in Figure 12 was used to allow the continuous logging of date/time stamped data. Data was later downloaded with Recon's interface software. The Data Log Module contained a real-time clock calendar with a backup battery and flash memory to retain data memory if module power is lost.



Figure 12. Data Log Module for the continuous logging of date/time stamped data.

The module was setup to log samples of controller-acquired data with a sample period of 1 minute. The downloaded data was saved in PC memory to then be displayed on the included strip chart or exported in a text format that is easily imported into Excel or into Recon Fuel Cell Analyzer software.

The included stripchart shown in Figure 13 allowed data to be viewed graphically. The X-axis displays the time and date of the acquired samples and the Y-axis of the top chart corresponds to fuel cell stack current (red trace) and fuel cell stack voltage (blue trace). The Y-axis of the bottom chart corresponds to cooling water temperature (green trace) and fuel cell stack temperature (orange trace). Zoom and pan functions allowed both X- and Y- axis to be expanded and to allow scrolling through data.

The section of data shown represents fuel cell operation during one day of testing. The times at which all 4 traces are at the zero baseline indicates times at which the system was turned off, possibly for control parameter changes. There are 2 occurrences of zero stack current (red trace) with no corresponding zeroing of temperature (green and orange traces). At these times the system was running, but no power (zero load) was being drawn from the stack. This allowed the fuel cell stack voltage to reach its open circuit voltage of about 0.1 volts per cell as indicated by the fuel cell stack voltage (blue trace) at the times of zero fuel cell stack current. Due to the internal impedance of the fuel cell stack, current increases cause a corresponding decrease in voltage.

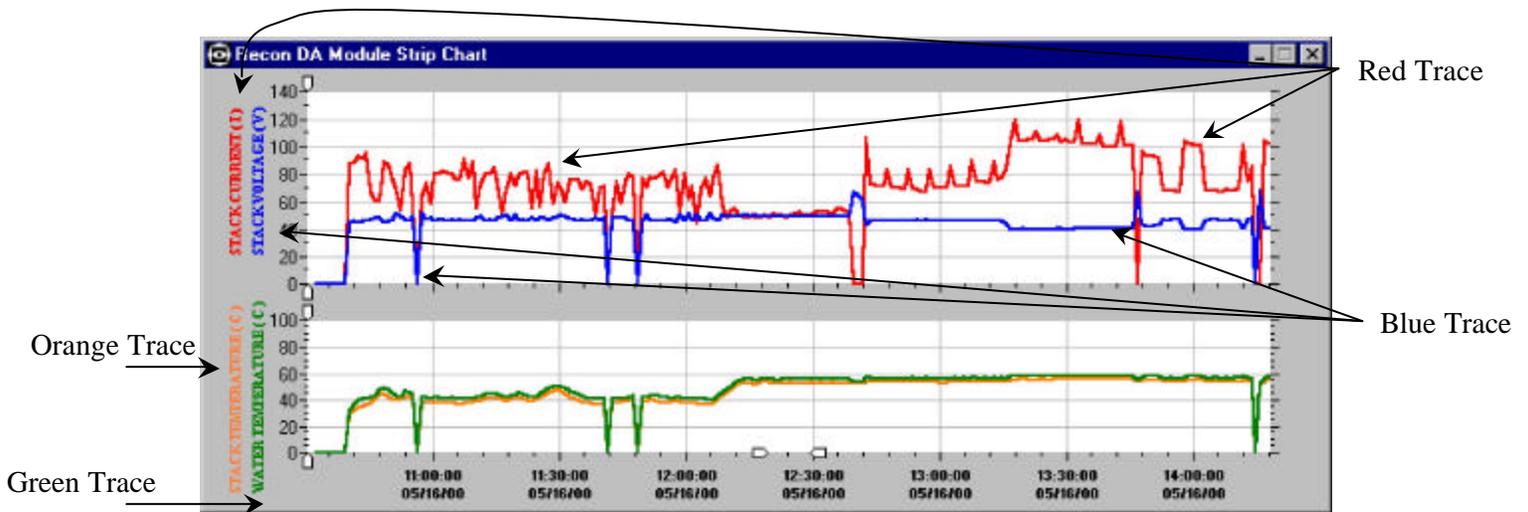


Figure 13. The calculation of the fuel cell stack voltage. Graph shown is based on 60 cell stack.

Cooling water temperature (green trace) is indicated as always about 2 degrees over fuel cell stack temperature (orange trace). This difference could be due to errors introduced by the use of two different sensor types or due to the placement of the fuel cell stack temperature sensor due to the stacks non-uniform temperature profile. As indicated at the beginning of the data, temperature stability was reached within about 6 minutes of system power up and current draw. The temperature rise about half way through the data is likely due to changes made in system cooling parameters.

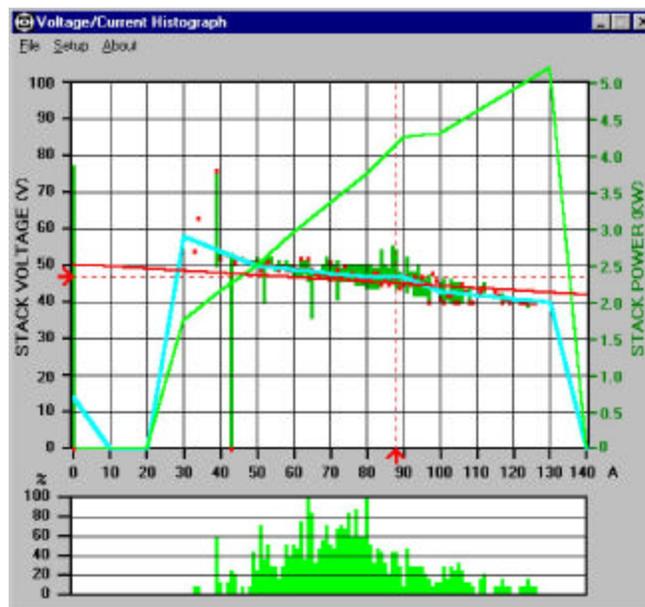


Figure 14. Polarization chart showing the voltage vs. current cross plotted against the stack power.

The data file created by the Data Log Module interface software was then imported into the Recon Fuel Cell Performance Analyzer software, which creates a polarization chart (voltage vs. current) and current histogram and is shown in Figure 14. The X-axis represents the fuel cell stack current, from zero current (no load) to high currents (full rated load and over) for both the top polarization chart and the bottom current histogram. For every data sample logged from the controller the fuel cell stack voltage point is placed on the chart at the corresponding current position.

The vertical bars on the lower graph indicate low to high ranges of voltages measured for particular currents, due to varying fuel cell performance (most influenced by membrane humidity and temperature changes).

The blue trace follows the averaged voltage values measured and the green trace follows the average fuel cell stack power values calculated from the measured currents and voltages. The straight red line is a best-fit line whose slope and position is calculated by means of a least squared method to provide an average of fuel cell stack impedance.¹²

Estimated Ability Calculations

To begin with, an overall efficiency must be calculated. During design meeting estimates of other systems overall efficiencies were substituted for an actual value. This practice continued until the initial bench tests were performed. The efficiency was measured in the lab using several system characteristics. This of course is a rather conservative estimate and was better estimated after actual testing.

To calculate the projected output as a result of the hydrogen generator working with the fuel cells requires several steps.

STEP 1 For use in the Hydrogen-On-Demand™ generator, a 20 percent by weight solution of sodium borohydride is stabilized by 1 percent by weight sodium hydroxide. Therefore the fuel composition by weight is 20 percent sodium borohydride (NaBH₄), one percent sodium hydroxide stabilizer, and 79 percent water. At this concentration, the system is capable of supplying hydrogen at about 520 liters of hydrogen gas per liter of fuel.

STEP 2 The fuel tank can hold up to 32 gallons (121.1 liters) of the 20 percent sodium borohydride (NaBH₄) fuel solution. Multiplying the hydrogen generation rate of 520 L/min by the storage capacity of the fuel solution of 121.1 L yields the total hydrogen supplied by the generator of 62,972 liters of hydrogen.

STEP 3 The fuel cell system requires an average hydrogen flow of 4.9 scfm (138.8 L/min). The flow rate will also vary with time; flow will increase when the fuel cell system purges. The actual flow rate will vary proportionally with the load. Dividing the net hydrogen from the generator 62,972L by the fuel cell usage of 138.8 L/min it results in a total runtime for the fuel cells of 454 minutes or approximately 7.57 hours.

Therefore for one tank of 20 percent sodium borohydride (NaBH_4) fuel solution the fuel cells can operate continuously for 7.57 hours.

STEP 4 The net output of the fuel cells is 11.7 kW. Of that net voltage there are approximately 2 to 2.5 kW of system loads and efficiency losses. After taking into consideration all these loads the total usable power supplied to the motor is on the order of 9.6 kW. Multiplying the total continuous runtime of 7.57 hrs by the net output (after loads and efficiency losses) to the motor of 9.6 kW, the result is 72,672 watt-hours.

Consumption rates and run times

The hydrogen generator has an average generation rate of about 130 L/min until the flow of sodium borohydride solution is ceased. This 130 L/min is slightly less than the demand from the fuel cell of 138.8 L/min. The generator produces about 520 L hydrogen per liter of fuel thus at 150 L of hydrogen per minute the generator can run continuously on the 121.1 L fuel storage for 8.07 hours. If the fuel cells ran continuously they would consume this amount of hydrogen in 7.57 hours. Thus in order for the generator to produce all of the hydrogen for use in the fuel cells there will be a delay of approximately 0.5 hours. This offset can be easily accommodated in a completely integrated system through the usage of a battery pack.

CONCLUSIONS

The two fuel cells that were used are larger than the one from the previous generation. However, several subsystems have been combined to reduce redundancy, instead of having true parallel system components such as air blowers, controllers, humidifiers, and heat exchangers they have been increased in size and the fuel cells linked in a series system. For example only one blower is used for both stacks. Also a new custom built fully automated controller monitors all fuel cell functions as well as optimizing flow rates and purge cycles thus increasing overall system efficiency. The control board is a critical component and is responsible for varying the speed of the air compressor and for continuously performing safety checks of the voltage and temperature of the fuel cell stacks.

Finally light weight materials have been used to not only reduce the weight of the fuel cell stacks but also the size. The original design of the fuel cells used many stainless steel components. The fuel cell stainless steel rods and end plates were replaced with titanium rods and plastic end plates. The plates that were used inside of previous generation fuel cell were made of stainless steel. The net weight savings per fuel cell stack was approximately 50 pounds. The result of the upgrades is that each fuel cell stack is now 10" X 12" X 24" and weighs only 75 pounds. A comparison between the two systems is shown in Table 1.

Safety in handling of the sodium borohydride should not be a problem if the necessary precautions are taken following the MSDS sheets in Appendix 1. The solution of the sodium borohydride in water can achieve hydrogen production rates between 120 to 150 L/min at about 100 psi. The products of power production will be pure water,

hydrogen gas, and sodium borate. It is possible to recycle the spent fuel sodium borate back into sodium borohydride, however this process is outside the scope of this project. Future research may show an inexpensive process to regenerate sodium borohydride from sodium borate, which would be a significant step in demonstrating this fuel as a renewable resource.

Regulatory controls will make sure pressure within the system not exceed safe levels. It was shown in this report that the output of the generator comes close (~.5 hour) to equaling the fuel cell output.

Potential refinements of the system include possibly lowering the weight of the entire system or possibly applying the system to a technology that would reasonably benefit from its use.

While it is clear that the objectives of this project have been met, this project has opened many broader issues as to the uses of this apparatus. Numerous projects could stem from this research. There are also technological advancements that will occur in generations to come.

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- ¹² Reference Information Provided by Joe Barbetta 6/27/01.
- ¹³ Sigma Chemical Company, Material Safety Data Sheets, Copyrighted information was used by permission of Sigma-Aldrich Co, June 2001
- ¹⁴ Rohm and Haas Literature, Data Sheets for VenPure® Powder, 9/93

APPENDIX 1 - MSDS AND PRODUCT INFORMATION ¹³



SIGMA-ALDRICH

Material Safety Data Sheet

Date Printed: 06/05/2001
Date Updated: 02/21/2001
Version 1.10

Section 1 - Product and Company Information

Product Name	SODIUM BOROHYDRIDE, POWDER, 98%		
Product Number	452882		
Brand	Aldrich Chemical		
Company	Sigma-Aldrich		
Street Address	3050 Spruce Street		
City, State, Zip, Country	St. Louis, MO 63103 US		
Technical Phone:	314 771 5765	Emergency Phone:	414 273 3850 Ext.5996
Fax:	800 325 5052		

Section 2 - Composition/Information on Ingredient

Substance Name	CAS #	SARA 313
SODIUM BOROHYDRIDE	16940-66-2	No
Formula	BH ₄ .Na	
Synonyms	Borohydrure de sodium (French), Borol, Hidkitex DF, Sodium borohydrate, Sodium hydroborate, Sodium tetrahydridoborate(1-), Sodium tetrahydroborate(1-)	

Section 3 - Hazards Identification

Emergency Overview

Flammable (USA) Highly Flammable (EU). Highly Toxic (USA) Very Toxic (EU).
Contact with water liberates extremely flammable gases. Toxic in contact with skin and if swallowed. Very toxic by inhalation. Causes burns.

HMIS Rating

Health: 3 Flammability: 0 Reactivity: 2 Special Hazard(s) : Water reactive

NFPA Rating

Health: 3 Flammability: 0 Reactivity: 2 Special Hazard(s) : Water reactive

For additional information on toxicity, please refer to Section 11.

Section 4 - First Aid Measures

Immediate Treatment - Work Site

In case of contact, immediately flush eyes or skin with copious amounts of water for at least 15 minutes while removing contaminated clothing and shoes.

Oral Exposure

If swallowed, wash out mouth with water provided person is conscious. Call a physician.

Inhalation Exposure

If inhaled, remove to fresh air. If not breathing give artificial respiration. If breathing is difficult, give oxygen.

Section 5 - Fire Fighting Measures

Explosion Hazards

Material readily reacts with water generating flammable and/or explosive hydrogen gas

Conditions of Flammability

Reacts with water to liberate flammable and/or explosive gas. Reacts with metals to liberate flammable hydrogen gas.

Explosion Limits: Lower: 3.02 %

Autoignition Temp: N/A

Extinguishing Media

Suitable

Dry chemical powder.

Unsuitable

Do not use water, foam, or carbon dioxide. Do not use halocarbon extinguishers.

Firefighting

Protective Equipment

Wear self-contained breathing apparatus and protective clothing to prevent contact with skin and eyes.

Specific Hazard(s)

Flammable solid. Emits toxic fumes under fire conditions.

Section 6 - Accidental Release Measures

Procedure to be Followed in Case of Leak or Spill

Evacuate area.

Procedure(s) of Personal Precaution(s)

Wear self-contained breathing apparatus, rubber boots, and heavy rubber gloves.

Methods for Cleaning Up

Sweep up, place in a bag and hold for waste disposal. Avoid raising dust. Ventilate area and wash spill site after material pickup is complete.

Section 7 - Handling and Storage

Handling

User Exposure

Do not breathe dust. Do not get in eyes, on skin, on clothing. Avoid prolonged or repeated exposure.

Storage

Suitable

Keep tightly closed. Store in a cool dry place.

Incompatible Materials

Reacts violently with water. Do not allow contact with water.

Special Requirements

Do not allow water to enter container because of violent reaction. Protect from heat.

Section 8 - Exposure Controls / PPE

Engineering Controls

Safety shower and eye bath. Use only in a chemical fume hood.

Personal Protective Equipment

Respiratory

NIOSH/MSHA-approved respirator.

Hand

Compatible chemical-resistant gloves.

Eye

Chemical safety goggles.

Skin-Specific

Chemical resistant apron.

Other

Faceshield (8-inch minimum).

General Hygiene Measures

Wash contaminated clothing before reuse. Wash thoroughly after handling.

Section 9 - Physical/Chemical Properties

Appearance

Physical State	Color
Solid	White

Molecular Weight: 37.83 AMU

pH	N/A
BP/BP Range	N/A
MP/MP Range	300 °C
Freezing Point	N/A
Vapor Pressure	N/A
Vapor Density	N/A
Saturated Vapor Conc.	N/A
SG/Density	1.074 g/cm ³
Bulk Density	N/A
Odor Threshold	N/A
Volatile%	N/A
VOC Content	N/A
Water Content	N/A
Solvent Content	N/A
Evaporation Rate	N/A
Viscosity	N/A
Partition Coefficient	N/A
Decomposition Temp.	N/A
Flash Point °F	N/A
Flash Point °C	N/A
Explosion Limits	Lower: 3.02 %
Autoignition Temp	N/A
Solubility	N/A

Section 10 - Stability and Reactivity

Stability**Stable**

Stable.

Conditions to Avoid

Do not allow water to enter container because of violent reaction. Protect from heat.

Materials to Avoid

Oxidizing agents, Chemically active metals, Acids, Reacts violently with water.

Hazardous Decomposition Products**Hazardous Decomposition Products**

Borane/boron oxides, Sodium oxide, Hydrogen gas.

Hazardous Decomposition Products Formed Upon Contact with Water

Material readily reacts with water generating flammable and/or explosive hydrogen gas.

Hazardous Polymerization
Hazardous Polymerization
Will not occur.

Section 11 - Toxicological Information

Route of Exposure

Skin Contact

Causes burns.

Skin Absorption

Toxic if absorbed through skin.

Eye Contact

Causes burns.

Inhalation

May be fatal if inhaled. Material is extremely destructive to the tissue of the mucous membranes and upper respiratory tract.

Ingestion

Toxic if swallowed.

Signs and Symptoms of Exposure

Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract, eyes, and skin. Inhalation may result in spasm, inflammation and edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Symptoms of exposure may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

RTECS Number: ED3325000

Toxicity Data

Oral - Rat: 162 mg/kg(LD50)

Skin - Rabbit: 230 mg/kg(LD50)

Inhalation - Rat: 36 mg/m3(LC50)

Oral - Rat: 162 mg/kg (LD50)

Remarks: Sense Organs and Special Senses (Nose, Eye, Ear, and Taste):Eye:Other.

Behavioral:Convulsions or effect on seizure threshold.

Lungs, Thorax, or Respiration:Structural or functional change in trachea or bronchi.

Inhalation - Rat: 36 mg/m3 (LC50)

Remarks: Sense Organs and Special Senses (Nose, Eye, Ear, and Taste):Eye:Other.

Behavioral:Convulsions or effect on seizure threshold.

Lungs, Thorax, or Respiration:Structural or functional change in trachea or bronchi.

Intraperitoneal - Rat: 18 MG/KG (LD50)

Subcutaneous - Rat: 177 MG/KG (LD50)

Remarks: Sense Organs and Special Senses (Nose, Eye, Ear, and Taste):Eye:Other.

Behavioral:Convulsions or effect on seizure threshold.

Lungs, Thorax, or Respiration:Structural or functional change in trachea or bronchi.

Oral - Mouse: 50 mg/kg (LD50)

Remarks: Sense Organs and Special Senses (Nose, Eye, Ear, and Taste):Eye:Other.

Behavioral:Convulsions or effect on seizure threshold.

Lungs, Thorax, or Respiration:Structural or functional change in trachea or bronchi.

Oral - Rabbit: 50 mg/kg (LD50)

Remarks: Sense Organs and Special Senses (Nose, Eye, Ear, and Taste):Eye:Other.

Behavioral:Convulsions or effect on seizure threshold.

Lungs, Thorax, or Respiration:Structural or functional change in trachea or bronchi.

Skin - Rabbit: 230 mg/kg (LD50)

Remarks: Sense Organs and Special Senses (Nose, Eye, Ear, and Taste):Eye:Other.

Behavioral:Convulsions or effect on seizure threshold.

Lungs, Thorax, or Respiration:Structural or functional change in trachea or bronchi.

Section 12 - Ecological Information

No data available.

Additional Results/Data from Relevant Scientific Experiments

Avoid contamination of the environment

Section 13 - Disposal Considerations

Appropriate Method of Disposal of Substance or Preparation

Contact a licensed professional waste disposal service to dispose of this material.
Observe all federal, state, and local environmental regulations.

Section 14 - Transport Information

DOT

Proper Shipping Name: Sodium borohydride
UN#: 1426
Class: 4.3
Packing Group: Packing Group I
Hazard Label: Dangerous when wet.
PIH: Not PIH

IATA

Proper Shipping Name: Sodium borohydride
IATA Number: 1426
Hazard Class: 4.3
Packing Group: I
Not Allowed - Aircraft: Cargo aircraft only. Not permitted on passenger aircraft.

Section 15 - Regulatory Information

EU Additional Classification

Symbol of Danger: F+ T+

Indication of Danger

Highly Flammable, Very toxic.

Risk Statements **R:** 15 24/25 26 34

Contact with water liberates extremely flammable gases. Toxic in contact with skin and if swallowed. Very toxic by inhalation.
Causes burns.

Safety Statements **S:** 22 26 36/37/39 43 45

Do not breathe dust. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Wear suitable protective clothing, gloves, and eye/face protection. In case of fire, use dry sand. Never use water. In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

US Classification and Label Text**Indication of Danger**

Flammable (USA) Highly Flammable (EU). Highly Toxic (USA) Very Toxic (EU).

Risk Statements

Contact with water liberates extremely flammable gases. Toxic in contact with skin and if swallowed. Very toxic by inhalation.
Causes burns.

Safety Statements

Do not breathe dust. In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

United States Regulatory Information

SARA Listed: No

TSCA Inventory Item: Yes

Canada Regulatory Information**WHMIS Classification**

This product has been classified in accordance with the hazard criteria of the CPR, and the MSDS contains all the information

Aldrich Chemical - 452882

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Page 5

required by the CPR.

Section 16 - Other Information

Warranty

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. Sigma-Aldrich Inc., shall not be held liable for any damage resulting from handling or from contact with the above product. See reverse side of invoice or packing slip for additional terms and conditions of sale. Copyright 2001 Sigma-Aldrich Co. License granted to make unlimited paper copies for internal use only.



SIGMA-ALDRICH

Material Safety Data Sheet

Date Printed: 06/05/2001
Date Updated: 10/15/1999
Version 1.0

Section 1 - Product and Company Information

Product Name	SODIUM METABORATE TETRAHYDRATE		
Product Number	S0251		
Brand	Sigma Chemical		
Company	Sigma-Aldrich		
Street Address	3050 Spruce Street		
City, State, Zip, Country	St. Louis, MO, 63103, US		
Technical Phone:	Emergency Phone:	414 273 3850 Ext.5996	
Fax	800 325 5052		

Section 2 - Composition/Information on Ingredient

Substance Name	CAS #	SARA 313
SODIUM METABORATE TETRAHYDRATE	10555-76-7	No
Formula		
Synonyms		

Section 3 - Hazards Identification

Emergency Overview

Irritant.
Irritating to eyes, respiratory system, and skin.

HMIS Rating

Health: 1 Flammability: 0 Reactivity: 0

NFPA Ratings

Flammability: 0 Reactivity: 0

For additional information on toxicity, please refer to Section 11.

Section 4 - First Aid Measures

Oral Exposure

If swallowed, wash out mouth with water provided person is conscious. Call a physician.

Inhalation Exposure

If inhaled, remove to fresh air. If not breathing give artificial respiration. If breathing is difficult, give oxygen.

Dermal Exposure

In case of contact, immediately wash skin with soap and copious amounts of water.

Eye Exposure

In case of contact, immediately flush eyes with copious amounts of water for at least 15 minutes.

Molecular Weight: N/A

<u>Property</u>	<u>Value</u>
pH	N/A
BP/BP Range	N/A
MP/MP Range	N/A
Freezing Point	N/A
Vapor Pressure	N/A
Vapor Density	N/A
Saturated Vapor Conc.	N/A
SG/Density	N/A
Bulk Density	N/A
Odor Threshold	N/A
Volatile%	N/A
VOC Content	N/A
Water Content	N/A
Solvent Content	N/A
Evaporation Rate	N/A
Viscosity	N/A
Partition Coefficient	N/A
Decomposition Temp.	N/A
Flash Point *F	N/A
Flash Point *C	N/A
Explosion Limits	N/A
Autoignition Temp	N/A
Solubility	N/A

Section 10 - Stability and Reactivity

Stability

Stable

Stable.

Conditions of Instability

May decompose on exposure to air Reacts with carbon dioxide in air to form sodium carbonate and sodium tetraborate

Materials to Avoid

Strong oxidizing agents.

Hazardous Decomposition Products

Hazardous Decomposition Products

Sodium/sodium oxides, Boron oxides.

Hazardous Polymerization

Hazardous Polymerization

Will not occur.

Section 11 - Toxicological Information

Route of Exposure

Inhalation

Material is irritating to mucous membranes and upper respiratory tract.

Multiple Routes

May be harmful by inhalation, ingestion, or skin absorption. Causes eye and skin irritation.

Signs and Symptoms of Exposure

To the best of our knowledge, the chemical, physical, and toxicological properties have not been thoroughly investigated.

RTECS Number:

Section 12 - Ecological Information

Section 13 - Disposal Considerations

Appropriate Method of Disposal of Substance or Preparation

For small quantities: cautiously add to a large stirred excess of water. Adjust the pH to neutral, separate any insoluble solids or liquids and package them for hazardous waste disposal. Flush the aqueous solution down the drain with plenty of water. The hydrolysis and neutralization reactions may generate heat and fumes which can be controlled by the rate of addition. Observe all federal, state, and local environmental regulations.

Section 14 - Transport Information

DOT

Proper Shipping Name: None

Non-Hazardous for Transport: This substance is considered to be non-hazardous for transport.

IATA

Proper Shipping Name: None

Non-Hazardous for Air Transport: Non-hazardous for air transport.

Section 15 - Regulatory Information

EU Additional Classification

Symbol of Danger: Xi

Indication of Danger

Irritant.

Risk Statements R: 36/37/38

Irritating to eyes, respiratory system, and skin.

Safety Statements S: 26 36

In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Wear suitable protective clothing.

US Classification and Label Text

Indication of Danger

Irritant.

Risk Statements

Irritating to eyes, respiratory system, and skin.

Safety Statements

In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Wear suitable protective clothing.

United States Regulatory Information

SARA 313 Listed: No

Section 16 - Other Information

Warranty

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. Sigma-Aldrich Inc., shall not be held liable for any damage resulting from handling or from contact with the above product. See reverse side of invoice or packing slip for additional terms and conditions of sale. Copyright 1999 Sigma-Aldrich Co. License granted to make unlimited paper copies for internal use only.

APPENDIX 2 – COMMERCIALY AVAILABLE SODIUM BOROHYDRIDE PRODUCT INFORMATION¹⁴



VenPure® Powder

Product Description

A proprietary formulation of sodium borohydride designed for purification of organic chemicals. VenPure powder contains an anticaking agent and trace quantities of sodium hydroxide (NaOH) and sodium metaborate (NaBO₂).

Typical Properties

Molecular weight:	37.85
Specific gravity, 25°C:	1.074
Form:	White crystalline solid
Melting point:	Decomposes above 400°C without melting
Typical solubility: (g/100 g solvent @ 25° C)	
Water	55.0
Liquid ammonia	104.0
Ethanol (reacts slowly)	4.0
Dimethyl ether of diethylene glycol	5.5
Dimethylacetamide	14.0
Isopropylamine	6.0

Application

VenPure powder is a water soluble reducing agent exhibiting unique activity in organic and inorganic chemical systems. Due to ease of handling and high reducing power, this product is used extensively in a broad range of industrial applications. Major industrial applications include: reduction of carbonyl and peroxy compounds, reduction of metal ions, and removal of color, odor, and oxidation precursors in organic chemical products. Additional information on these and other applications of sodium borohydride is available from Morton International.

Recommended Use Level

The amount of VenPure products required for reduction depends upon the compound being reduced and the general reaction conditions. Reductions with VenPure products normally proceed as represented in the following typical reactions:



Under ideal reaction conditions, one mole of NaBH₄ (37.8 g) will reduce four moles of an aldehyde or ketone to the corresponding alcohol or provide eight electrons for the reduction of metallic ions. Thus, 37.8 pounds of VenPure powder will theoretically reduce 176 pounds of acetaldehyde to ethanol or 235 pounds of nickel ion (Ni²⁺) to the free metal. However, under industrial reaction conditions, the use level of VenPure products will be greater than the stoichiometric level due to losses from side reactions, primarily hydrolysis/solvolysis. Consequently, the actual use level should be experimentally derived.

The rate and efficiency of reductions with VenPure products are dependent upon the concentration of the reactants, temperature, and pH. The reaction rate is generally more rapid at higher temperatures, however, satisfactory results can be obtained at low and moderate temperatures. One of the most important parameters in the efficient use of VenPure products is pH control. Reductions with VenPure products should be carried out at a pH of 10-11 or higher. If the pH is below 10, consumption of VenPure products may increase due to hydrolysis. Additional information on optimizing VenPure products use levels and reaction conditions is available from the VenPure Technical Services Group.

Availability

Six VenPure product forms are commercially available: VenPure powder, VenPure granules (10/40), VenPure granules (+10), VenPure caplets, VenPure solution (a stabilized water solution of 12% NaBH₄ and 40% NaOH) and VenPure K powder (KBH₄).

® registered trademarks of Rohm and Haas Company.

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To the best of our knowledge the information contained herein is correct. All products may present unknown health hazards and should be used with caution. Although certain hazards are described herein, we cannot guarantee that these are the only hazards which exist. Final determination of suitability of the product is the sole responsibility of the user. Users of the product should satisfy themselves that the conditions and methods of use assure that the product is used safely. NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE OR ANY OTHER NATURE ARE MADE HEREUNDER WITH RESPECT TO THE INFORMATION CONTAINED HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. Nothing herein is intended as a recommendation to use our products so as to infringe any patent. We assume no liability for customer's violation of patent or other rights. The customer should make his own patent investigation relative to his proposed use.

Toxicity and First Aid

1. Composition:

98% NaBH₄ minimum

CAS Number

16940-66-2

2. VenPure powder has an acute dermal LD₅₀ on dry skin of 4-8 gm/kg, and is not a skin sensitizer. However, severe irritation and skin burns may result when VenPure products come into contact with moist skin. The acute oral LD₅₀ of VenPure powder is 50-100 mg/kg. Sodium metaborate, the reaction or decomposition product of VenPure products, is considered slightly toxic orally (LD₅₀, 2000-4000 mg/kg), and non-toxic dermally.

3. All precautions should be taken against ingestion of VenPure products, inhalation of the dust, or contact with skin. In case of accidental contact with skin, the particles should be brushed off and the affected areas flooded with water. VenPure products and concentrated solutions of these are *very corrosive to the eye* and should be handled according to generally accepted procedures for corrosive chemicals. In case of accidental contact, *flush eyes with water and seek immediate medical attention.*

Product Handling and Storage

1. Personal Protection:

Protective rubber gloves, clothing and safety goggles should always be worn when handling VenPure products and their solutions. Dust respirators should be worn when handling VenPure dry products.

2. Handling:

VenPure dry products should be handled in the same manner as other *Division 4.3 Dangerous When Wet* materials. It is stable to shock and does not ignite on contact with moisture, but form a dihydrate which slowly hydrolyzes. VenPure powder is hygroscopic and should not be unnecessarily exposed to air or moisture. It is stable indefinitely in dry air or in sealed containers.

Solutions of VenPure products will decompose and evolve hydrogen if overheated, subjected to neutral or acidic pH conditions, or brought into contact with oxidizers, metal salts or finely divided metallic precipitates of nickel, cobalt, copper, or iron. Reactions of VenPure products should always be carried out in adequately vented vessels with standard provisions for pressure relief. A nitrogen atmosphere is also recommended. Reactions of VenPure products should only be conducted in explosion proof equipment with proper grounding.

3. Storage:

VenPure products should be stored in closed containers in a dry, cool and well ventilated area and kept separate from acids and oxidizing materials. Partially consumed containers should be carefully resealed.

Solutions of VenPure products may be stored in stainless steel, mild steel, or approved fiberglass vessels that are adequately vented to the outside atmosphere. Caustic solutions of VenPure products should not be stored in vessels which react with sodium hydroxide, such as aluminum.

4. Firefighting:

VenPure powder is classified as a *Division 4.3 Dangerous When Wet* solid. It will ignite in air from a free flame due to hydrogen formed from thermal decomposition, continuing to burn as hydrogen is evolved. VenPure products can also be ignited if they are brought in contact with acids, oxidizers, or transition metal salts or precipitates. The non-volatile product of combustion is sodium metaborate. Fire extinguishers available where VenPure products are stored or used should only be of the dry chemical type, such as those manufactured by Ansul Chemical Company or equivalent. **WATER, CARBON DIOXIDE OR HALOCARBON EXTINGUISHERS MUST NOT BE USED ON VENPURE PRODUCTS FIRES.**

Waste Disposal

1. VenPure products and their solutions containing unreacted NaBH₄ can be disposed of (hydrolyzed) by dissolving in a large excess of water, followed by slow addition of a dilute solution of acetic acid or acetone in a well ventilated area. Provisions should be made to safely vent hydrogen gas given off during the decomposition of unreacted VenPure products and their solutions. VenPure products and their solutions should not be flushed to the sewer.

2. In case of accidental spillage, absorb the VenPure product with an inert material such as sand or dolomite. Absorbed material should be allowed to weather in an outdoor disposal area or hydrolyzed as per above.

3. Any vessels which have been used for storage or reactions of VenPure products should be carefully vented, drained, and adequately flushed with water and purged with nitrogen and air before any repair operations are undertaken. Exposure to an open flame (e.g., welding torch) should be avoided. Morton International, Inc., should be contacted for more detailed procedures.

APPENDIX 3 - ADDITIONAL TECHNICAL PICTORIAL



Figure 15 Millennium Cell, Inc. Hydrogen Generator Model 1 designed to power a 1.2 kW fuel cell.



Figure 16 Millennium Cell, Inc. hydrogen generator test stand used to develop hydrogen generator design prior to installation in the vehicle.



Figure 17 Hydrogen generator test stand condenser water reclamation tank used to develop hydrogen generator design prior to installation in the vehicle.



Figure 18 Fuel cell air compressor assembly.



Figure 19 Custom stainless steel radiator to cool fuel cell system.



Figure 20 Two 7 kW (gross) fuel cell stacks installed in a vehicle.

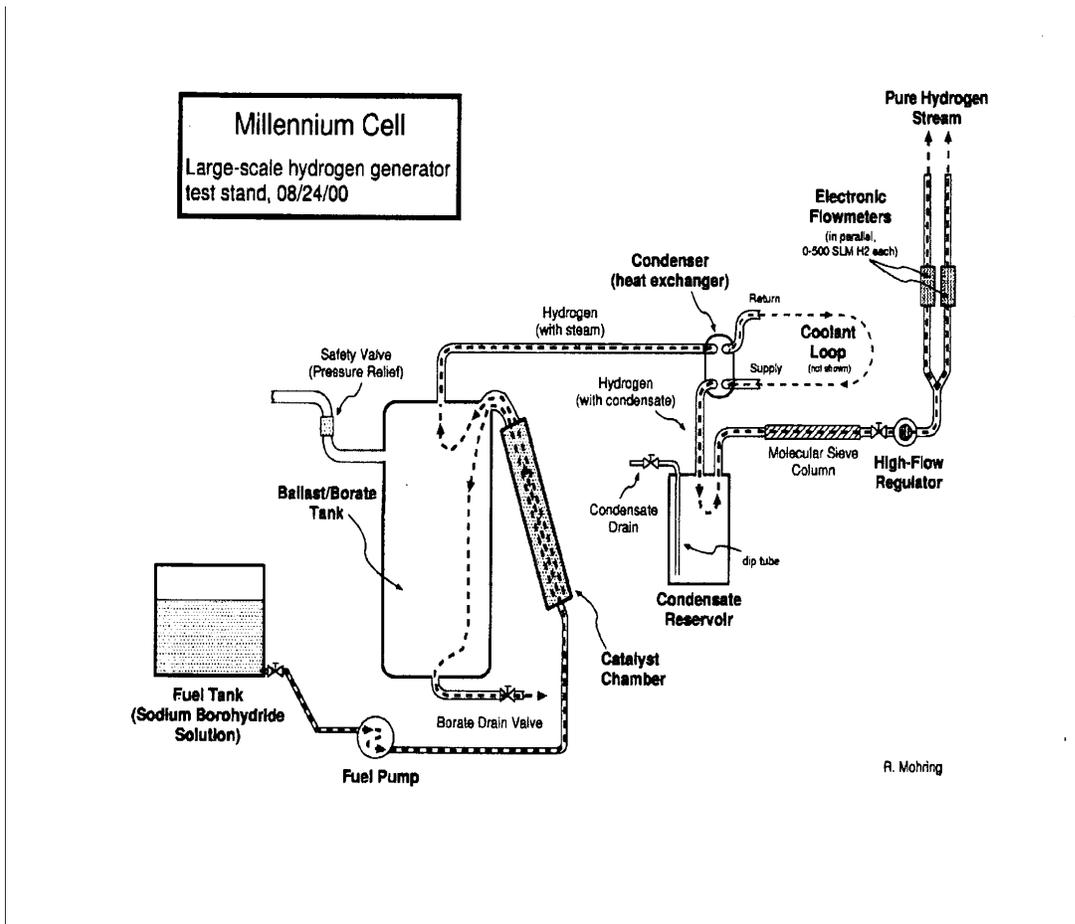


Figure 21 Schematic of Hydrogen Generation System