- The pressure used may have been higher than tolerable by the tool. (The owners manual was consulted, and no maximum pressure was listed. The highest pressure the airbrush was subjected to was approximately 40 psi for a few very brief periods, but the normal operating pressure was below 20 psi.) This does not seem to be a very likely cause for the fracture of the nozzle.

In order to rectify this situation, the tool needs to be either repaired or replaced. The vendor from whom the airbrush was purchased has been contacted about repairs. Jerry Hestbeck was also contacted and asked to retrieve the purchase order information. Other methods of spraying the catalyst solution are also being investigated such as atomizers and aerosol can kits.

This incident was believed to be a result of bad luck rather than negligence, for extreme care was taken while using, cleaning and disassembling the airbrush. Even though many precautions were taken, the following measures will be taken in the future to ensure that this does not occur again:

- Oxygen will not be used to propel the solution. An inert gas such as nitrogen or argon will be used instead.
- Only very dilute liquid catalyst solution will be used.
- High pressures will not be allowed to build up in the system.

Review of MSDS

Chemicals we are concerned with include lead, isopropyl alcohol, hydrogen, oxygen, glycerol, tetra butyl ammonium hydroxide, hydrogen peroxide, Vulcan XC 72-R, Nafion solution (from Aldrich), hydrosulfuric acid and sodium chloride. Lead can be poisonous if swallowed. Isopropyl alcohol is a potentially toxic chemical if swallowed or inhaled for long periods of time. In addition isopropyl alcohol should be used carefully as it may be ignited by heat; its flash point is 11.7 ° C. Acute effects of high exposure to hydrogen include nausea and dizziness. If hydrogen is inhaled the person should be removed to fresh air immediately, and a physician should be called if major symptoms arise. If oxygen is inhaled the person should be kept alert and a physician should be called. When exposed to glycerol it can cause headaches. Glycerol should not be mixed with hydrogen peroxide for a violent reaction can occur. This pertains only to a glycerol and H_2O_2 mixture and not other combinations such as glycerol and isopropanol. Tetrabutyl ammonium hydroxide is a relatively stable base that can cause severe irritation of the skin. This is a highly toxic chemical and should always be used in a hood. In the case of hydrogen peroxide, it is unstable with heat or contamination. If inhaled drink plenty of water while skin contact does not pose problems. Vulcan XC 72-R in powder form can cause irritation to lungs. The hazards of Nafion solution at this point are unknown because a MSDS does not exist. Sulfuric acid can cause skin burns if it exists in high molar concentrations. Sodium chloride is a highly stable chemical and poses no great health hazard.

Baseline Safety Assessment

When operating or testing the single fuel cell, one should always be aware of the potential of hydrogen and oxygen mixing in the fuel cell stand. When working with glycerol, hydrogen peroxide should not be mixed because a violent reaction can occur between these chemicals according to the MSDS for glycerol. Some basic safety precautions include wearing safety goggles and non-loose clothing. In addition group members should be aware of the location of the fire extinguisher, eye wash stand and sink. The potential for burns is mainly present when using the furnace and when operating the fuel cell stand. Attached to the stand are gas humidifiers which become extremely hot. In the near future we will be using lead wire to conduct resistance tests so this is a reminder to use appropriate gloves for lead and other harmful chemicals. When using the air brush make sure to use Argon as the compressed gas and not oxygen. In addition when preparing the ink powder (Vulcan XC 72-R) for the catalyst, proper facial mask needs to be worn.

List of Additional Figures

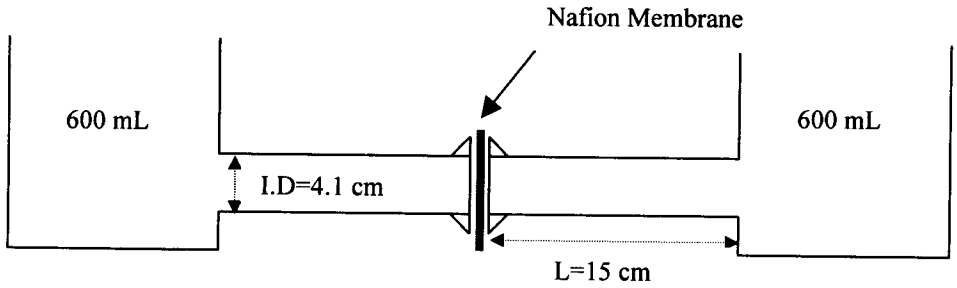

Figure A.1: Schematic of an Electrochemical H-cell.

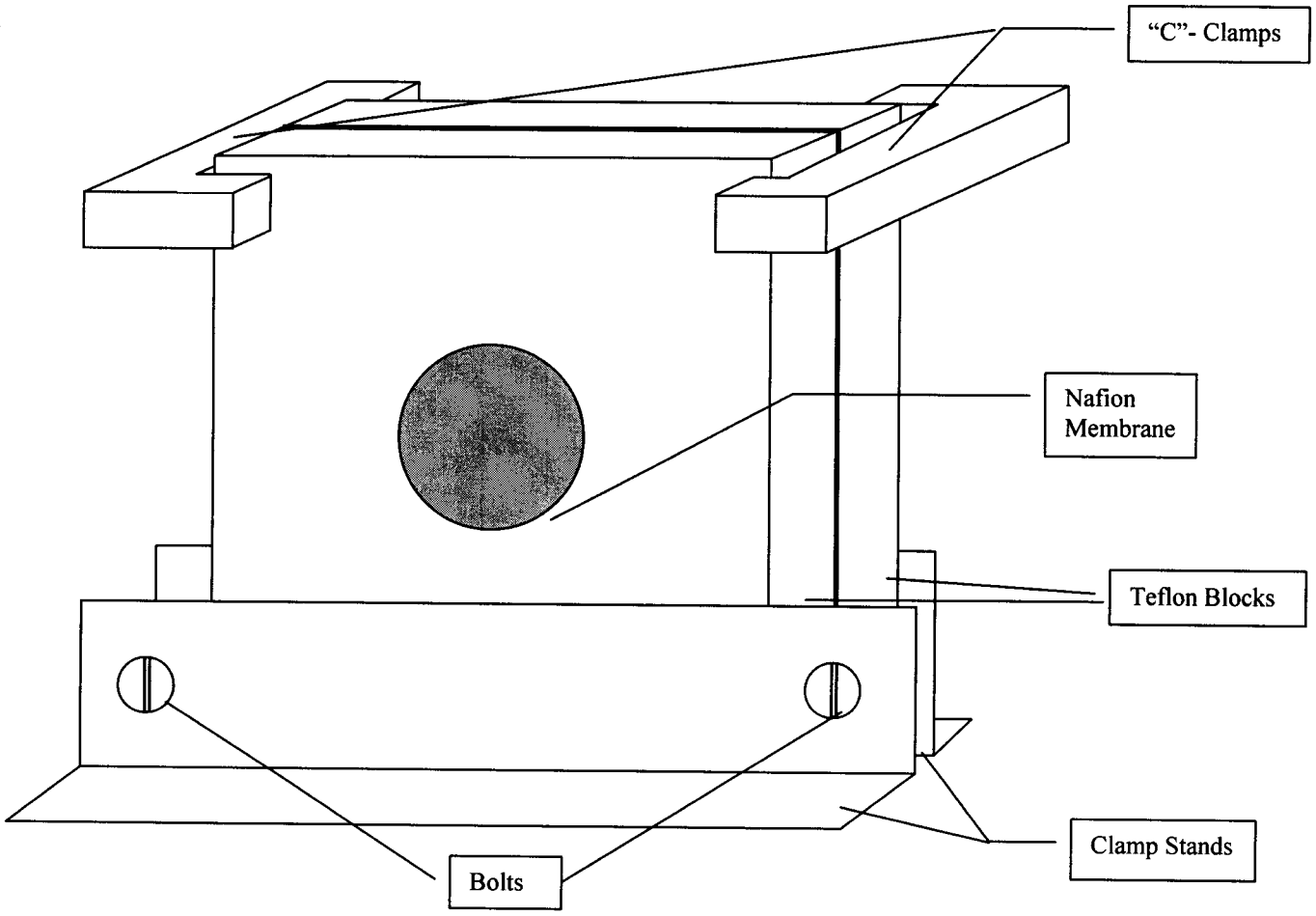

Figure A.2: Schematic of Catalyst Application Assembly

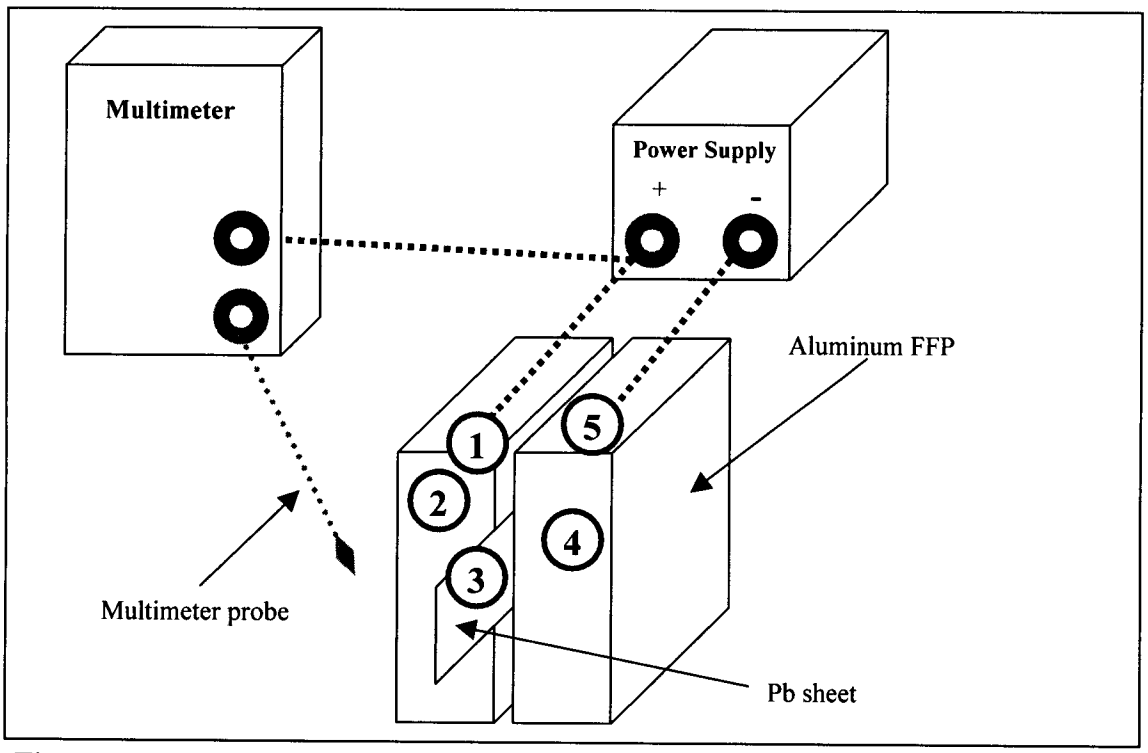

Figure A.3: A schematic of the setup of the resistance test with the multimeter connected in series to the positive outlet of the power supply along with points where the voltage readings were taken.

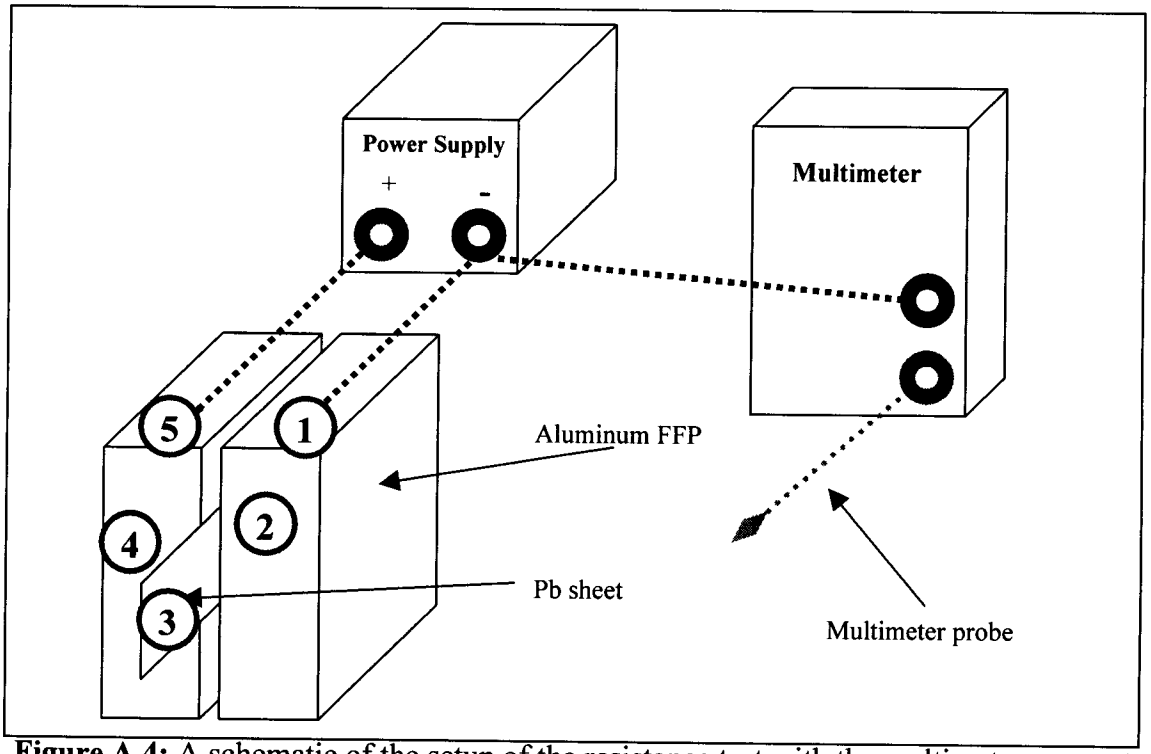

Figure A.4: A schematic of the setup of the resistance test with the multimeter connected in series to the negative outlet of the power supply along with points where the voltage readings were taken.

References:

1. Ferarra et al., "Fall Quarter Proposal", DOC/FCP-109706 (1997).

2. Wilson, M.S., Valerio, J.A., Gottesfeld, S., "Low Platinum Loading Electrodes for Polymer Electrolyte Fuel Cells Fabricated using Thermoplastic Ionomers," Electrochim Acta. 40 (1995) 355-363.

3. Bailey, L, Ed Goldman, and Michael Nguyen, "Winter Quarter Final Report", 1997

Appendix A: Raw Data from Aluminum FFPs Resistance Test

Table A.1 below lists the voltage reading when the multimeter was connected in series with the positive power outlet of the power supply during the first trial of the resistance test.

Table A.1: Voltage readings for trial 1 when the multimeter is connected to the positive power outlet.

	Voltage reading (V)
Clip #1	0.014
Al FFP #1	0.039
Lead	0.045
Al FFP #2	0.051
Clip #2	0.133

Table A.2 below lists the voltage reading when the multimeter was connected in series with the negative power outlet of the power supply during the first trial of the resistance test.

Table A.2: Voltage readings for trial 1 when the multimeter is connected to the negative power outlet.

	Voltage reading (V)
Clip #1	0.014
Al FFP #1	0.094
Lead	0.100
Al FFP #2	0.105
Clip #2	0.126

Table A.3 below lists the voltage reading when the multimeter was connected in series with the positive power outlet of the power supply during the second trial of the resistance test.

Table A.3: Voltage readings for trial 2 when the multimeter is connected to the positive power outlet.

	Voltage reading (V)
Clip #1	0.014
Al FFP #1	0.038
Lead	0.043
Al FFP #2	0.049
Clip #2	0.070

Table A.4 below lists the voltage reading when the multimeter was connected in series with the negative power outlet of the power supply during the second trial of the resistance test.

Table A.4: Voltage readings for trial 2 when the multimeter is connected to the negative power outlet.

	Voltage reading (V)
Clip #1	0.014
Al FFP #1	0.106
Lead	0.111
Al FFP #2	0.116
Clip #2	0.151

Table A.5 below lists the voltage reading when the multimeter was connected in series with the negative power outlet of the power supply during the third trial of the resistance test.

Table A.5: Voltage readings for trial 3 when the multimeter is connected to the negative power outlet.

	Voltage reading (V)
Clip #1	0.623
Al FFP #1	0.56
Lead	0.53
Al FFP #2	0.537

Appendix B: Checklist for the Electrochemical H-cell Test

NOTE: The H-cell is very fragile. Avoid any unnecessary move or impact.

1. Position each half of the H-cell on a clean, dry, and soft surface.
2. Place an O-ring into the groove of the glass joint.
3. Place the membrane onto the O-ring. Center it carefully.
4. Bring the other half of the H-cell closer so that its groove aligns with the O-ring on the other side of the membrane. Hold the two halves together tightly.
5. Put the clamp on the glass joints.
6. Tighten the screw on the clamp until the tape mark is reached.
7. Add about 25 mL of DI water slowly into one side of the H-cell. Tilt the cell slightly to observe for sight of leakage.
8. A. If no leak is found, proceed to step 9.

   B. If a leak is found, tighten the clamp or remove the membrane and reposition it.
9. Pour out the water.
10. Add an equivalent amount of DI water to the other side of the cell. Repeat the above leak test in steps 7-9.
11. Make sure that the cell is dry before adding electrolyte, in this case sulfuric acid, into the cell.
12. Set up the Pt electrodes and the rest of the proton conductivity test as instructed by Professor Stuve. Details to be completed later.

Appendix C: Schematics of the Entire System and the Single Fuel Cell Test Stand

Note: Several valves are not needed if we test our MEAs using dead-end hydrogen.

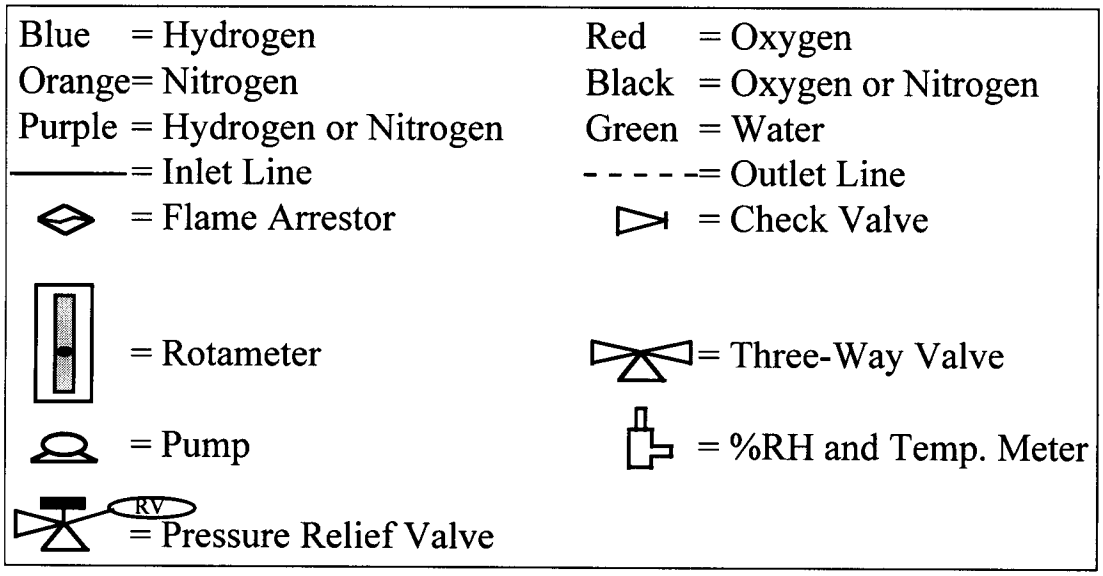

Figure C.1: Legend for Figures C-2 and C-3

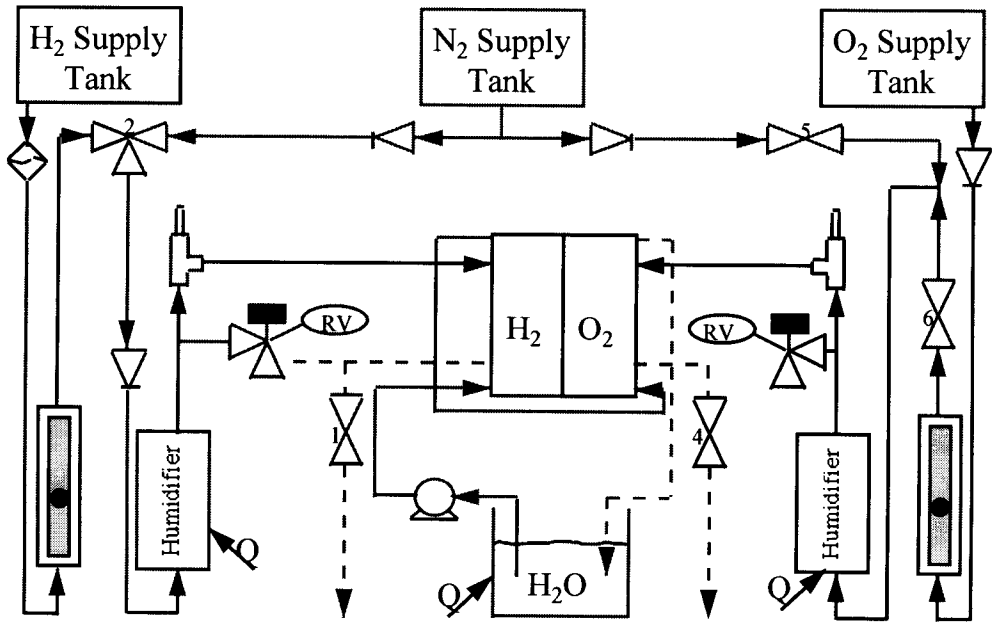

Figure C.2: Flow Diagram of Fuel Cell Test Stand

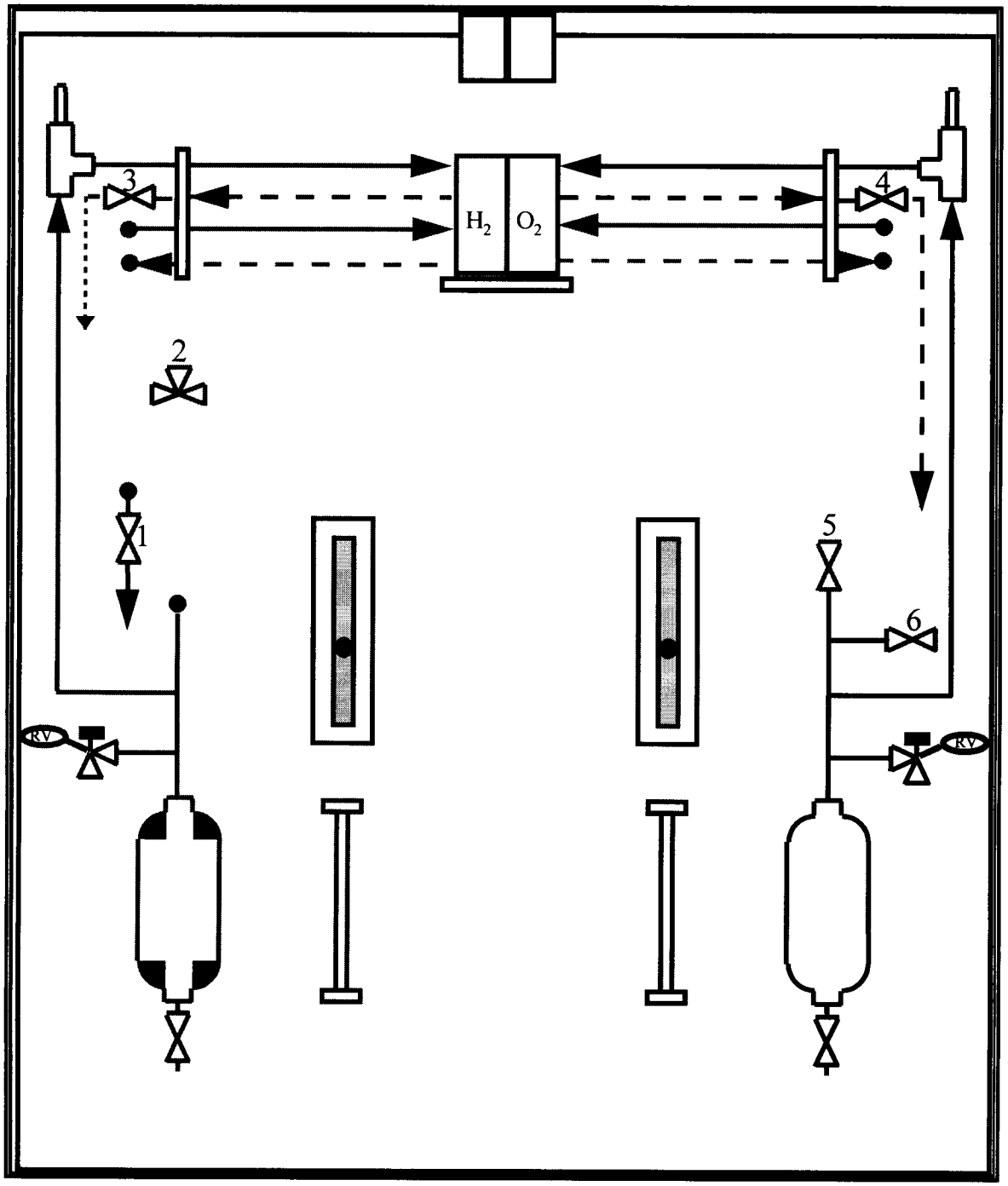

Figure C.3: Front of the Test Stand

Valve 1 = hydrogen purge; **Valve 2** = three-way hydrogen/nitrogen selection valve;

Valve 3 = hydrogen flow control valve; **Valve 4** = oxygen flow control valve;

Valve 5 = nitrogen feed for oxygen side; **Valve 6** = oxygen feed

Appendix D: Standard Operating Procedure for Test Cell and Stand (With Dead-Ended Hydrogen Operation)

0. Emergency Shutdown Method

0.1 For a runaway cell, immediately turn off main hydrogen and oxygen cylinder valves, making sure you cannot be burned from this action. If this is not possible, turn off gases with small valves on front of apparatus.

0.2 Flush both sides of gas delivery system with nitrogen gas.

0.3 Disconnect electrical power supply to band heaters and water supply system.

NOTE: Under no circumstance are hydrogen and oxygen gases allowed to contact one another while at elevated temperatures or in the presence of the platinum catalyst.

1. Test Cell Assembly

1.1 Flow field plates (FFPs) should be coated with ElectroDag and be in good condition. If either has been dropped or severely scratched, it should be checked to ensure that it can still function properly and then repaired.

1.2 O-rings are installed in each half, each should be retained in the gland even

1.3 A piece of Teflon coated carbon paper is placed in the recess in one of the halves, which has been suspended with the flow field facing up (the corner of a box works well for this).

1.4 A prepared membrane is then placed over the carbon paper, centering the electrode as well as possible on the flow area.

1.5 An identical piece of carbon paper is placed on top of the membrane, again centering as well as possible.

1.6 The second half of the cell is then brought down on this sandwich of parts. As it is brought down, it is imperative that the carbon paper finds its way into and remains in the recesses above the flow fields. IF IT IS SUSPECTED THAT THE CARBON PAPER IS NOT SEATED PROPERLY, DO NOT CONTINUE!!! PROCEEDING WITH THIS MISASSEMBLY WILL RESULT IN MEMBRANE DAMAGE AND A VERY EXPLOSIVE MIXTURE OF HYDROGEN AND OXYGEN

1.7 After it has been made certain that the carbon papers are seated and the Nafion is making contact all around with the O-rings, bolt up can now be done. Using the nylon bolts, insert them all finger tight, then use the deep socket wrench only to tighten then in a circular pattern. Tighten to about 4 foot-pounds each (not very tight). Never use a wrench other than a box type or socket, as the nylon could be easily damaged.

1.8 After bolt up, unit is placed on the shelf of the test stand and all connections are made.

1.8.1 Attach feed gas tubes (these must connect to one of the center ports on the test cell.

1.8.2 Attach purge gas tubes to the other center ports.

1.8.3 Attach water feed and discharge lines to the outer ports on the test cell.

1.8.4 Connect resistive load to the cell.

1.8.5 Connect voltmeter to the cell.

2. Start Up of the System

2.1 Leak testing must first be performed.

2.1.1 With all valves closed, open main nitrogen cylinder valve 1/4 turn. Regulator pressure should not exceed 45 psi.

2.1.2 SLOWLY crack the valves feeding into the water bottles, one side of the cell at a time. This will charge the system through the cell. After charging, leave this valve open 1-2 turns.

2.1.3 Crack each exit valve for about 5 seconds to bleed air and charge entire system with feed gas. Then close these valves securely.

2.1.4 Close main cylinder valves tightly. A leak, if any, is now evidenced by a falling pressure reading on the high pressure gauge. If this pressure is falling faster than 100 psi/min., a substantial leak is present, and should be located/corrected. Detect and fix the leak before continuing.

2.2 Disconnect gas supply to the test cell

2.2.1 Release gas pressure to both sides of the cell using the purge valves

2.2.2 Disconnect the gas supply lines to the cell

2.3 Start heating

2.3.1 Start water bath for heating of the cell, and turn on bottle heater VariACs to a setting of '60 V'. It may take up to 1/2 hour for substantial temperature rise.

2.3.2 Flow nitrogen gas through the system to heat the gas delivery lines (nitrogen will be vented to atmosphere at the cell gas supply lines).

2.4 When gas delivery lines reach the desired temperature and the nitrogen gas venting from the lines appears humid (collect it on a piece of paper), cell testing can begin.

3. Testing

3.1 Reconnect the gas supply lines to the test cell and disconnect the exit lines from the cell.

3.2 Start heating/cooling water flow to the cell.

3.3 Allow the test cell to come up to the desired temperature while nitrogen gas flow continues.

NOTE: The membrane must remain humidified. Do not heat the cell to a higher temperature than that of the humidified feed gas!

3.4 Reconnect the exit gas lines to the cell.

3.6 Be sure that valve 3 is at least partially open and start the hydrogen recirculation pump.

3.7 Open the main valves on the hydrogen and oxygen supply tanks.

3.7.1 Adjust the pressure regulators to the desired pressure (less than 45 psig!)

3.7.2 BE SURE that the pressure on the nitrogen regulator is higher than that on the hydrogen or oxygen regulators. This ensures that there is no cross-flow of these gases through the nitrogen supply system.

3.7.3 Perform a second leak test as described previously in 2.1

3.8 Purge the nitrogen from both sides of the system using the two vent valves.

3.9 The cell should now be operational

3.10 Select different loads (resistance) to measure the voltage. Voltages are measured, and the resultant voltage versus current graph produced.

3.11 NOTE: Make sure water level and temperature in humidifier tanks is high enough to maintain good operation. This is one of the major problems with long term operation of the cell.

4. Shut Down of the system

4.1 Turn off hydrogen main cylinder valve, allow system hydrogen pressure to come to atmospheric pressure.

4.2 Turn off oxygen main cylinder valve, allow system to come to atmospheric.

4.3 Turn off heating units, water bath and bottle heaters.

4.4 Purge both sides of the system with nitrogen

4.5 Turn off the voltmeter.

4.6 Allow unit to cool sufficiently before handling

4.7 Disassemble the cell and inspect components as desired.

5. Storage of the Test Cell

5.1 Unit should be stored disassembled with membrane faces secure from any form of damage

5.2 Over time, the ElectroDag coating may need to be replaced. This should be done with all components removed from the blocks, save the plugs in the water jacket. Follow the procedure appearing in previous works.

5.3 Membranes should be stored under deionized water in labeled containers. If it is necessary to store more than one in the same container, then they should all be discernible in some way. A suggested method is to cut very small notches into the periphery of the membrane, the part falling outside the sealing area.

5.4 Dry membranes should not be placed into the test cell. When put into use, the membrane will swell. Only fully saturated membranes should be installed.

6. Other Notes

6.1 Carbon papers are cut using cookie cutter and nylon block in the press. Only run the press to 1500 psi, this is sufficient to cut through the paper. In the future, a new cutter will be made from a more suitable material (steel) once the correct size is known.

6.2 Nafion discs are cut from DRY stock with cutter an nylon block in press. This time around 2000 psi should be used. Don't worry if they appear small, upon saturation with water it will grow to the appropriate size.

6.3 It is unknown at this point the actual longevity of this cell. The ElectroDag coating is supposed to render the areas treated quite inert to this type of environment, but it unknown whether or not sufficient coating is being achieved.

Appendix E: Fuel Cell Membrane Fabrication Procedure

This procedure is a newly revised version of last year [1]. Initially, the procedure was basically a reproduction of the procedure outlined in [2]. Preliminary results from last year using this procedure were very encouraging. However, reproduction of these results was problematic.

Preparation:

Nafion Membrane

Materials:

- Nafion 117 membrane (MW = 1100, 0.18 mil thickness)
- 3% aqueous peroxide solution
- 1 M sodium chloride solution

Procedure:

- Cut membrane with the Aluminum cutter.
- Boil in a 3% by weight aqueous peroxide solution for 1 hour
    - performed to clean the membrane and saturate it with water
- Boil in approximately 1 M aqueous sodium chloride solution for 1 hour
    - performed to ensure complete conversion to the sodium form

Catalyst Ink

Materials:

- Nafion solution from Aldrich, 5% solubilized Nafion by weight
- Platinum catalyst
    - 10 % platinum by weight on carbon black Vulcan XC 72-R, the Electrosynthesis Company

- Tetrabutyl ammonium Hydroxide solution (TBOH), 1 M from Aldrich

- Glycerol, from Aldrich

- Isopropanol

Procedure:

- Combine Nafion solution and platinum catalyst material (1:3 Nafion to platinum ratio by mass) in a small flask

- Add isopropanol and glycerol until "desired" consistency is achieved

-**Sonicate a minimum of 2 hours**. This helps ensure the catalyst is distributed evenly in the Nafion solution

- Add a 25 to 50% excess of the TBOH solution to create an alkaline mixture to minimize heat degradation of the membrane.

- TBOH must react with each sulfonate group in the Nafion solution. Knowing the amount of Nafion present, its molecular weight (1100), and the molecular weight of TBOH, the correct amount can be calculated.

- Sonicate a minimum of 2 hours

Membrane Assembly:

Materials:

- Small oven

- Airbrush

- Catalyst ink assembled above

- Copper gaskets from 2.75" Conflat flange

- Small screw-type hose clamps

- Lab press with heated platens

- Previously prepared Nafion Membrane

- Two Teflon (6in x 6in) sheets.

Procedure:

- Preheat small oven and press platens to 150 ^{o}C

- Clamp Nafion membrane between gaskets, centering it carefully

- At this point, there are two options, both of which have been utilized successfully

    1. Apply the catalyst ink onto the membrane surface using the airbrush.

    2. Place two to three drops of Nafion solution on one side the membrane, followed by a paintbrush application of the catalyst ink.

    NOTE: The addition of Nafion solution prior to the catalyst application aids adhesion of the ink.

-Place assembly in front of the small oven and allow it to dry for about 30 sec

-Repeat the above two steps to the other side of the membrane

- Remove membrane from clamp assembly

- Place membrane between Teflon sheets

- Press membrane and Teflon at 150 ^{o}C and 30 atm for five minutes

- Remove the membrane and boil in 0.5 M Sulfuric Acid solution for 2 hours to convert the membrane completely into the proton form.

- Boil in deionized water for 2 hours

- Allow to cool