



Procedure for Performing PEM Single Cell Testing



Work Performed by
the Florida Solar Energy Center
under DOE Contract # DE-FC36-06G016028

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FSEC's Procedures For Performing PEM Single Cell Testing

Test protocol for Cell Performance Tests
Work Performed Under
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EXECUTIVE SUMMARY

This document defines, in detail, a test protocol for performing proton exchange membrane (PEM) single cell testing. Development of this test protocol is a part of the requirements of FSEC's Task 5, Characterize MEA Performance, under DOE Contract #DE-FC36-06GO16028. This test protocol has been evaluated and is used at FSEC for single-cell testing.

Under this contract, FSEC will perform single-cell tests to assess the relative merits of the candidate membranes that are submitted by the Task 1 team members. These tests will be conducted at operating conditions that are of interest to the DOE and, where appropriate, will be run using identical procedures on all tested cells. Task 1 team members that are continuing beyond the Go – No Go milestone are requested to review this document and identify “conditions that are of concern.” FSEC will iterate with these Task 1 team members to resolve these concerns

Researchers following the test protocol will be able to reproduce results that have been obtained by FSEC.

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1.1 Introduction to the Test Protocol for PEM Cell Tests

PEM single-cell testing for performance, durability and accelerated stress is conducted on a “Fee for Service” basis at a number of government as well as commercial laboratories. These services are available for the individual Task 1 team members with customer-specific protocols to meet their individual testing requirements. This document defines the PEM test procedures that will be used at FSEC for conducting the screening tests that are required under FSEC’s Task 5 “Characterize performance of MEA” of the DOE program DE-PS36-6095020. The purpose of this testing is to assess the relative merits of the candidate membranes that are submitted by the Task 1 team members. These tests will be conducted at operating conditions that are of interest to the DOE. The goal is to show the current capability of these membranes rather than simply to provide a Go/No Go rating against the Program Requirements. A further objective of this protocol is to clearly define the operating conditions to which the individual membranes will be subjected. This allows the details to be understood by the DOE; and, with this information, each team member can set limits of processing and operation based on the capabilities of their individual membranes. To the extent that all membranes are tested to the same protocol, the results provide a fair and accurate evaluation of the performance capabilities of each configuration. It is understood that the fabrication history for the membranes, as well as the characteristics of the interfacing cell components, the cell assembly procedures, and the cell operating history can have a significant impact on the cell performance. The impact of these “histories” is normalized in this protocol by using identical procedures on all the cells.

FSEC has followed this protocol on numerous cells and this test protocol produced repeatable results.

1.2 Components to be Delivered

Task 1 team members deliver sample membranes developed under this DOE program. A sufficient number of pieces sized 12 cm by 12 cm (4 ¾ inches by 4 ¾ inches) will be submitted to enable the tests to be completed. These membranes, along with the fabricators’ constraints for further processing, are represented to the DOE as the Task 1 team members’ best effort of performance under this program.

1.3 Details of the Cell

The test CCM is fabricated using membranes provided by the Task 1 team members. Prior to fabrication of the CCM, membrane samples will be examined for uniformity (no bubbles, debris, fogginess, stretches, cracks, and thickness variations), gas crossover, and flexibility to ensure the membranes are suitable for testing. Sample pieces will be cut from the membrane and sent to Bekktech, LLC and Scribner Associates for conductivity testing and to Material Science at the University of Central Florida for SEM/EDAX characterization. A diagram depicting how each supplied membrane will be used is shown in Figure 1.1. The CCM is fabricated by spraying a catalyst ink (ionomer, Pt/C, methanol mixture) on a membrane to achieve a nominal loading of 0.4 mg Pt/cm² of TEC10E50E -- Pt 50 wt% catalyst supported on a high surface area carbon (SA = 800 m²/g) supplied by TKK. FSEC has achieved baseline performance by mixing the catalyst

with 32% 1100 EW Nafion[®] using methanol and then spraying using a nitrogen-driven spray gun, which is carried by a numerically-controlled X/Z plotter. The CCM is then dried at 100 °C, heat-treated at 136 °C and compressively pressed 5.1 atm (75 psi) compressive pressure for five minutes, and then protonated using a 0.5 M sulfuric acid solution and washed in distilled water. A second CCM and two coupons are made at the same time, with identical processing. This second CCM and one coupon will be returned to the membrane fabricator untested for their evaluation and analysis. FSEC will communicate with the team members about the fabrication constraints for each membrane and an attempt will be made to accommodate the membrane requirements and the supplier wishes, while still achieving comparable results. Depending on the membrane composition and the supplier requirements, it may be necessary to adjust the type of ionomer, ionomer loading, drying temperature, hot press temperatures, and solvent.

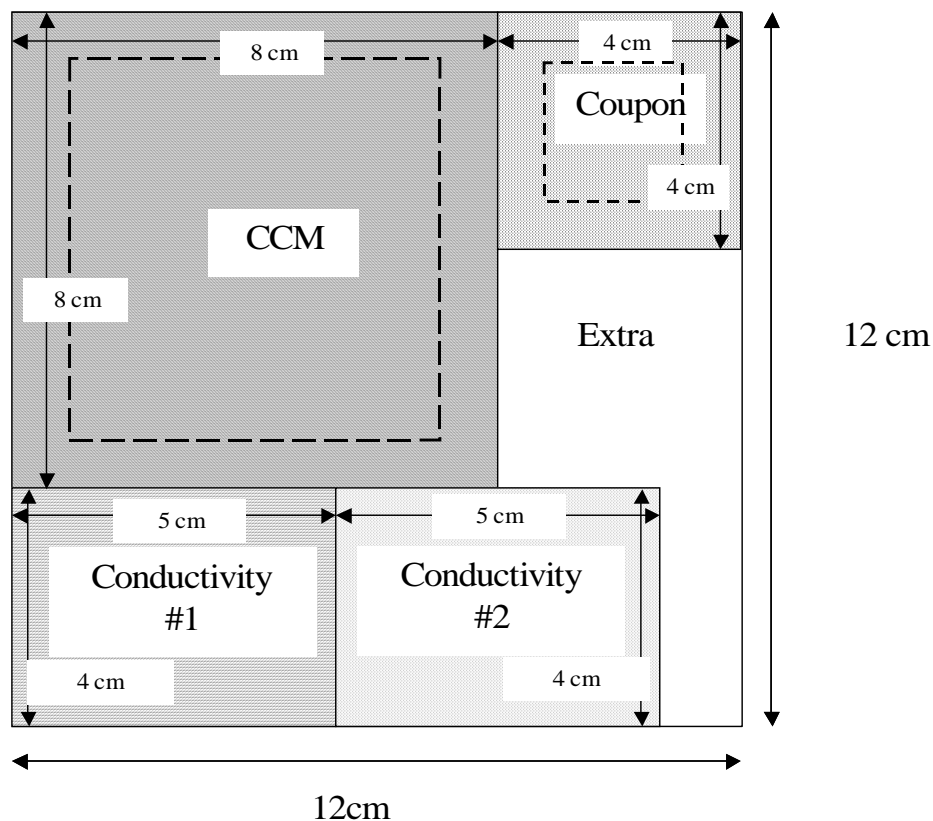


Figure 1.1. Depiction of membrane sample pieces. CCM and coupon pieces will be sprayed at the same time. Conductivity pieces will be sent to Scribner and Associates and Bekktech for testing.

The single-cell hardware (25 cm²) used for this program was manufactured by Fuel Cells Technologies Incorporated (FCT) www.fuelcellstechnologies.com. It consists of a pair of graphite bipolar plates with a serpentine flow pattern, gold-coated current collector plates, and aluminum pressure plates. The design of the anode bipolar plate flow field has been rotated 90° to give a cross flow pattern and the dimensions of the serpentine flow field for both anode and cathode have been modified (width and depth) to decrease pressure drop. All other aspects of this hardware are as originally designed by FCT.

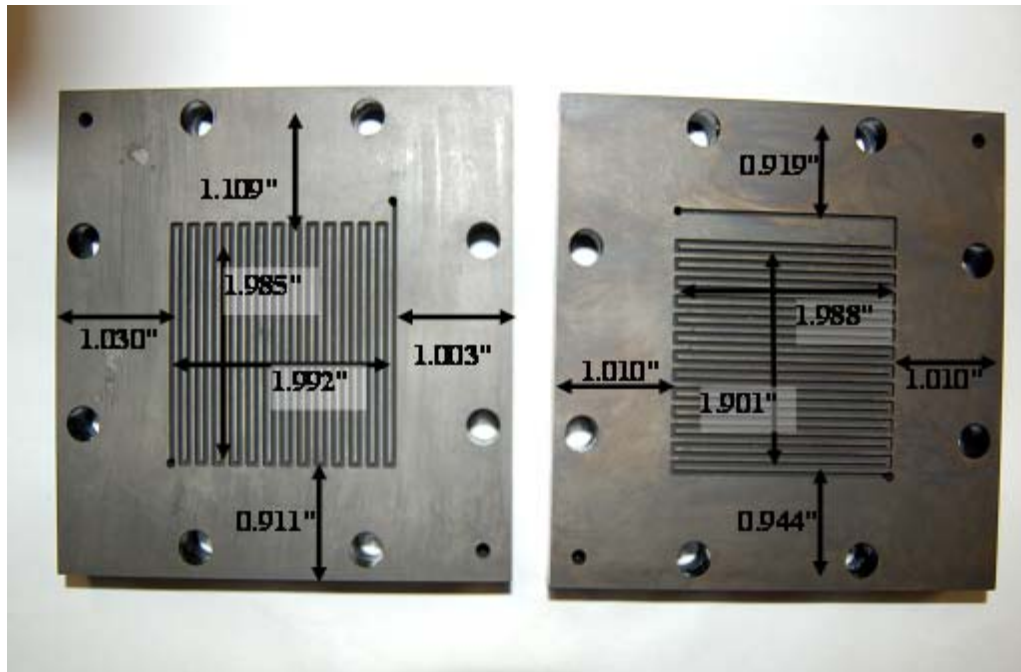


Figure 1.2. Bipolar plates (anode, left and cathode, right) showing single serpentine flow field.

All the MEAs for these cells use type 10BB gas-diffusion layers purchased from SGL Carbon (Sigracet®). Each of these gas-diffusion layers were tested for through-plane permeability prior to use, following the procedure in Appendix A. The minimum acceptable permeability for the cathode GDL is given by a Gurley Number of 0.024 L/min/cm H₂O/cm². The anode GDL permeability is allowed to be much lower. The cell seals are made of a stack-up of Teflon® gaskets with the thickness of the gaskets thinner than the cell assembly giving a “pinch” of 230 to 250 μm (9-10 mils).

1.4 Membrane, CCM, and GDL Thickness Measurement

The thickness of the membrane, CCM, and GDLs are measured using a Mitutoyo Gauge (Figure 1.3). Component thicknesses are defined using average of at least nine readings evaluated over the entire component at locations shown on the template in Figure 1.4. The membrane, CCM, or GDL is sandwiched between thin sheets of Teflon® of known thickness when making the measurements. The template is used to maintain the consistency of the location of successive measurements. The Teflon® sheets are measured first and then the membrane, CCM, or GDL is inserted between them and a second set of readings is taken. Subtracting the first reading from the second provides the component thickness.



Figure 1.3. Mitutoyo Gauge for measuring thickness of membranes, CCMs, MEAs, and GDLs

| | | | |
|---|---|---|---|
| X | X | X | Measure each Membrane in 9 places Take the average |
| X | X | X | |
| X | X | X | |

Figure 1.4. Template for measuring thickness of membrane, CCM, GDL, and MEA

1.5 Cell Assembly

Figure 1.5 shows an expanded view of a complete PEM fuel cell hardware. The design of the hardware is for 25 cm² cells. For this program, cells are assembled by hand using bipolar plates, load plates, and end plates purchased from FCT. Prior to assembly, GDLs are tested for permeability, and cut to the required size. Teflon[®] sheets are also die cut to size, and the membrane is spray-coated with a mixture of TKK platinum on carbon catalyst to form the CCM. CCMs have 0.4 mg/cm² catalyst on each electrode for the screening tests; however they can be fabricated with catalyst loadings down to 0.05 mg/cm² by the same processing equipment. After all components have been prepared, cleaned and documented, cells are assembled as defined in the “Assembly Procedure” included as Appendix B. Cell “pinch”, which establishes the compressive load on the active area of the MEA is controlled by selecting Teflon[®] sealing gaskets that are thinner than the sum of the component thicknesses in the active area of the cell. This pinch is a critical parameter for cell assembly and is controlled by selecting Teflon[®] gaskets of appropriate thickness as shown in Figure 1.6.

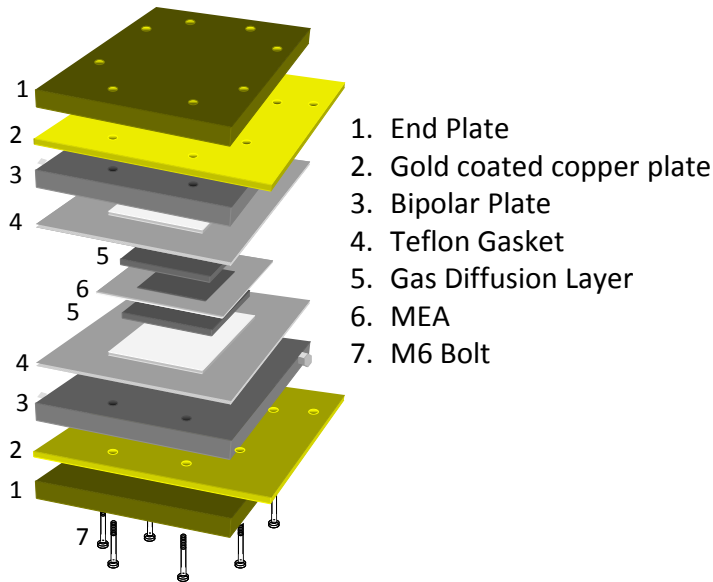


Figure 1.5. Expanded view of PEM fuel cell hardware

GDL Thickness

| | | |
|-----|-----|-----|
| 398 | 402 | 396 |
| 398 | 405 | 400 |
| 400 | 398 | 400 |

Cathode

| | | |
|-----|-----|-----|
| 405 | 398 | 395 |
| 405 | 406 | 400 |
| 402 | 409 | 404 |

Anode

CCM Thickness

| | | |
|----|----|----|
| 56 | 57 | 70 |
| 62 | 62 | 74 |
| 64 | 62 | 70 |

Pinch

| | | |
|-----|-----|-----|
| 239 | 237 | 241 |
| 245 | 254 | 254 |
| 246 | 249 | 254 |

All numbers are in μm .

The gaskets were each $310 \mu\text{m}$

Measure each component in 9 places

Build cell with $229\text{-}254 \mu\text{m}$ pinch

$$\text{Pinch} = T_{\text{GDL (Anode)}} + T_{\text{GDL (Cathode)}} + T_{\text{CCM}} - T_{\text{Gaskets}}$$

Figure 1.6. Schematic showing sample calculation of pinch by measuring thicknesses of each component using the template.

1.6 Pre-Test Integrity Test

After the cell is built and before it is delivered to the test stand, it is externally leak-tested, internally leak-tested, and electrically isolation-tested, each according to the procedures in Appendices C and D. These tests are to ensure that the cell integrity is sufficient to warrant the test effort and to establish a pre-test baseline database. After the cell is mounted in the test stand, the external leak-test of the cell and the stand, and the resistance tests of the cell and

the stand are repeated to validate test readiness. Allowable limits for these tests have been established by a combination of FSEC’s cumulative test experience and an “impact analysis” of the worst-case level of leakage and cell resistance on cell performance. These levels were also verified by the current FSEC database.

1.7 Tests Performed on Each Cell

1.7.1 Linear Sweep Voltammetry, Cyclic Voltammetry (CV), and Cell Conditioning

Prior to performance evaluation, electrochemical active area (ECA) and H₂ crossover (CO) tests are performed on the cell at ambient pressure at room temperature and 100% RH, 80 °C and ~100%RH, 100 °C and 69%RH, and 120 °C and 35% RH. The cathode is the working electrode and the anode, using hydrogen, is the counter as well as the reference electrode. Flows of 0.4 L/min of hydrogen and nitrogen are introduced to the anode and cathode side of the cell, respectively. The CV is conducted at a scan rate of 30 mV/s between zero and 0.8 V to determine the ECA. Hydrogen crossover is measured by the limiting current density method with H₂ flowing on the anode and nitrogen on the cathode. The cell potential is scanned potentiodynamically at 4 mV/s from zero to 0.8 V. A PAR 263A potentiostat and CorrWare software is used to control the potential. The crossover test shows both the level of gas diffusion through the membrane and any electrical short. The level of diffusion is given by the flat portion of the curve and the level of short is calculated from the sloping linear relationship between the applied voltage and the measured current, specifically the measured current between 0.3 V and 0.8 V. Shorts must be less than the equivalent of 10 mA to be allowable, and to clarify the CV data, the linear slopes resulting from these electrical shorts are removed from the data. Figure 1.7 shows sample CO data and Figure 1.8 shows sample ECA data as a function of cell temperature. In the CV data, the hydrogen desorption peaks can easily be observed at room temperature. CV and CO tests were repeated before each test condition to provide data in support of the cell analysis.

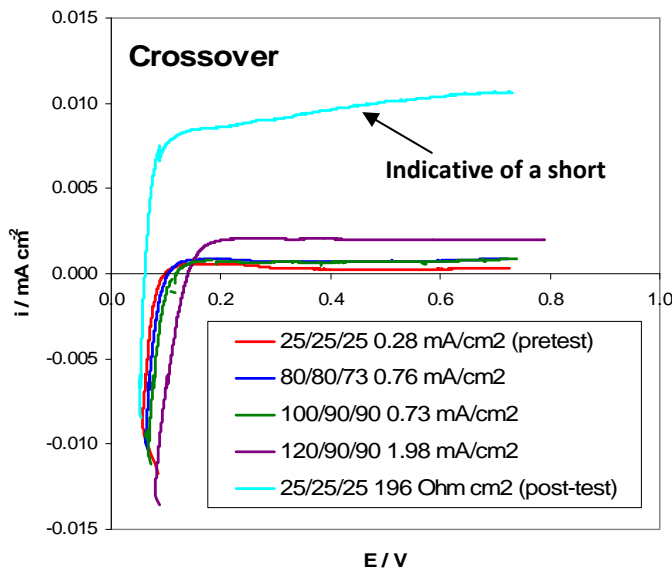


Figure 1.7. CO data at different cell temperatures. Linear sweep voltammogram of FSEC-1; scan rate = 2 mV/s, 0.4 L/min (H₂/N₂), T_{cell} = 25, 80, 100, 120 °C, working electrode = cathode, counter/reference = anode.

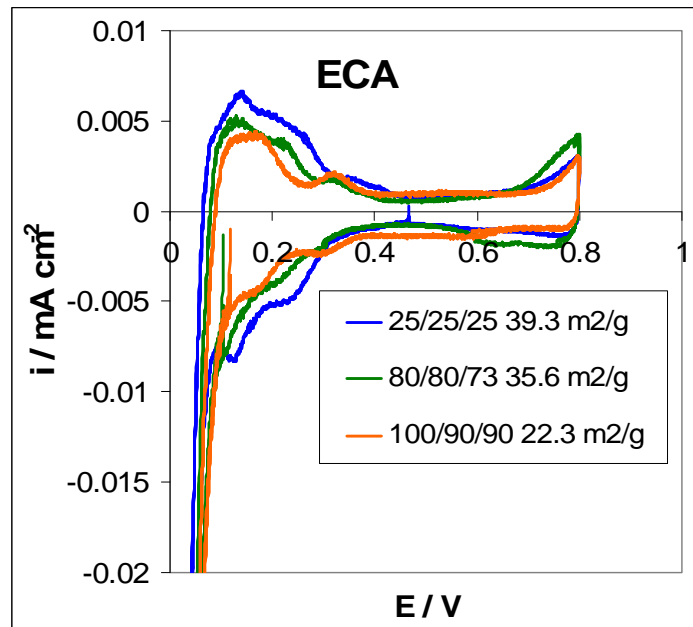


Figure 1.8. ECA data as a function of cell temperature. Cyclic Voltammogram of FSEC1; scan rate = 30 mV/s, 0.4 L/min (H₂/N₂), T_{cell} = 25, 80, 100 °C, working electrode = cathode, counter/reference = anode.

Analysis of the cyclic voltammogram provides a measure of the electrochemical surface area (ECA) of the cathode. Periodically repeating this test during the program provides a history of the changes in cathode ECA for sample MEAs as a function of a fixed test history. Analysis of these test results shows comparative merits of the submitted membranes.

It is recognized that the initial testing of CO/CV prior to conditioning the CCM does not represent the full potential of the cell, but these tests provide important data about the initial condition of the cell. In addition, this data is required as input for the analysis of the cell during operation. After the CO/CV test, the cell is heated to 80 °C and the saturators are heated to 80 °C for the anode and 73 °C for the cathode, and the cell is conditioned for operation by adding water to the electrolyte. This is a two-step procedure, where water is first added as a vapor from saturated gases (hydrogen on the anode and nitrogen on the cathode), and second by operation of the cell at a fixed voltage of 0.55 V and run on hydrogen/air. Each step is for three hours, which, for Nafion[®], has proven to be sufficient to “wet-up” the electrolyte.

After the electrolyte has been “wet-up”, a series of performance curves are run at ambient pressure and high stoichiometry. These tests are run at near 100% RH with the cell at 80 °C. The air dew point is a few degrees lower than the cell temperature to avoid flooding the cathode.

Performance sweeps are taken on H₂/Air, H₂/O₂, and H₂/Air out to I_{Lim} or I_{Max}. The limiting current, I_{Lim}, is the current when the cell voltage becomes 0.3 V. This value is used rather than 0V to avoid damage to the cell. The maximum current, I_{Max}, is based on the test stand reactant

flow capability at 2000 mA/cm^2 . The repeat of the H_2/Air sweeps allows performance between the two tests to be compared to show that the cell has reached a stable starting performance point, and to indicate that the differences between the air and the oxygen performance is being measured accurately. Test points are held for five minutes during this testing as a compromise between fully stable data and a reasonable amount of total test time.

The cell is then held at these same conditions (80°C , 80°C , 73°C) while the initial test data is reviewed and the cell integrity is judged to be sufficient to proceed with the remainder of the testing. If the cell performance is not adequate, it is cooled to room temperature, the CO/CV is repeated and further data analysis is conducted to determine the next steps.

1.7.2 Performance Verification Test

The purpose of the Performance Verification Test is to provide data on cells operating at a series of conditions that are at the DOE goals or that represent significant progress toward those goals and show the relative merits of each of the candidate membranes at those conditions. Having conditioned the cell and verified its integrity, the Performance Verification Test is started without shutting down. The cell is pressurized to 1.5 atmospheres while holding the cell temperature and the anode and cathode saturator temperatures at 80°C , 80°C , 73°C respectively; and a series of H_2/Air , H_2/O_2 , H_2/Air performance sweeps are taken at high stoichiometry (above 3 to minimize the effects of reactant utilization). Following these tests, the cell and saturators are heated to 100°C , 90°C , 90°C , which changes the inlet RH at the cell to 69%. The three performance sweeps are taken and the temperatures are increased again to 120°C , 90°C , 90°C to further reduce the relative humidity to 35%. In all cases, the cell is returned to 100°C , 90°C , 90°C and held at 400 mA/cm^2 overnight to evaluate the stability between the different sets of lower relative humidity tests. Cell temperatures are held at 100°C , 90°C , 90°C in preparation for the stability test.

1.7.2.1 Polarization Curve

Figure 1.9 shows typical performance data, which shows three characteristic regions. The first (low current density region) is dominated by activation overpotential losses, the second (mid current density) is dominated by resistance losses, and the third (high current density region) is dominated by mass transport losses. Analysis of these curves, and the changes to the various regions of the curves, as the testing progresses, provides key insights into the performance and stability of the membrane as well as the catalyst layer and the catalyst-membrane interface.

Fuel Utilization and air Utilization have significant impacts on the shape of these curves. Although utilizations used are higher than the goal values, this data is valuable in comparing performance with lower fuel and air flows. The highest obtainable current density is I_{Lim} , which is an important input to the analysis of the internal resistance of the cathode.

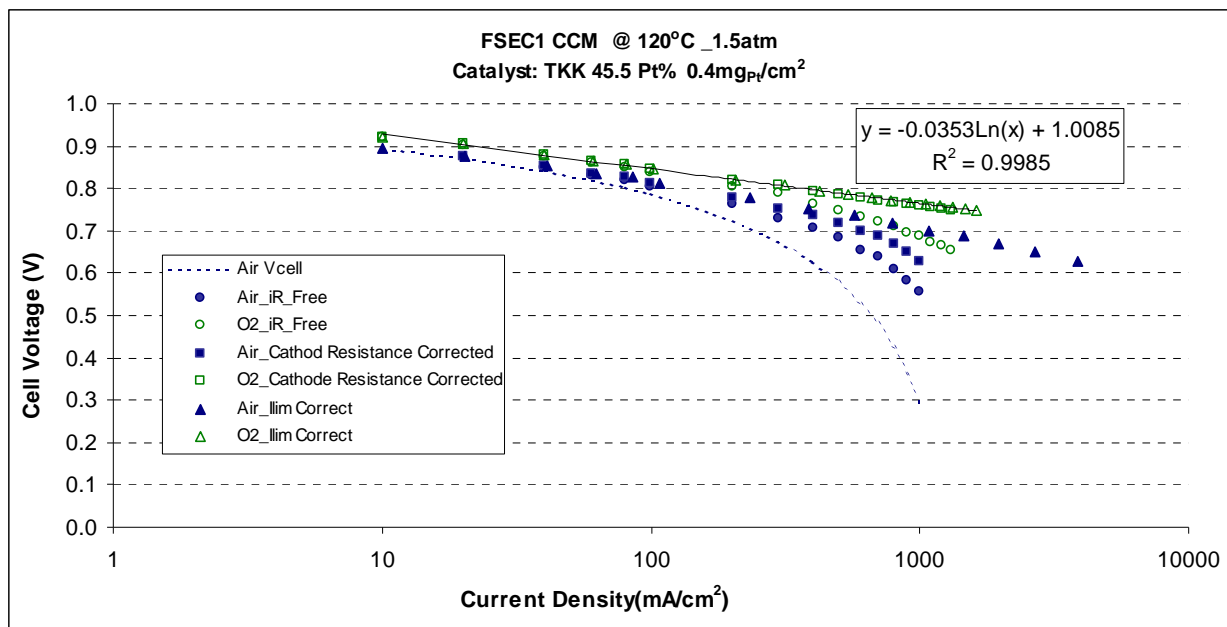


Figure 1.9. Typical performance for a load calibration sweep

1.7.2.2 Stability Test

A 64-hour endurance test is run on the 25-cm² fuel cell under H₂/Air. The operating conditions are 400 mA/cm², 100 °C, 1.5 atm, and 69% RH for both the H₂ and air reactants. The cell is maintained at a constant current while cell performance and resistance are measured. Initial and final electrochemical active area, fuel crossover, and performance are determined at cell operating temperature. Water from the exit streams is condensed, collected, and analyzed for fluoride ion concentration.

Typical cell voltage and cell resistance stability curves are shown in Figure 1.10. The voltage and resistance are both expected to be stable throughout the stability test. Any change in performance or resistance is a result of MEA degradation due to chemical and mechanical stress.

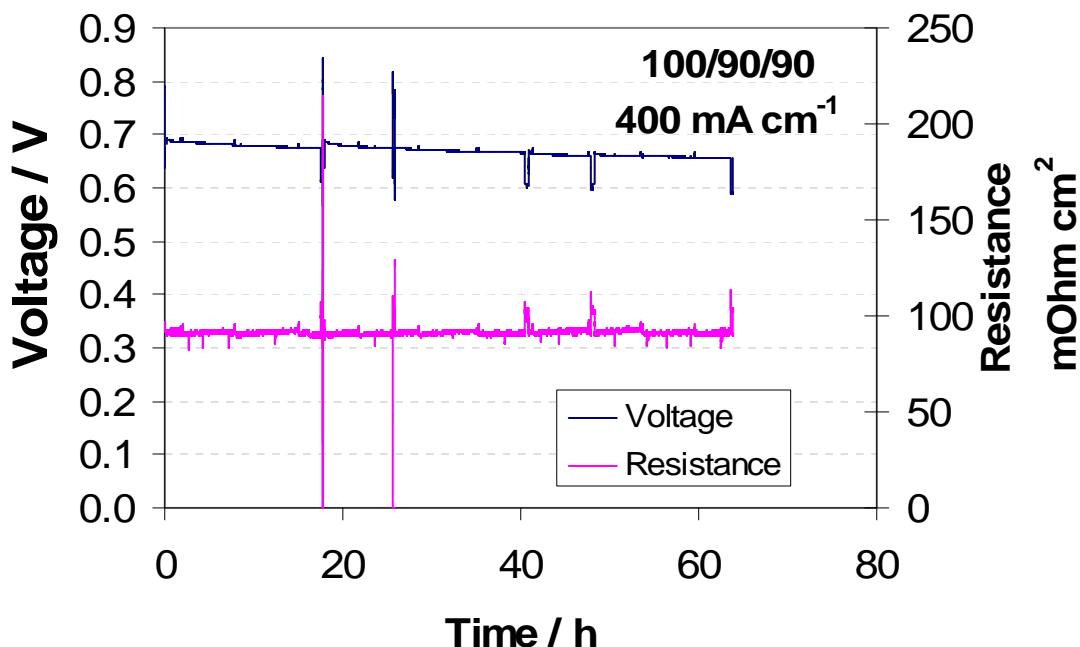


Figure 1.10. Typical performance and cell resistance stability curve. Voltage and resistance of FSEC-3, 0.2 L/min (H₂/Air), T_{cell} = 90 °C, %RH = 30, i = 400 mA/cm².

1.7.3 Post-Test Integrity Test

After the cell is tested, and before it is removed from the test stand, the external leak-test of the cell and the stand, and the resistance tests of the cell and the stand are repeated to quantify the end-of-test condition. In addition, before the cell is disassembled, it is externally leak-tested, internally leak-tested, and electrical isolation tested on the bench, each according to the procedures in Appendices C and D. Bolt torque is also measured. These tests are to determine the changes in the cell integrity as a result of the protocol testing. These tests provide data on some of the changes to the membrane as well as data external to the MEA that is needed to support the cell analysis.

1.7.4 Post-test Analysis

FSEC has developed and verified a process to evaluate sources of polarization, mainly associated with the cathode, in hydrogen/air proton exchange membrane fuel cells and is currently using this process to analyze cell performance and guide development efforts. This process quantifies the six sources of polarization using data from the standardized test program. Non-electrode ohmic overpotential, electrode ohmic overpotential, non-electrode concentration overpotential, electrode concentration overpotential, activation overpotential from the Tafel slope, and activation overpotential from catalyst activity are analytically separated into their distinct elements. The analysis is based on hydrogen/air polarization curves of an in-house membrane electrode assembly (MEA) using hydrogen/oxygen polarization curves as a diagnostic tool. The analysis results compare three cell temperature/relative humidity /oxygen partial pressure (p_{O₂}, atm) conditions: 80°C/100% RH_{anode}/75% RH_{cathode}, 100°C/69% RH, and 120°C/35% RH, which represent near fully-humidified, moderately humidified, and low humidified conditions, respectively, at 1.5 atm operating pressure. The technique is useful for

diagnosing the main sources of loss in MEA development work, especially for high temperature/low relative humidity operation where several sources of loss are present simultaneously. The following verification test data and analysis is reported.

1.7.5 Cell Data Report

The experimental testing of a new membrane will result in the following data to enable the evaluation of membrane performance in a cell:

- Cell build verification test data
 - Electronic resistance for short determination
 - External leakage rate
 - Internal leakage rate
- Cell characterization at start-up
 - Cyclic voltammetry at 25 °C to enable electrochemical area determination
 - Linear sweep voltammetry at 25 °C for crossover and electronic resistance determination
- Performance test data
 - Cell voltage and cell resistance (OCV to 0.3V) at the following conditions:
 - H₂/O₂ and H₂/Air reactants at 1.5 atm, 80 °C/80 °C/73 °C (100% RH)
 - H₂/O₂ and H₂/Air reactants at 1.5 atm, 100 °C/90 °C/90 °C (69% RH)
 - H₂/O₂ and H₂/Air reactants at 1.5 atm, 120 °C/90 °C/90 °C (35% RH)
 - A tabulation of cell current density and resistance using H₂/Air reactants at 0.7 V for the above three conditions (This tabulation is provided to enable rapid membrane comparisons.)
- Stability test data
 - Cell voltage and resistance for 100 hours at the following conditions:
 - H₂/Air reactants at 400 mA/cm², 150 kPa, 100 °C, 69% RH
 - Cyclic voltammetry to enable post-test electrochemical area determination
 - Linear sweep voltammetry for post-test crossover and electronic resistance determination
 - Fluoride present in exhaust condensate

1.8 Fuel Cell Hydrogen Safety Plan

Florida Solar Energy Center (FSEC) complies with the University of Central Florida (UCF) Chemical Hygiene Plan (CHP), as required by OSHA. This plan and its associated documentation (“Safety Standards for Hydrogen and Hydrogen Systems - SSHHS”) provides a written description of safety policies and procedures that all university laboratory personnel must follow. All faculty researchers, student trainees and visiting scientists and engineers working at FSEC's hydrogen research laboratories are provided with training and a copy of SSHHS document. SSHHS document contains guidelines for hydrogen system design, material selection, operation, storage, handling and transportation. Furthermore, FSEC's hydrogen laboratories and field facility meet and/or exceed the design and safety requirements imposed by the Florida State Fire Marshall and all the state and federal codes (NFPA 45 Standard on Fire Protection for Laboratories Using Chemicals, NFPA 50A Standard for Gaseous Hydrogen

Systems at Consumer Sites, and NFPA 70 National Electric Code) for handling large volumes of hazardous and flammable gases and chemicals including both gaseous and liquid hydrogen. In addition, FSEC's hydrogen field facility has been upgraded with explosion proof electrical systems and meets NFPA 50B Code "Liquefied Hydrogen Systems at Consumer Sites."

Before each new research activity is initiated, the personnel safety at and near the facility is reviewed and emergency procedures implemented at the earliest planning and design stages. Advance planning for a variety of emergencies such as fires and explosions are conducted and proper procedures developed and implemented. All hydrogen systems will be instrumented and checked for:

- i. Process monitoring and control.
- ii. Collection of performance data.
- iii. Providing warnings and/or alarms for out-of-limits conditions.
- iv. Early detection of hazardous condition(s).
- v. Compatibility with hydrogen service.
- vi. Establishment of local and/or remote operation and monitoring of the hydrogen system.
- vii. Having appropriate range, accuracy, and response time.

The Safety Assessment Review shall be updated anytime a system or process is changed. An annual facility inspection shall be conducted and documented. A formal Operating and Support Hazard Analysis shall be performed as directed by the UCF Environmental Health and Safety Office. Significant hazards identified shall be eliminated or reduced to acceptable risk levels.

Mr. Randy Fowler is the FSEC's Hydrogen R&D Laboratories' operational and safety manager. He has attended and completed the following safety related courses and training activities:

- i. A full day course on HazMat and received his certification on spill control and respirators.
- ii. Has successfully completed "NASA Hydrogen Safety" class.
- iii. Attended the "Lab Safety Basics and Beyond" at PITTCON.
- iv. Taken and passed UCF's "Chemical Safety and Environmental Management for Laboratories" class.
- v. Has post-secondary vocational certificate given by the Brevard Community College on "Chemical Laboratory Specialist," December of 2002.

Mr. Fowler oversees the enforcement of all hydrogen and chemical safety procedures and training of the graduate research students working in FSEC's hydrogen laboratories. In addition, a comprehensive safety plan will be developed and submitted to DOE at the time of contract award. In preparation for this plan, the following steps will also be taken:

- The Chemical Hygiene Plan (CHP) will be re-evaluated, modified as needed and periodically checked for compliance.
- A standard operating procedure (SOP) will be established for all experiments and operations taking into account hazard level of materials to be used.

- Procedures will be developed for reporting any events or deviations from SOP.
- Failure risk mitigation plan will be developed based on vulnerabilities identified by Modes and Effects Analysis.

2. Day by Day PEM MEA Test Sequence

2.1 Assemble the Cell

- The Sample MEA
 - From team members two pieces – 12 cm x 12 cm (4.75" x 4.75")
- Anode and Cathode Catalyst
 - TKK 46% Pt catalyst
 - 0.4 mg/cm²
 - 32 wt% Nafion[®] 1100 binder
 - Spray
 - 136°C heat treat
 - Protonation
 - Two identical: One tested/One returned untested
- GDL
 - SGL 10BB
- Pinch 0.25 mm +/- 0.02 mm (0.01" +/- 0.001")
- Teflon[®] Gasket Lay-up
- FCT Cell
 - Hardware – Single pass serpentine
 - Cross flow cathode horizontal
 - FSEC bar and groove dimensions
- Bolt Load
 - Star pattern
 - 4.5 N m torque (40 inch pounds)
- Pretest Conductivity
 - Tested at BekkTech

2.2 Day 1

- Measure mechanical crossover
 - < 1 mA eq. is acceptable
- Measure External Leakage
 - < 20 mA eq. is acceptable
- Cell Resistance acceptable limits
 - Anode to Ground > 100 Ohms
 - Cathode to Ground > 100 Ohms
 - Anode to Cathode > 30 Ohms
- Mount in test stand
 - Resistance Anode/Cathode to ground > 100 Ohms acceptable
- Room temperature CO and CV
- Wet-Up
 - 80/80/73
 - H₂/N₂ 3h – No Load
 - H₂/Air ~ 3h – 0.55V

- Cool-down to room temperature
 - Hold on N₂/N₂

2.3 Day 2

- Room temperature CO and CV
- Heat up
 - H₂/N₂
 - 80/80/73 °C
 - Ambient pressure
- Measure
 - Open circuit voltage (OCV),
 - Performance (volts vs. current) curve (VI)
 - Cell resistance (by current interrupt)
- Stoichiometry
 - Anode 3, Cathode 3.6
- H₂/Air
- H₂/O₂
- H₂/Air
- CO and CV at 80/80/73 °C
- Hold overnight at 400 mA/cm²

2.4 Day 3

- Pressurize to 1.5 atm
- Measure at 80/80/73 °C
 - OCV, VI curve, cell resistance
- Stoichiometry
 - Anode 3, Cathode 3.6
- H₂/Air
- H₂/O₂
- H₂/Air
- Heat to 100/90/90 °C
- CO and CV at 100/90/90 °C
- Measure
 - OCV, VI curve, cell resistance
- Stoichiometry
 - Anode 3, Cathode 3.6
- H₂/Air
- Hold overnight at 400 mA/cm²

2.5 Day 4 (at 1.5 atm)

- Run VI curve at 100/90/90 °C
 - Stoichiometry
 - Anode 3, Cathode 3.6
 - H₂/Air

- H₂/O₂
- H₂/Air
- Heat to 120/90/90 °C
- Record CO and CV at 120/90/90 °C
- Measure
 - OCV, VI curve, cell resistance
- Stoichiometry
 - Anode 3, Cathode 3.6
- H₂/Air
- H₂/O₂
- H₂/Air
- Cool to 100/90/90 °C
- Hold overnight at 400 mA/cm²

2.6 Day 5, 6, 7 (at 1.5 atm) – Stability Test

- Record CO and CV at 100/90/90 °C
- Measure
 - OCV, VI curve, cell resistance
- Stoichiometry
 - Anode 3, Cathode 3.6
- H₂/Air
- H₂/O₂
- H₂/Air
- Start stability test
 - 1.5 atm
 - 100/90/90 °C
 - H₂/Air
 - 400 mA/cm²
- Stoichiometry
 - Anode 3, Cathode 3.6
- Measure during stability test
 - Cell voltage
 - Fluoride emission rate (FER) in reactant exhausts

2.7 Day 8 (at 1.5 atm)

- Record CO and CV at 100/90/90 °C
- Measure
 - OCV, VI curve, cell resistance
- Stoichiometry
 - Anode 3, Cathode 3.6
- H₂/Air
- H₂/O₂
- H₂/Air

- Cool down to room temperature
- H₂/N₂

2.8 Day 9

- Room temperature CO and CV
- Resistance test
 - Anode to ground
 - Cathode to ground
- Remove the cell
- Measure mechanical crossover
 - < 1 mA eq. desired
- Measure external leakage
 - < 20 mA eq. desired
- Measure cell resistance
 - Anode to ground – Record Ohms
 - Cathode to ground – Record Ohms
 - Anode to cathode – Record Ohms
- Post-test
 - Measure bolt load
 - Disassemble cell
 - Conduct visual inspection
 - Bag and Seal Components
- Data analysis
 - Delta cell resistance
 - Delta CO
 - Delta CV
 - Delta activation loss
 - Delta Tafel slope
 - Delta diffusion
 - Delta I_{lim}
 - Delta conductivity
 - FER

Appendix A: Gurley Number Test Procedures

Document Number WP0012WPQRS

A1. Reference materials

A1.1 Min-Kyu Song, "PEMFC Unit Cell Assembly for NT Membrane and E-TEK Electrode", March 13, 2001, Uconn

A1.2 B.Mueller, E.De Castro etc., "CARBON CLOTH GAS DIFFUSION BACKINGS FOR HIGH PERFORMANCE PEFC CATHODES", Electrochemical Society Proceedings Volume 98-27

A2. Introduction

For porous and/or fibrous gas filtration media, resistance to flow is often used as a characteristic measure for quality control and performance. Gurley Number basically measures gas permeation rate through the gas diffusion backing samples.

The Gurley number represents the gas flow rate (in LPM) for a Gas Diffusion Layer sample at a fixed pressure difference (in cm of H₂O) through a fixed area of sample (cm²), and thus indicates the resistance to gas flow.

Samples of gas diffusion layers were cut and fitted into a manifold of main gaskets and sub-gaskets, such that the uncoated side of the backing is oriented towards the nitrogen inlet and that there is no pinch on the Gas Diffusion Layer. The total gasket thickness has considerable influence on the pressure distribution pattern over the active area on both inlet and outlet sides. Therefore, it is very important to employ enough thickness gaskets to get relatively uniform pressure distribution, resulting in more meaningful Gurley numbers.

Prior to evaluation of a backing sample, the system inherent resistance to gas flow is evaluated by measuring pressure drop across the cell over a range of flow rates. This system resistance to flow is used as a correction in the subsequent measurement of backing gas flow characteristics. The configuration of the system is illustrated as in Figure A1.

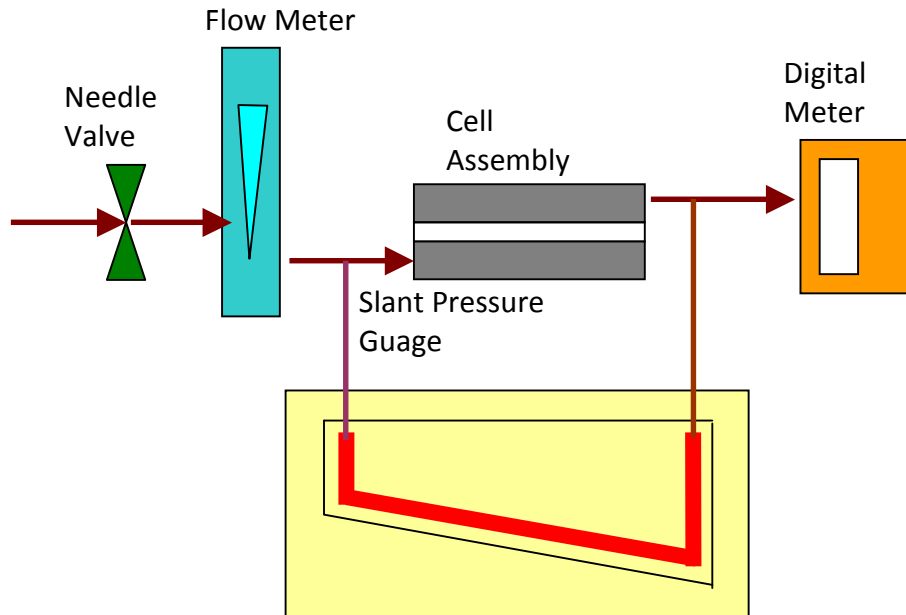


Figure A1. Apparatus for Measuring Gurley Number

A3. Materials and Equipment

A3.1 Materials

- Teflon film (0.25 mm (10 mil) thickness)
- Krytox (Dupont performance lubricants, Teflon® Grease)
- Gas Diffusion Layers
- Snoop Liquid Leak Detector
- Ethyl Alcohol 200 Proof, Absolute (Dehydrated) USP Grade, Pharmco Products INC
- Deionized Water (From 153 Fuel Cells Laboratory- ERI)

A3.2 Equipment

- Electronic Digital Micrometer (Mitutoyo)
- CARVER Hydraulic Press (Unit Model #3912)
- Clicker Dies
- Torque Wrench (0-5.6 N-m, 0 to 50 in-lbs)
- 3/8 inch Nut Driver
- Nut Plate
- Tweezers
- Knife
- Single Cell Parts
 - Graphite Flow Channel
 - Gold Plated Copper Plates
 - Insulator Sleeves
 - M6 Bolts / M6 Nuts
 - Flat Washers

A3.3. Quality control (test and criteria)

- QS 0012 Gas Permeability, Gas Diffusion Layer.

A4. Main Gasket Preparation

To ensure sealing over the gas diffusion layer edges, the main gasket thickness should be at least $51\ \mu\text{m}$ (2 mil) thinner than the gas diffusion layer thickness. In general, the carbon papers and carbon cloths fall in the range between 356 and $457\ \mu\text{m}$ (14 and 18 mil). In this case, a $254\ \mu\text{m}$ (10 mil) thick Teflon main gasket would be appropriate. However, it is important to keep in mind that we should always use a little thinner main gasket than gas diffusion layers.

Using clicker die and hydraulic press, make a normal size gasket, then make it $6.5\ \text{mm}$ ($1/4''$) larger on each side. Using a $25\ \text{cm}^2$ cell as an example, a normal gasket would be like in Fig A2a. After made larger, the gasket would look like in Fig A2b.

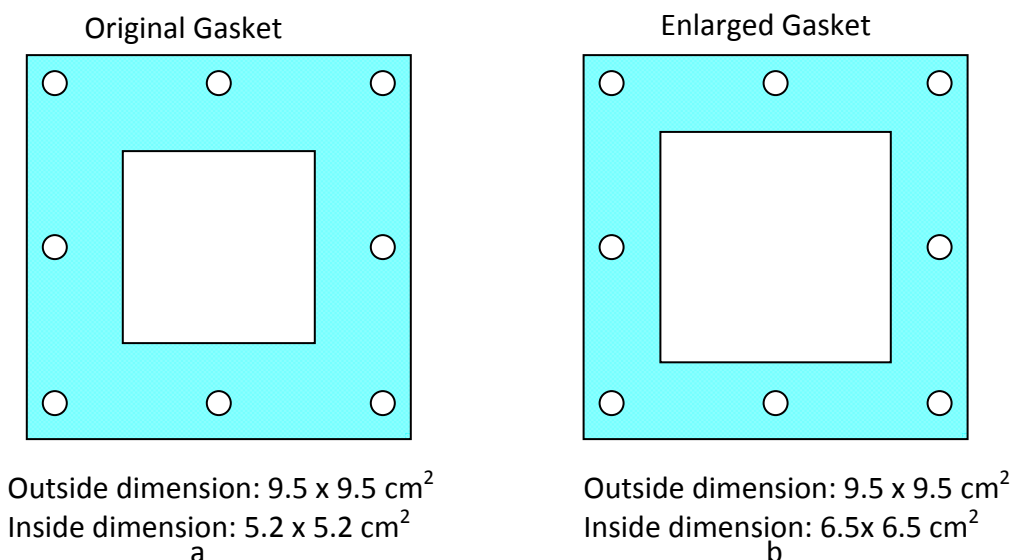


Figure A2. Preparation of Main Gaskets ($5.2 \times 5.2\ \text{cm}^2$ cell size)

A5. Preparation of Sub-gaskets

Sub-gaskets are designed with a $5.8\ \text{cm} \times 5.8\ \text{cm}$ inside dimension and the outside dimension is the same as for normal gaskets. However, it is very important to prepare enough thick gaskets to guarantee that we can have a uniform pressure distribution on the active area, which will lead to a meaningful Gurley Number. The correct total thickness depends on the active area, and on the physical properties of gas diffusion layers.

It is also important to alternate thin and thick gaskets in the final assembly to ensure proper sealing by the gaskets.

From experience, for 5 cm², 6.25 cm², and 25 cm² cells, 3810 μm (150 mil) of total gasket thickness is enough to generate a uniform pressure distribution. Suppose the thickness of the main gasket is 254 μm (10 mil). We could use seven 254 μm (10 mil) gaskets, then one 254 μm (10 mil) main gasket. The total gasket thickness would be 1778 + 1778 + 254 = 3810 μm (70 + 70 + 10 = 150 mil).

A6. Preparation of Gas Diffusion Layer samples

Gas Diffusion Layers should be strictly cut according to the size of the main gasket (Figure A2b, 6.5 x 6.5 cm). To ensure proper sealing on the edges of the gas diffusion layer, it is important to make sure that the sample and the main gasket matches nicely.

A7. Cleaning of Fuel Cell Components

- i. Rinse the gold plated copper plate, graphite flow channel block and Teflon gaskets with deionized water by soft brushing.
- ii. Wash them with ethanol.
- iii. Dry them at room temperature.

A8. Assembly Procedure

Note. All parts should be oriented as shown in Figure A3.

- i. Lubricate the surface of thread of M6 Bolts using Krytox Teflon grease.

Note. Verify nuts turn freely on M6 bolts before placing bolt in the nut plate.

- ii. Place M6 bolts head down in the Nut Plate.
- iii. Put flat washers on through the Nuts.
- iv. Place Insulation Sleeves on through the Nuts.
- v. Place Gold Plated Copper Alloy Plate on.
- vi. Put Bipolar Plate on.
- vii. Put 1778 μm (70 mil) Teflon[®] sub-gasket on (7 layers, 254 μm or 10 mil for each).

Note. Align notches in each gasket.

- viii. Put the main gasket (254 μm or 10 mil thick) on and align subgaskets and main gasket.
- ix. Put the gas diffusion layer on the main gasket. Make sure they fit nicely and the coated side faces up.
- x. Put another 254 μm (70 mil) Teflon[®] sub-gasket on top (7 layers, 254 μm or 10 mil for each).

Note. Align notches with first seven gasket layers (step 7).

- xi. Put the other bipolar plate on.
- xii. Put the other gold plated copper-alloy plate on.
- xiii. Put insulation sleeves in.
- xiv. Put flat washers on.
- xv. Place Nuts on. Tighten nuts using a Torque Wrench and Nut Driver in the sequence 1 to 8 as shown in Figure A4. Apply 2.8 N-m (25 in-lb) torque for each bolt.

Note. Nuts must turn freely by hand before applying torque.

Note. Be sure to put the cell onto the nut plate to tighten nuts. After that, it can be hard to take the cell off. In this case, put a screwdriver between the cell and the nuts plate and gently move the screwdriver upward (on both sides), the cell will be easily taken out of the nut plate. Also, use nut plate to loosen the nuts.

xvi. Connect gas tubes as in Figure A3.

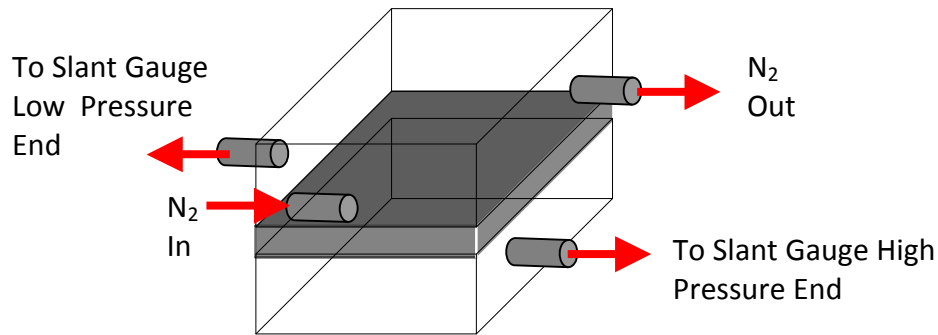


Figure A3. Cell Connections in Gurley Test

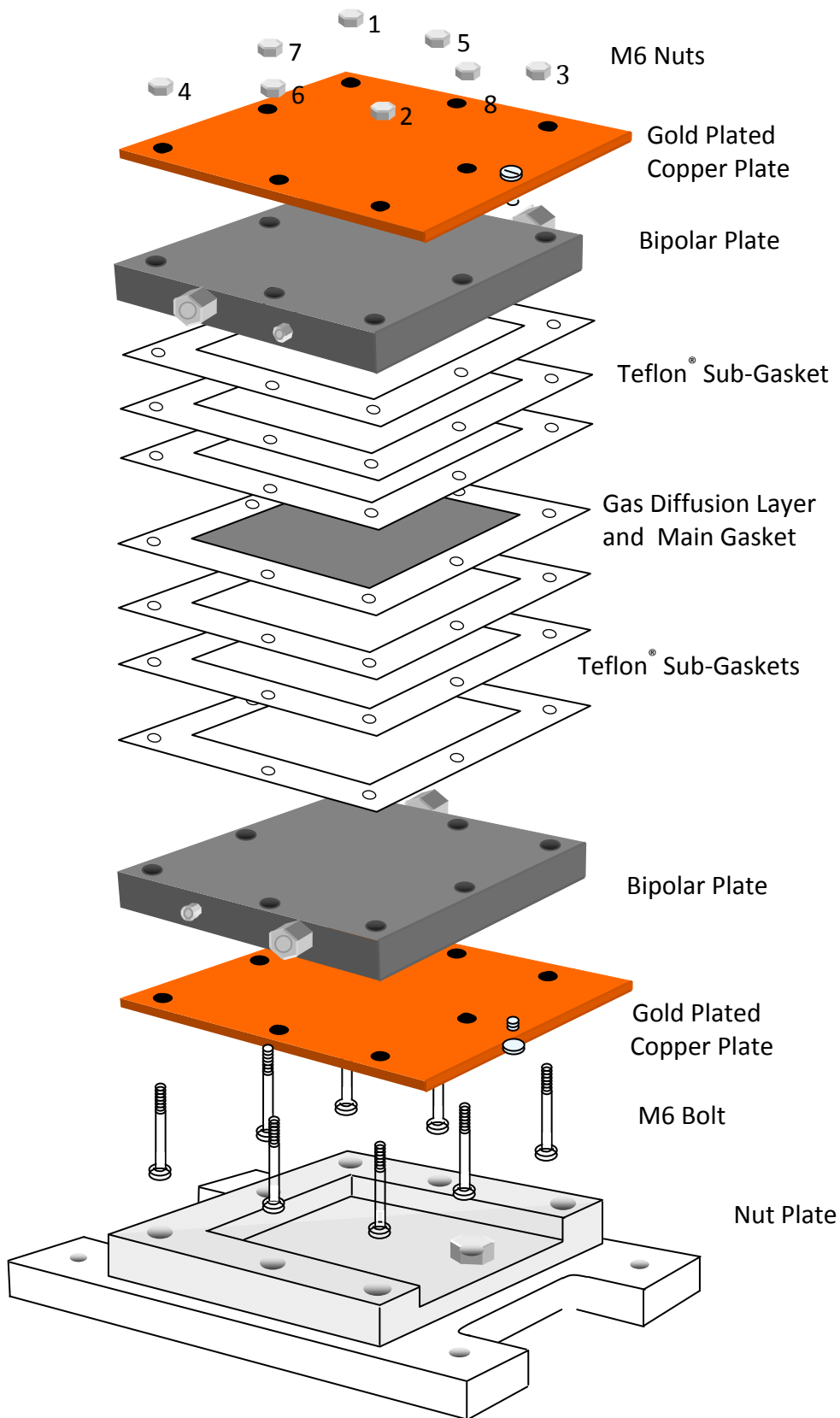


Figure A4. Cell Assembly Explosion View in Gurley Test

A9. Leaking Check Procedure

Every connection shown in Figure A3 should be leak tested before taking measurements. For the cell itself, the gasket side should be tested to make sure there is no leak in between the gaskets. The leak test procedure is as follows:

- i. Verify that the pressure drop meter is in horizontal position (that should be checked using a level), and the slant liquid level is at 0 position with no pressure applied.

Note. Verify needle valve is closed.

- ii. Open N₂ gas, keep pressure ~0.68 atm (10 psi).
- iii. Adjust pressure drop at 0.05 inch H₂O. Open valve slowly, make sure that pressure drop can be held at 0.05 inch slant liquid exactly.
- iv. Hold Snoop tube end at connection and squeeze upright bottle to apply solid stream of Snoop liquid leak detector. Bubbles form if connection leaks. Bubble size indicates approximate leak size.

Note. For different size gaskets and different gas diffusion layers, pressure drop across the cell may vary tremendously when measuring Gurley Number. Since pressures drop and flow rate have a nearly linear relationship, in general, pressure drop can be used as a control factor to take flow rate measurements.

- v. Fill reservoir with Snoop Leak detector Solution if it is not enough.
- vi. Turn On Digital Flowmeter, press ball gently, and make sure that one single bubble is generated at one time.
- vii. Repeat until the flow rate is relatively constant (± 0.03 L / min.), and the surface of flowmeter is wet enough. Record the flow rate result on the form (below).
- viii. Adjust pressure drop at 0.1, 0.15 and 0.2 inch H₂O, repeat steps (1-2).

A10. Data process

- i. Calculate Gurley Number with the average of flow rate with equation 1.

$$GurleyNumber = \frac{FlowRate(LPM)}{PressureDrop * 2.54 * ActiveArea} \quad (1)$$

Active Area is the area of subgasket opening area, for 25cm² cell is 5.8×5.8 cm².

- ii. Calculate Average Gurley Number (GN), if the Percent Relative Standard Deviation (% RSD) of Gurley Number > 10%, at all pressure drop, need REDO it.

$$RSD = \frac{\sqrt{\frac{\sum (x - x_i)^2}{n-1}}}{x} \cdot 100\% , \quad (2)$$

where x- GN average, x_i- measured GN at adjust pressure drop at 0.05, 0.1, 0.15 and 0.2 inch H₂O.

A10.1 Data sheet Gurley number calculations:

Sample number: GDL-

Date:

| | Pressure | | Drop | | |
|---------------------------|----------|-----------------------------|------|-----|-------------------|
| LPM | 0.05 | 0.1 | 0.15 | 0.2 | |
| LPM-1 | | | | | |
| LPM-2 | | | | | |
| LPM-3 | | | | | |
| LPM-4 | | | | | |
| LPM-5 | | | | | |
| Avr. LPM | | | | | GN Average |
| Gurley Number (GN) | | | | | |
| RSD | | % | | | |
| RSD Accept | | <10 % | | | |
| RSD Reject | | >10 % | | | |
| GDL Accept | | 0.10÷0.18 | | | |
| GDL Reject | | <0.10 or >0.18 | | | |

A11. Acceptance

Inspect in accordance with QS 0012 Gas Permeability, Gas Diffusion Layer.

Appendix B: PEM Fuel Cell Unit Cell Assembly Procedure - 25 cm² - Document Number WPxyz

B1 Reference materials

J. C. Lin, "Preparation and Characterization of Composite Proton Exchange Membranes for Fuel Cell Application", Ph.D. Thesis, UConn (2000).

B2 Bill of Materials

B2.1 Materials

- i. A catalyst coated membrane (CCM) consisting of
 - A piece of membrane (either FSEC1, FSEC3, NRE[®] 211, NRE[®] 212, Nafion[®] 112, etc.)
 - A cathode catalyst layer on one side ($\sim 0.4 \text{ mg/cm}^2$), active area of 25 cm^2 .
 - An anode catalyst sprayed onto the other side ($\sim 0.4 \text{ mg/cm}^2$), active area of 25 cm^2 .
- ii. One piece of gas diffusion layer, $5.8 \times 5.8 \text{ cm}^2$, with Gurley number of at least $0.024 \text{ L/min/cm-H}_2\text{O/cm}^2$ for CATHODE GDL
- iii. One piece of gas diffusion layer, $5.8 \times 5.8 \text{ cm}^2$, with Gurley number of at least $0.01 \text{ L/min/cm-H}_2\text{O/cm}^2$ for ANODE GDL
- iv. Teflon[®] film ($25.4 \mu\text{m}$ (1 mil) thickness)
- v. Krytox (Dupont performance lubricants, Teflon Grease)
- vi. Deionized Water and ethanol for washing

B2.2 Equipment

- i. A single-cell hardware set
A "Fuel Cell Technology" (FCT) hardware set consists of:
 - Two uniquely machined graphite flow fields (with FSEC low delta P serpentine grooves)
 - Two aluminum end plates – 1.27 cm ($1/2$ ") thickness
 - Two gold-plated current collectors – 0.32 cm ($1/8$ ") thickness
 - Four plastic rubber tubes – 0.95 cm ($3/8$ ") long, 0.2 mm in diameter (called "line-up pins")
 - Four black rubber o-rings (to seal all the line-up pins)
 - Eight Stainless screws
 - Eight black rubber insulating socks (one for each screw)
- ii. Digital Balance (METTLER AE240, S/N G56273)
- iii. Electronic Digital Micrometer (Mitutoyo)
- iv. Torque Wrench (0 to 5.6 N-m or 0 to 50 in-lbs)

B3 Objectives:

To describe how to assemble a PEM fuel cell unit cell (called Membrane Electrode Assembly, MEA) using a Nafion[®]-based membrane and SGL 10 BB GDL's.

B4 General Instructions

- Lubricate the bolts every two cells to make certain the right amount of torque is applied to the cell (not the friction force)
- Alignment of all components vertically is VERY CRITICAL. Take care to ensure good alignment (even if it means STARTING OVER, if you feel unsure). Misalignment will cause the cell to fail and waste all the effort of the CCM preparation.

B5 Preparation for Cell Assembly

- i. Check that the hardware set contains all the parts. Separate the “cathode” parts and “anode” parts as labeled.
- ii. Clean the surface of the two graphite flow fields with ethanol. Take care not to leave excess ethanol in flow fields prior to assembly.
- iii. Measure the thickness of the CCM by placing it between two 1-mil Teflon[®] films (for surface protection) and measure the overall thickness with a Mitutoyo caliper. Then subtract off the Teflon[®] film thickness. Measure AT LEAST 9 VALUES and obtain an average value.
- iv. Repeat Step iii for cathode GDL and anode GDL.
- v. Set the desired amount of “pinch” – in this case, 229-254 μm (9-10 mils)
- vi. Calculate the total thickness of needed Teflon gaskets by:
Total thickness of the gaskets = $(T_{\text{ccm}} + T_{\text{GDL,C}} + T_{\text{GDL,A}}) - \text{Pinch}$
Total thickness of each side (Cathode and Anode) = Total thickness of the gaskets/2

Note 1. If the total thickness of the gaskets is an ODD number, make total cathode gasket thickness LESS than total anode gasket thickness.

Note 2. Usually $(T_{\text{ccm}} + T_{\text{GDL,C}} + T_{\text{GDL,A}})$ is in the order of 762-889 μm (30 – 35 mils)

- vii. Prepare different combinations of Teflon gaskets (available as 25, 51, 76, 127, and 254 μm or 1, 2, 3, 5 and 10-mil) for the cathode and anode sides.

Note. Cut each gasket using an appropriately designed “die” specific to each hardware design.

- viii. Cut both GDLs to the size that will fit PERFECTLY (within 0.3 mm) inside the middle opening of the prepared gaskets. The opening is slightly bigger (1-2 mm) than the cell active area).

B6 Assembling the Cell – Fuel Cell Technology (FCT) hardware set

Refer to Figure B1.

- i. Place four bolts through the cathode end plate for alignment of cell parts.
- ii. Place the cathode end plate with bolts at the bottom on a sturdy surface.
- iii. Place the cathode gold-plated current collector plate on top of the cathode end plate.
- iv. Place the two line-up tubes and two o-rings into line-up holes of the cathode end plate to align the end plate with the cathode gold-plated current collector.

- v. Place the cathode graphite flow field on top of the cathode gold-plated current collector (with the grooved side up).
- vi. Place the set of cathode gaskets on top. Make SURE it aligns well with all the bolt holes. Use a 1 cm long piece of scotch tape to attach the gaskets to the flow field plate to ensure alignment in the cell, IF NECESSARY.
- vii. Place the cathode GDL in the middle of the set of gaskets (micro-porous layer side UP).
- viii. Place the CCM on top (ANODE side UP); align the catalyst active area in the middle of the gasket middle opening.
- ix. Place the set of anode gaskets on top. Make SURE it aligns well with all the bolt/nut holes.
- x. Place the anode GDL on top, align it with the anode GDL (micro-porous layer side DOWN).
- xi. Place the anode flow field plate on top (groove side DOWN).
- xii. Place the anode gold-plated current collector plate on top.
- xiii. Place the anode end plate on top (with the two plastic line-up tubes and o-rings in place).
- xiv. Tighten all the bolts in a cross-wise or star pattern. In six cycles:
 - A. Tighten loosely by hand (avoid over tightening and applying too much torque)
 - B. Tighten with a torque-wrench, set to 1.1 N-m (10 in-lbs), by feeling the click
 - C. Tighten with a torque-wrench, set to 2.3 N-m (20 in-lbs), by feeling the click
 - D. Tighten with a torque-wrench, set to 3.4 N-m (30 in-lbs), by feeling the click
 - E. Tighten with a torque-wrench, set to 4.5 N-m (40 in-lbs), by feeling the click
 - F. Recheck all bolts with the torque-wrench set at 4.5 N-m (40 in-lbs), by feeling the click
- xv. The assembled cell is now ready for a single-cell performance test.

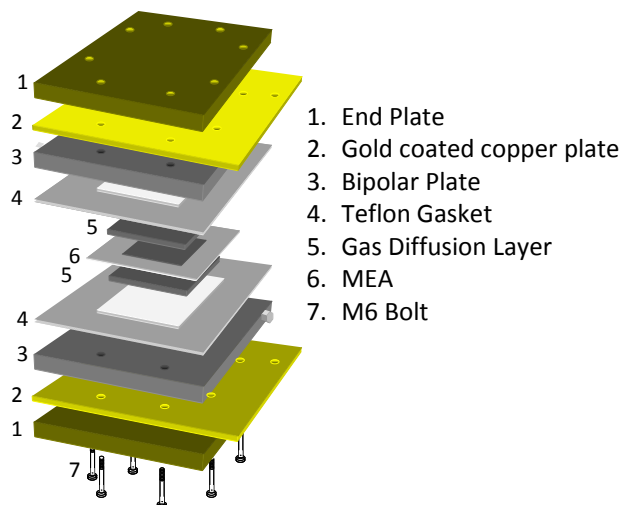


Figure B1. Expanded view of assembled fuel cell

Appendix C: Procedure for Leak Testing a Single Cell after Assembly

Document No. WP0023 and WP0024

C1 External Leak Test

C1.1 Procedure

- i. Setup per Figure C1
- ii. Connect N₂ to hydrogen side with a flow meter followed by an isolation valve.
- iii. Connect gas out line to hydrogen side, with an inlet to the air/O₂ side
- iv. Connect gas out line to air/O₂ side with a 0 to 0.39 atm (0 to 300 mmHg) pressure gauge followed by an isolation valve.
- v. Pressurize the hydrogen and air/oxygen sides to 0.03, 0.07, 0.14, 0.2 atm (0.5, 1, 2, 3 psi) with nitrogen, respectively.
- vi. Record data of the flow meter.
- vii. Leak check with DI water, write down where the bubbles come from. Can use “Snoop” on plumbing connection, not on cell.
- viii. If no leak can be observed and flow meter remains at zero, close the valve, record pressure drop in 5 min.

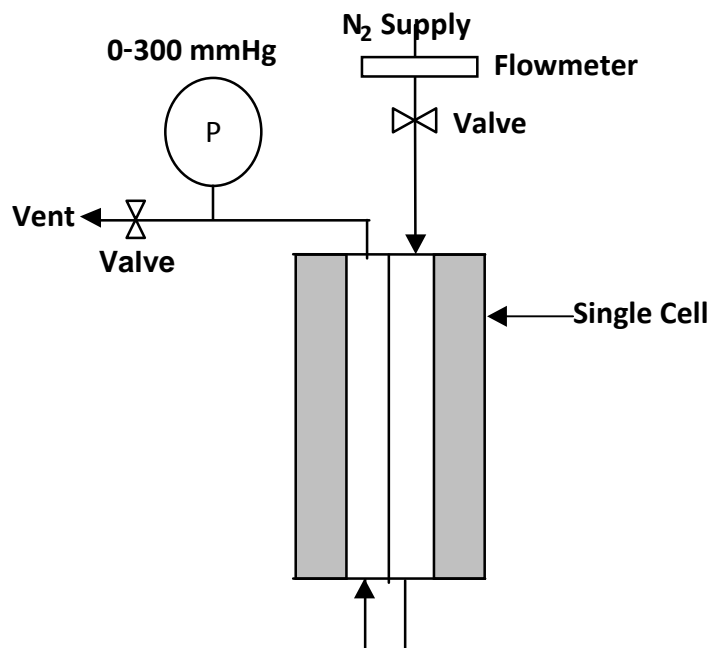


Figure C1. Setup for External Leak Test

C2 Internal Leak Test

C2.1 Procedure for Internal Leak

- i. Verify that external cell leakage satisfies the requirements
- ii. Set up Figure C2
- iii. Connect N₂ inlet to the hydrogen side, with a flow meter followed by an isolation valve
- iv. Connect gas outlet to the hydrogen side with a 0-0.39 atm (0-300 mmHg) pressure gauge followed by an isolation valve
- v. Connect bubble meter to air side
- vi. Close the hydrogen vent and pressurize the hydrogen side to 0.03, 0.07, 0.14, 0.2 atm (0.5, 1, 2, 3 psig) with N₂, respectively
- vii. Check the flow rate at the air/oxygen vent with the bubble flowmeter and flow meter, record data on data sheet
- viii. If no leak can be found and flow meter remains at zero, close the valve and record pressure drop in 5 min.

C2.2 Procedure for Internal Leak

- i. Set up Figure C2
- ii. Connect N₂ inlet to the air/oxygen side, with a flow meter followed by an isolation valve
- iii. Connect gas outlet to air/oxygen side, with a 0-0.39 atm (0-300 mmHg) pressure gauge followed by an isolation valve, other outlet should be closed
- iv. Connect bubble meter to hydrogen side
- v. Close the air/oxygen vent and pressurize the hydrogen side to 0.03, 0.07, 0.14, 0.2 atm (0.5, 1, 2, 3 psig) with N₂, respectively
- vi. Check the flow rate at the hydrogen vent with the bubble flowmeter and flow meter, record data on data sheet
- vii. If no leak can be found and flow meter remains at zero, close the valve and record pressure drop in 5 min.

Single Cell Assembly

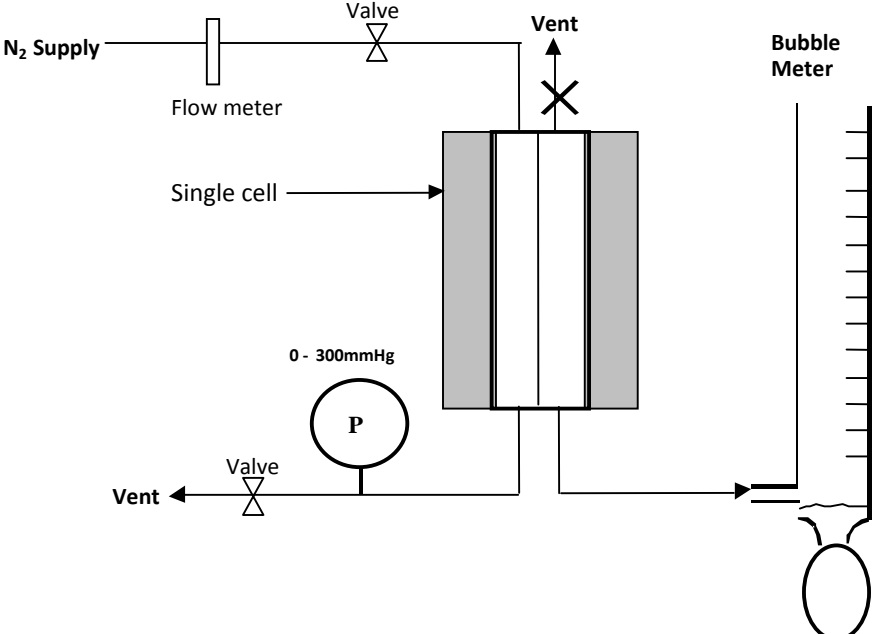


Figure C2: Setup for Internal Leak Test

Appendix D: PEMFC Unit Cell Test

D1. Reference materials

- i. "Fuel Cell Test System Configuration", Scribner Associates, Inc.
- ii. "FuelCell for Windows: Fuel Cell Test Software", Scribner Associate, Inc.
- iii. Operating Manual of Fuel Cell for Windows, Fuel cell Test Software, Scribner Associates model 890 load systems, Version 2.0
- iv. Scribner Associates Inc. 850C Fuel Cell Test System Operating Manual
- v. Configuration and Application Manual of Fuel cell Test System Configuration
- vi. Operating Manual of CorrWare for Windows, Electrochemistry/Corrosion Software, Scribner Associates Inc., Version 2.2
- vii. The Princeton Applied Research Potentiostat/Galvanostat Model 263A Operating Manual

D2. Bill of Materials

D2.1 Materials

D2.1.1 Gases

- | | |
|--------------|---|
| i. Nitrogen | Ultra high purity (99.999%), Airgas N.E. |
| ii. Oxygen | Zero Grade (99.8%), Airgas N.E. |
| iii. Air | Zero Grade (90%), Compressed, Airgas N.E. |
| iv. Hydrogen | Ultra high purity (99.999%), Airgas N.E. |

D2.1.2 Deionized water

Facility DI water

D2.2 Equipment

- i. Fuel Cell Test Station
- ii. Fuel Cell Test Load Box
- iii. Scribner Series 890B or 850C Electronic Load and Data Acquisition System
- iv. Scribner Model 871 Reformate Simulator (optional – not active)
- v. IBM PC with National Instruments GPIB-PCI Interface and GPIB Cable
- vi. Cell Cable Sets
- vii. Potentiostat/ Galvanostat Model 263, Princeton Applied Research
- viii. Insulation jacket (Cloth)
- ix. A plastic jack
- x. Wrench (13mm)
- xi. Multimeter, OMEGA[®], HHM-25 KIT, OMEGA ENGINEERING INC. STAMFORD CT, U.S.A.
- xii. Plastic bottle for deionized water

D3 Objectives

To describe the performance test procedure of a PEM fuel cell.

The overall objective of this test sequence is to determine the performance characteristics of a single, bench-scale, proton exchange membrane fuel cell over a range of temperatures from 80 °C to 120 °C. The cell active area is 25 cm².

D4 General Instructions

Before the cell is mounted in the test stand, a review should be performed of the procedures described below and a check made of the availability of all of the materials and facilities to perform the test program.

D4.1 Fuel Cell System Configuration

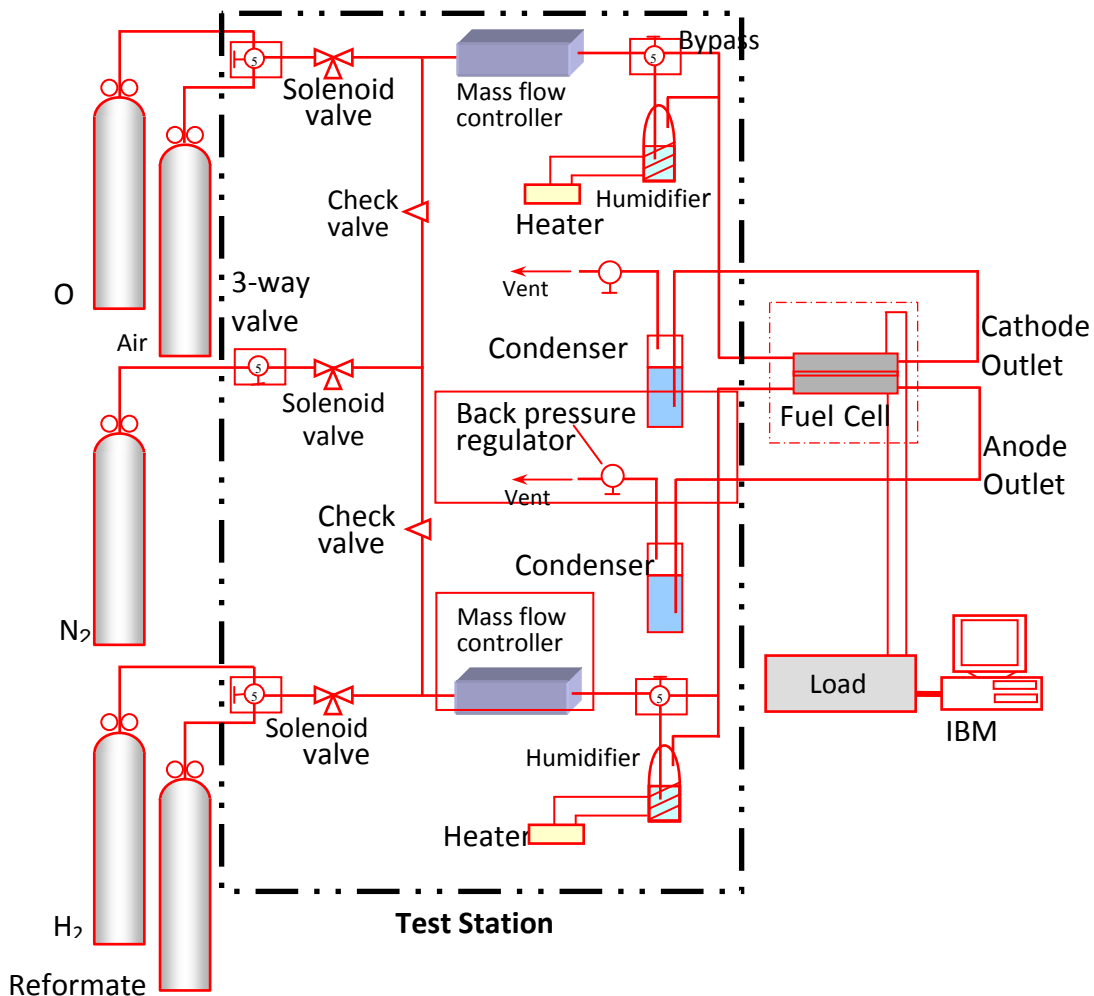


Figure D1. Schematic of Fuel Cell System Configuration

D4.2 Explanation of the test station

- Gases flow through mass flow controller and humidifier to the fuel cell.
 - Gas flow rate is controlled by MKS 1179A mass flow controllers
- Gas temperature and humidity are adjusted by sparging the gases through stainless steel humidifier tanks.

- The humidified gases are introduced to the fuel cell hardware.
- The gases diffuse to the electrodes through gas diffusion layer while they are circulating along the gas channel.
 - The exhausted gases pass through the back-pressure regulator and then to the vent.

D4.3 Hardware Specifications

D4.3.1 Electronic Load System:

| | |
|---------------------|-------------------------------|
| Load Current Range | 10, 25, 100 A, full scale |
| Load Voltage | 20 V max. |
| Load Power Range | 100 W |
| Min Load Resistance | 0.5 to 1 mΩ |
| IR Measurement | Current Interrupt method |
| Current Measurement | Hall Effect Device |
| Load Cooling | Forced air, one or three fans |

D4.3.2 Power Requirements and Physical

| | |
|----------------------|---|
| Main Power | 90 – 125 V AC, 50 – 60 Hz, 4 A |
| Load and Data Unit | 17" W × 10" H × 21" D (43 × 25 × 53 mm) |
| Weight | 35 – 50 lbs (16 – 23 kg) |
| System Interface Box | 8" W × 3" H × 10" D |

D4.4 Data File Name Convention

The data file naming convention is **"BxxxDyyRzzAA"**, where

- "B" stands for "BUILD"
- "xxx" represents the cell number that is assigned by the database upon entry of the cell assembly record information
- "D" stands for "DAY"
- "yy" represents the number of days that the cell has been "tested"
 - (Do not count days elapsed, only the days that the cell has been operated: the first day that you operate the cell YY=1, if you operate the cell the next day, or any number of days later, then YY=2, the third day the cell is operated, YY=3, and so on.)
- "R" stands for "RUN"
- "zz" represents the number of the run during day
- "AA" represent the "type of data collected" – Use proper nomenclature as follows:
 - CO stands for "cross over" (to estimate hydrogen crossover)
 - CV stands for "cyclic voltammetry" (to estimate ECA – electrochemical surface area)
 - VI stands for "voltage-current" (performance or polarization curve)
 - CC stands for "constant current" (constant current operation)

D4.6 Nomenclature of the test condition (temperatures) is "B/C/D":

- "B" refers to T_{cell} in °C
- "C" refers to anode humidifier controller temperatures (both top and bottom) in °C
- "D" refers to cathode humidifier controller temperatures (both top and bottom) in °C

- When user controls gas lines, they are always 10 °C higher than T_{cell} .

D4.7 Handling of gas tanks

- At the beginning of EACH day, verify that all gas (N_2 , H_2 , Air, and O_2) cylinders have enough pressure (Above 13 atm or 200 psi).
- At the beginning of Day 1, OPEN gas tanks for N_2 , H_2 , and Air (all gases used that day). Set each gas regulator at 3.4 atm (50 psi).

D5 Day 1 Test

D5.1 Setup Cell Hardware on the Test Station

Time required for this step: ~ 30 minutes

- Use a multimeter to measure the resistance between two graphite plates (anode and cathode). Wait until the value is stabilized and record the exact value.

Note. This step is to see that the resistance value is high enough (~50 ohm) that there is no indication of short. If the resistance is lower than 10 ohm, rebuild the cell.

- Connect four gas tubes to the fuel cell hardware

Note 1. Make sure that anode and cathode plates correspond to those of the MEA inside.

Note 2. Make sure there is no leak on the INLET GAS LINES. Check with soapy water.

D5.2 Preparation of cell test on the test station

Time required for this step: ~ 5 minutes

- Insert the outside thermocouple into the end of a little hole on the graphite plate of the cell hardware for measuring the cell's temperature.

Caution: Make sure that the thermocouple stays at the end of the hole and use the scotch tape to keep it in place, if needed. If the thermocouple is not in place, the cell will be OVERHEATED to maintain a set point temperature (a cell failure).

- Switch both the cathode and anode gas valves on the test station to N_2 gas.

D5.3 Operation of computer to start up cell

Time required for this step: ~ 5 minutes

- Turn on Computer 1 AND Fuel Cell Load Unit 1.
- Select the icon for FuelCell software on MS Windows and open it.
- A menu box titled "SETUP CELL" (See Figure D2) will pop up. Set cell surface area as APPROPRIATE for the MEA tested (usually 25 cm^2). Set cell temperature at 25 °C

Note. If the Beep box is checked, FuelCell software program will produce a beep from the computer speaker when the Min or Max limits are exceeded.

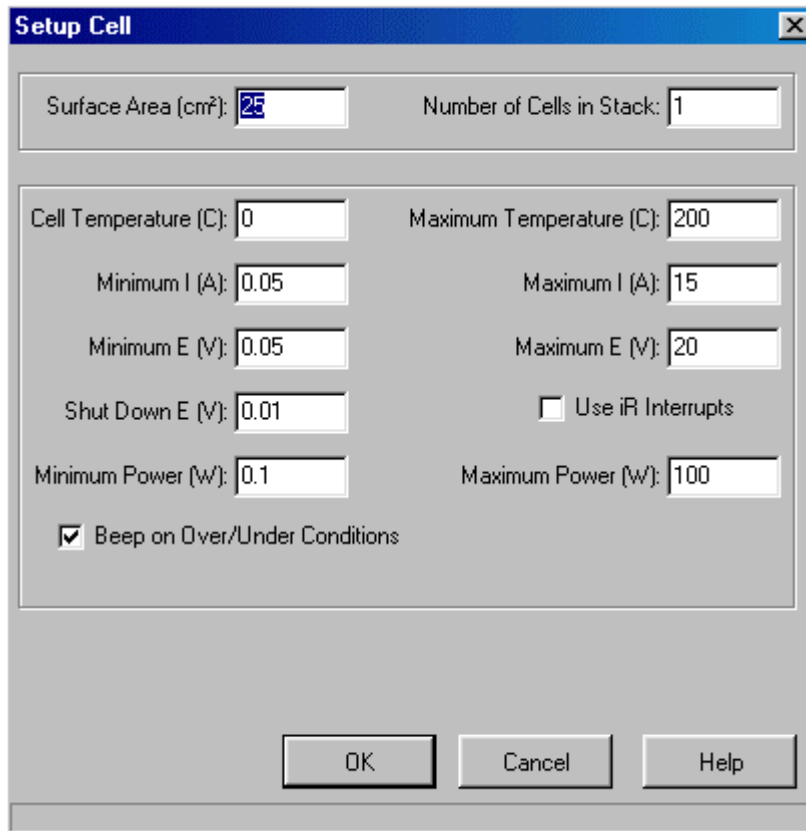


Figure D2. Set up Cell Menu

- iv. Another menu box titled “Setup Fuel Flow” (See Figure D3) will pop up. Set the flow rates at:
- Anode
 - Minimum Flow: 0.4 L/min
 - Load based flow: 0 L/min/cell + 0.023 L/min/Amp/cell (not used in this case)
 - Temperature: 25 °C
 - Cathode:
 - Minimum Flow: 0.4 L/min
 - Load based flow: 0 L/min/cell+ 0.066 L/min/Amp/cell (not used in this case)
- Temperature: 25 °C

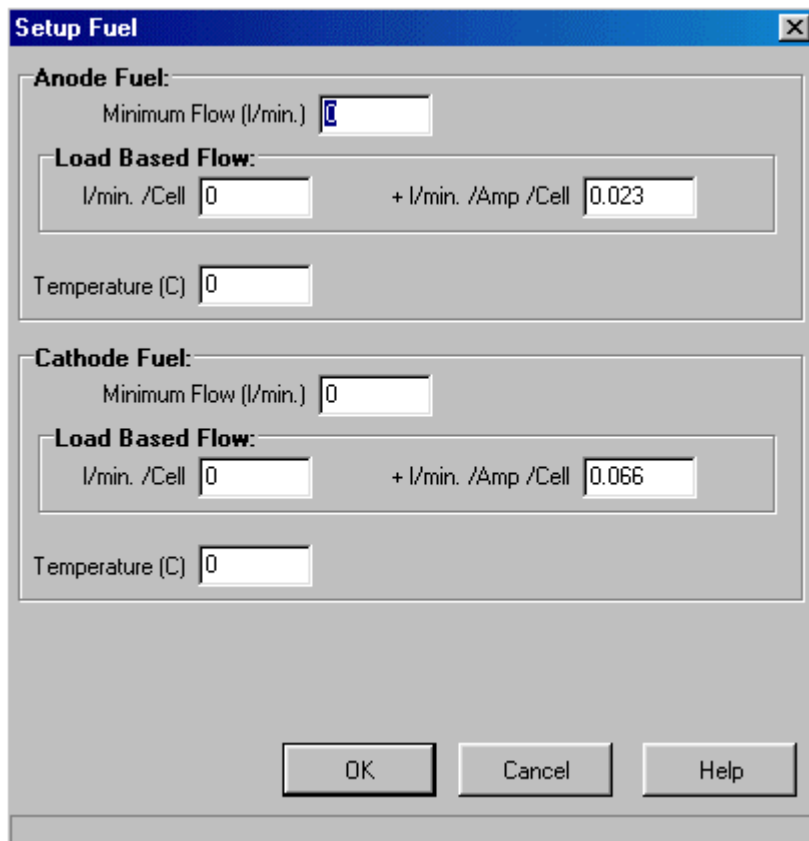


Figure D3. Setup Fuel Menu

D5.4 Crossover and Cyclic Voltammetry Tests

Time required for this step: ~ 40 mins

Note. This is a series of tests to determine the basic soundness of the cell to see if it meets minimum requirements.

- i. On the side of the test station, switch ANODE gas valve to H₂ and CATHODE gas valve to N₂.
- ii. Connect WORKING and SENSE electrodes to the CATHODE and the COUNTER and REFERENCE electrodes to the ANODE. (See Figure D4)
- iii. Turn on the Princeton Applied Research 263A (Connected to Computer 3).
- iv. Turn on Computer 3, which is used for measuring short-circuit, crossover current and cyclic voltammetry.
- v. Select the icon for CorrWare2 software on the main screen, and double click it to activate the program.
- vi. Right click at "Potentiodynamic" and select "Set up cell" option
- vii. Set "cell surface area" to 25 cm² and then click "OK" (see Figure D5)
- viii. Right click at "Potentiodynamic" and select "Set up instrument" option. Set "bandwidth" to high stability

- ix. Double click “Potentiodynamic” to wait until the OCP becomes lower than 0.1 V. Name file according to the Ionemem Database files (See Section D4.4) and select where it will be saved. Select 4 mV/s as the scan rate.

Note. This open circuit voltage value would be shown at the upper-right corner of the menu panel

- x. Select “Potentiodynamic” from experiment types and click the button for “Measure Selected” in toolbar and begin this measurement

Note. This green label is shown in the toolbar of the menu panel.

- xi. Right click at “Cyclic Voltammogram” and select “Set up instrument” option. Set “bandwidth” to high speed.
- xii. Double click “Cyclic Voltammogram” and name file according to the Ionemem Database files (See Section D4.4) and select where it will be saved. Select 30 mV/s as the scan rate.
- xiii. Select “Cyclic Voltammogram” from experiment types.
- xiv. Click at the button for “Measure Selected” in toolbar and begin this measurement;
- xv. Turn off the PAR263A when finished.
- xvi. Disconnect the electrodes (working, counter, sense, and reference) from the cell hardware plates

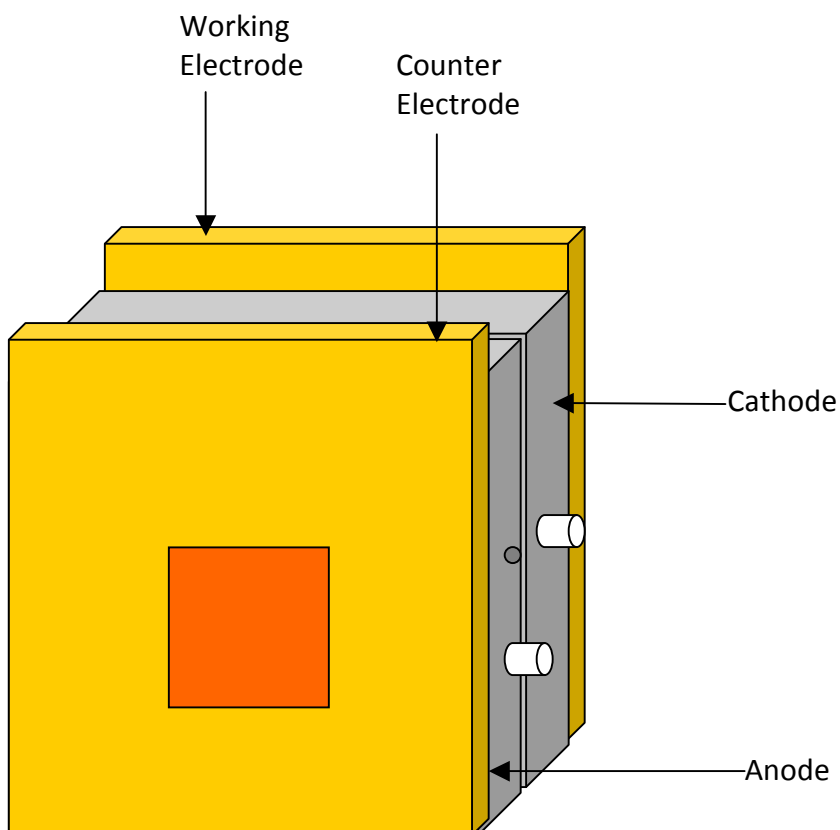


Figure D4. Connection of Electrode with Fuel Cell Hardware for CO/CV Measurements

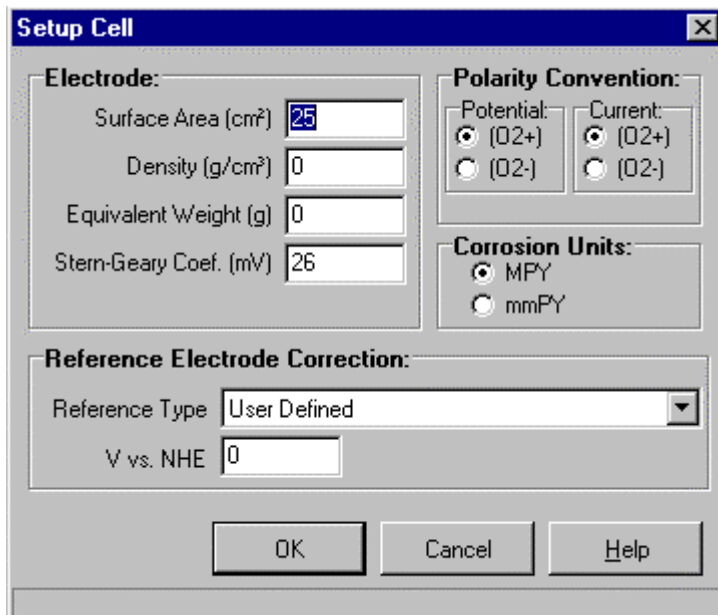


Figure D5. CorrWare Setup Cell Menu

D5.5 Humidification of the Membrane

Time required for this step: ~ 3.5 hours

Note. This step is performed to introduce water into the membrane to improve its ionic conductivity and reactant permeability.

- i. Switch the ANODE gas valve to H₂ and CATHODE gas valve to N₂.
 - ii. Connect the LOAD LINES to the fuel cell hardware:
- Connect the cathode load line (red line) directly with the cathode metal plate of the fuel cell.
 - Connect the anode load line (black line) directly with the anode metal plate of the fuel cell.

Caution: Be very certain that the connection is tight!

- iii. Insert the SENSE LEADS into the holes of the leads on the fuel cell hardware:
- Insert the cathode sense lead (red line) directly into the hole in the red line attached to the cathode graphite plate
 - Insert the anode sense lead (black line) directly into the hole in the black line attached to the anode graphite plate

Caution: Never connect the sense leads to the test fixture without the large LOAD connections being made first. Failure to do this with the load unit active may result in damage to the load.

- iv. Change all set points to the "HEAT UP" Step (40/50/50 condition)
- Set cell temperature at 40 °C
 - Set anode and cathode gas lines at 50 °C (If applicable). (Recall 10 °C higher than cell temperature)
 - Set anode and cathode humidifier controllers at 50 °C.
- v. After the temperatures reach the set points, hold it there for 5 to 10 min.
 - vi. Change all set points to the 80/80/73 condition:
- Set cell temperature at 80 °C

- Set anode and cathode gas lines at 90 °C (If applicable). Anode and cathode gas line temperatures are 5-10 °C higher than cell temperature (T_{cell}).
- On the test panel, set anode humidifier controllers at 80 °C (100% RH)
- On the test panel, set cathode humidifier controllers at 73 °C (75% RH)
 - vii. Set the flow rates at:
 - Anode
 - Minimum Flow: **0.17 L/min**
 - Load based flow: 0 L/min/cell + **0.021 L/min/cell** (for 3 stoichiometry of H₂)
 - Cathode:
 - Minimum Flow: **0.17 L/min**
 - Load based flow: 0 L/min/cell + **0.066 L/min/cell** (for 4 stoichiometry of Air)
 - viii. Maintain at this condition (H₂/N₂, 80/80/73) to humidify to membrane for **3 hours**.

D5.6 Operation of the Cell in a Break-In Mode

Time required for this step: ~ 3 hours minimum

Note. This test is performed to let the cell come to a steady state while operating in a benign way as a fuel cell.

- i. Set the condition to H₂/N₂, 80/80/73 °C condition (maintain previous step).
- ii. On the side of the test station, keep the ANODE gas valve on H₂ and switch the CATHODE gas valve to Air.
- iii. Wait until the cell voltage (which is the open circuit voltage at this point) stabilizes. (~ 3 minutes). Then record the exact open circuit voltage.

Note 1. The value of open circuit voltage will increase with the time. Just wait until it is stable.

Note 2. NEVER leave the cell at the OCV (Open Circuit Voltage) for more than 5 minutes. Platinum and carbon will corrode and cause performance decay. While changing conditions with fuel (hydrogen) and oxidant (air/O₂) present, always apply load at 100 – 400 mA/cm². This is to not leave the cell at OCV to avoid cell corrosion.

- iv. Click “Apply Fuel” and “Apply Load” buttons.
- v. Set the cell voltage at 0.55 V. Record the TOTAL current density to Value #1 (t = 0 hours).
- vi. Hold it for one hour, record the TOTAL current to Value #2 (t = 1 hour)
- vii. Hold it for another hour, record the TOTAL current to Value #3 (t = 2 hours).
- viii. Repeat the recording every hour until the TWO last current values are within 5% of each other. (Usually takes 3-5 hours)

D5.7 Cool-Down Procedure

Time required for this step: ~1 hour

- i. Set the cell temperature to 80/60/60 °C

Note 1. The cell temperature must be about 10-20 °C above the saturator temperatures during cool down.

Note 2. Water can be added to the saturators, and insulation can be removed from the cell to promote cool down.

- ii. While waiting for temperatures to reach the first set points, disconnect the positive load cable, to protect from current leak.

- iii. Set the min flow rate on the anode and the cathode at 0.2 L/min.
- iv. Switch the cathode valve on the side of the station to N₂ for purging the cell.
- v. Wait until the voltage falls to 0.4 V, then switch the anode valves in the front panel of the station to N₂ for purging the cell.
- vi. After the temperature reaches 80/60/60 °C, set it to 60/50/50, then 50/40/40, then 0/0/0.
- vii. Wait until the cell temperature, the humidifier temperatures, and the gas line temperatures are all below 50 °C.
- viii. Turn off the power of heater for humidifiers.
- ix. Set the anode and cathode flow rate back to zero.
- x. Click "Exit" to close the FuelCell software.
- xi. Turn off the Fuel Cell Load Unit
- xii. Close all valves of the gas cylinders.
- xiii. Close anode and cathode valves of the test station.

D6 Day 2 Test

D6.1 Crossover and Cyclic Voltammetry Tests

Time required for this step: ~ 0.5 hour

Repeat the procedure for measuring CO and CV on day 1.

D6.2 Polarization curve measurement at 80 °C (Air/O₂ alternating)

Time required for this step: ~ 7 hours (Air/O₂/Air)

Note. This test is performed to determine the cell performance at atmospheric pressure, which is the condition where the electrolyte is near saturation.

- i. Set the flow rates as done for the humidification.
- ii. Keep the ANODE gas valve on H₂ and switch the CATHODE gas valve to Air (1st Air run).
- iii. Let the cell reach OCV (~2-3 min). Record OCV.
- iv. Click the "Apply Fuel" button on the main window as shown in Figure D6.
- v. Click the "Apply Load" button on the main window as shown in Figure D6.
- vi. Apply 400 mA/cm² load. Maintain until voltage is constant.
- vii. If no experiments are listed under the "Setup Experiments" panel, click on "New..." button on the right of the panel, and select "Arbitrary control". If there are experiments listed in the "Setup Experiments" panel, double click one of them.
- viii. The menu box titled "Setup Arbitrary Control Experiment" will pop up
- ix. Choose a name and location for the data file (file with the results of the experiment).
- x. Select an appropriate control setup file for the experiment, then click "OK"

Note. Control Setup files are text files (i.e. ".txt" files) that are used to control the load. We are currently testing the following loads: 0, 10, 20, 40, 60, 80, 100, 200, 300, 400, 500, 600, 700, 800, 900, 1000, 1100, 1200, 1300, 1400, 1500, 1600, 1700, 1800, 1900, 2000 mA/cm², held at each point for 5 minutes.

- xi. Click the "Run Cell" Button on the main window

- xii. Select “Graphs” in menu bar, then click “vs Time” command and click “Voltage” as shown in Figure D7.
- xiii. The menu box titled “Graphs #1” will pop up.
- xiv. Change “Background data” to “Experiment data” at the upper-right corner of this menu box
- xv. Software starts data acquisition for a Cell Voltage vs. Time plot
- xvi. Wait until the experiment is finished. While the experiment is in progress, check regularly that the water level in the humidifier is not below the minimum level. Also check that the experiment is progressing without jumping to OCV.
- xvii. After the experiment is finished, the load should return to whatever load was being used prior to the experiment, in this case 400 mA/cm².
- xviii. Switch the cathode valve on the side of station to O₂ and wait until voltage stabilizes. Repeat the measurement in O₂ (at the same temperature).
- xix. After the measurement on O₂ is finished, switch the cathode valve back to Air. Repeat the measurement for air (2nd Air run).

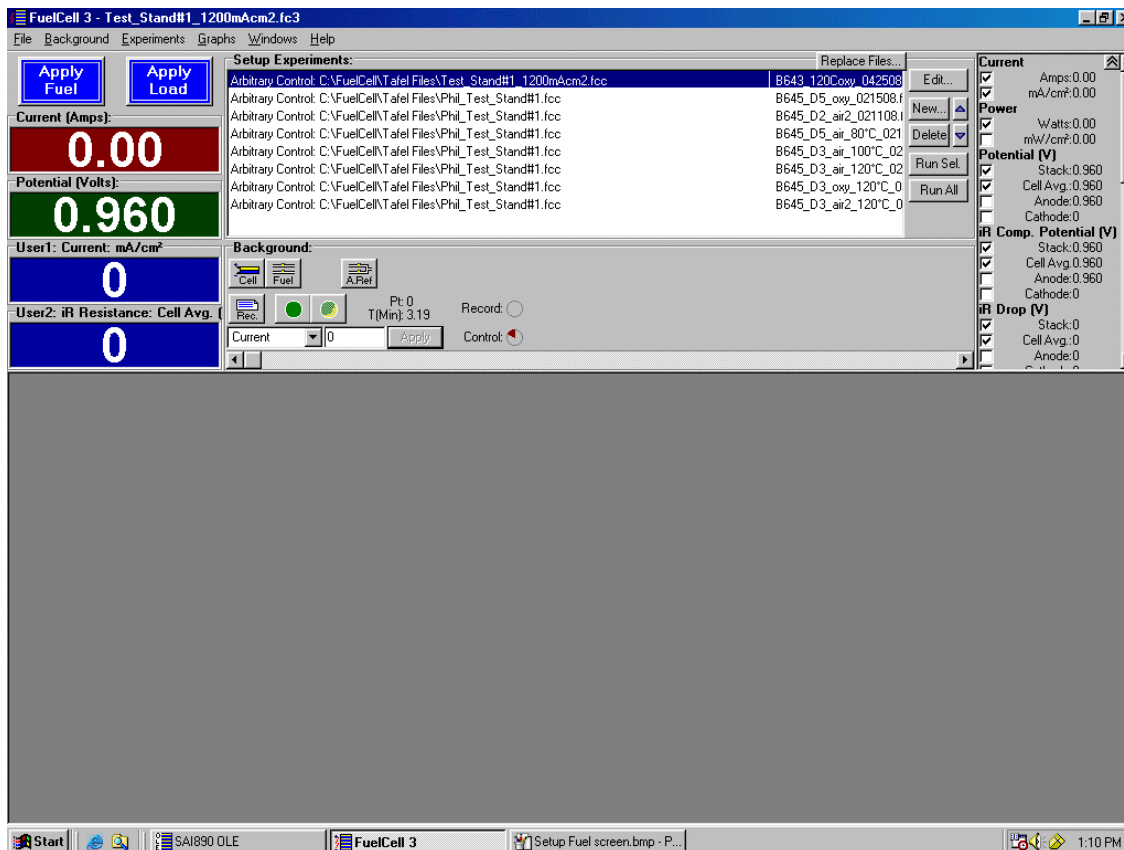


Figure D6. Main Window for FuelCell software

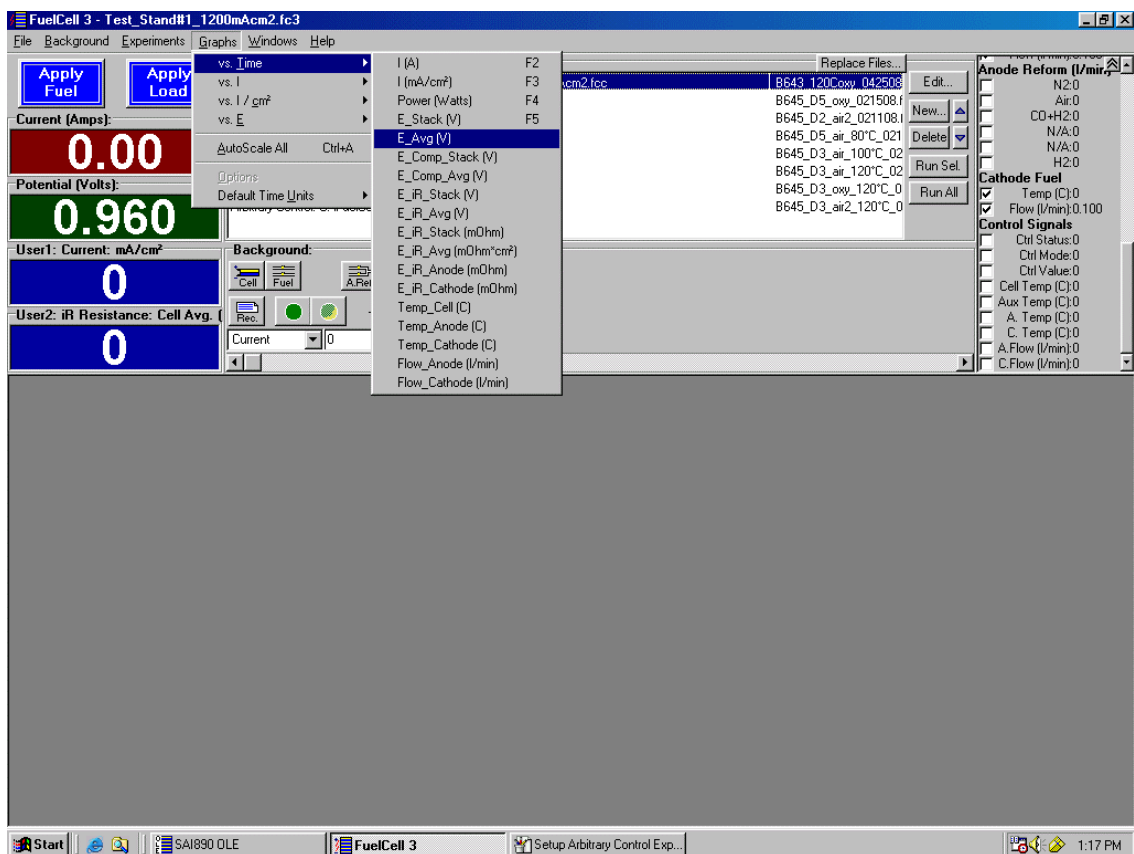


Figure D7. Choose Voltage vs. Time graph

D6.3 Crossover and Cyclic Voltammetry Tests at 80/80/73

Time required for this step: ~ 0.5 hour

Repeat the crossover and cyclic voltammetry procedure from day 1 at 80/80/73.

D6.4 Hold at 400 mA/cm² overnight

D7 Day 3 Test

D7.1 Performance Measurement at 80/80/73 (Air/O₂ Alternating, 1.5 atm)

Time required for this step: ~ 6 hours

- i. Pressurize cell to 1.5 atm.
- ii. Run the cell at the 80/80/73 condition with H₂/Air.
- iii. Wait until the whole sweep measurement is finished then repeat with oxygen and again with air.

D7.2 Performance Measurement at 100/90/90, Air, 1.5 atm

Time required for this step: ~ 2 hours

- i. Change the temperatures to the 100/90/90 condition (using H₂/Air)
 - Set cell temperature at 100 °C

- Set anode and cathode gas lines at 110 °C, if applicable
- Set anode humidifier controllers at 100 °C
- ii. Apply 400 mA/cm² of load, and wait until temperatures and voltage stabilize
- iii. Start the measurement of the performance using H₂/air
 - Wait until the whole sweep measurement is finished.

D7.3 Hold over night at 400 mA/cm²

D8 Day 4 Test

D8.1 Performance Measurement at 100/90/90 (O₂/Air Alternating, 1.5 atm)

Time required for this step: ~ 3 hours

- i. Run the performance test at the 100/90/90 condition with H₂/O₂
- ii. Wait until the whole sweep measurement is finished then repeat with air.

D8.2 Crossover and Cyclic Voltammetry Tests at 120/90/90

Time required for this step: ~ 1 hour

- i. Release pressure on cell to ambient
- ii. Change the temperatures to the 120/90/90 condition
 - Set cell temperature at 120 °C
 - Set anode and cathode gas lines at 125 °C (NOT 10 °C higher than T_{cell} as usual)
 - On the test panel, set anode humidifier controllers at 90 °C (same as previous)
- iii. Repeat the CO/CV procedure of Day 2

D8.3 Performance Measurement at 120/90/90 (Air/O₂ Alternating, 1.5 atm)

Time required for this step: ~ 3 hours

- i. Pressurize cell to 1.5 atm.
- ii. Change cathode gas to air
- iii. Apply 400 mA/cm² of load and wait until voltage stabilizes
- iv. Start the measurement of the performance using H₂/Air
- v. Switch to O₂, apply 400 mA/cm² while waiting
- vi. Run the performance test with H₂/O₂ then with H₂/Air

D8.4 Reduce Cell temperature to 100°C and hold overnight at 400 mA/cm²

D9 Day 5 Test

D9.1 Crossover and Cyclic Voltammetry Tests at 100/90/90

Time required for this step: ~ 1 hour

- i. Release pressure on cell to ambient
- ii. Change temperature to 100/90/90 condition
- iii. Repeat the crossover and cyclic voltammetry procedure of Day 1

D9.2 Performance Measurement at 100/90/90 (Air/O₂ Alternating)

Time required for this step: ~ 6 hours minimum (Air/O₂)

- i. Pressurize cell to 1.5 atm

- ii. Change cathode gas to air
- iii. Apply 400 mA/cm² of load and wait until voltage stabilizes
- iv. Start the measurement of the performance using H₂/Air
- v. When measurement is finished, switch to O₂ and apply 400 mA/cm² while waiting
- vi. Run the performance test with H₂/O₂.
- vii. When measurement is finished, switch to air and apply 400 mA/cm² while waiting
- viii. Run the performance test with H₂/Air.

D9.3 Stability Test at 100/90/90 (H₂/Air, 400 mA/cm², 1.5 atm, Days 5, 6, and 7)

- Under “Set-up experiments” click on “New...” button on the right of the panel and select “Constant current”
- Save the file under an appropriate name and input test time (~70 h) and current (400 mA/cm²)
- Collect condensate water from anode and cathode exhaust each morning and evening, in separate, labeled vials (end up with 12 vials total). Analyze condensate water for fluoride ions according to Appendix E.

D10 Day 8 Test

D10.1 Crossover and Cyclic Voltammetry Tests at 100/90/90

Time required for this step: ~ 0.5 hour

- i. Release pressure on cell to ambient
- ii. Repeat the crossover and cyclic voltammetry procedure of Day 2

D10.2 Performance Test at 100/90/90 (Air/O₂ Alternating, 1.5 atm)

Time required for this step: ~ 6 hours

- i. Pressurize cell to 1.5 atm
- ii. Repeat Performance measurement at 100/90/90 from day 5

D10.3 Cool-down

- i. Set the cell temperature to 80/60/60.

Note 1. The cell temperature must be about 10-20 °C above the saturator temperatures during cool down.

Note 2. Water can be added to the saturators, and insulation can be removed from the cell to promote cool down.

- ii. While waiting for temperatures to reach the first set points, disconnect the positive load cable, to protect from current leak.
- iii. Set the min flow rate on the anode (H₂) and the cathode (air) at 0.2 L/min.
- iv. Switch the cathode valve in the front panel of the station to N₂ for purging the cell.
- v. Wait until the voltage falls to 0.4 volts, then switch the anode valves in the front panel of the station to N₂ for purging the cell.
- vi. After the temperature reaches 80/60/60, set it to 60/50/50, then 50/40/40, then 25/25/25.

- vii. Wait until the cell temperature, the humidifier temperatures, the saturated gas water, and the gas line temperatures are all below 50 °C.

D11 Day 9 Test

D11.1 Repeat the crossover and cyclic voltammetry procedure of Day 2 (at room temperature)

D11.2 Leak test per Appendix C

D11.3 Resistance Test per Section D5.1

D11.4 Shut-down and Remove from the test stand

- i. Turn off the power of heater for humidifiers (if applicable).
- ii. Set the anode and cathode flow rate back to zero.
- iii. Click "Exit" to close the FuelCell software.
- iv. Turn off the Fuel Cell Load Unit
- v. Shut down computer.
- vi. Close all valves of the gas cylinders.
- vii. Close anode and cathode valves of the test station.
- viii. Disconnect gas tubing and electrical line from the cell hardware.

D11.5 Post-test

- i. Measure bolt load
- ii. Disassemble the Cell
- iii. Conduct visual Inspection
- iv. Bag and Seal Components With 10 drops of DI Water in each Bag

D12 Troubleshooting: Hardware problems

| INDICATION | CAUSE | CORRECTION |
|--|---|---|
| <ul style="list-style-type: none"> • No power when unit is turned ON • AC Power not available to model 890 | <ul style="list-style-type: none"> • Fuse on rear panel of the control unit may be blown | <ul style="list-style-type: none"> • Replace with 4 A, slow blow 3AG type fuse |
| <ul style="list-style-type: none"> • No heater power available for one or more of the temperature controller | | <ul style="list-style-type: none"> • Check internal fuses that protect the temp controller output. |

| | | |
|--|--|---|
| <ul style="list-style-type: none"> • Alarms when FUEL ON button is pressed in software | <ul style="list-style-type: none"> • Fuel Gas unit cables not connected • Model 890 Fuel System Interface Box not properly connected | <ul style="list-style-type: none"> • Check gas pressures and low pressure safety switch wiring. • Low gas pressure alarm switches must be closed for alarms to be off. |
| <ul style="list-style-type: none"> • No load current when load is turned ON | <ul style="list-style-type: none"> • Load cables not connected • Sense leads not connected or reversed • Sense lead protection fuse (internal fuse) blown | <ul style="list-style-type: none"> • Remove all power to system and disconnect anode cell lead and anode sense lead first • Resistance should measure between 10 and 15 W • If incorrect, remove the unit cover and check the fuse by removing it from its socket. |
| <ul style="list-style-type: none"> • Inadequate cooling air flow causing over-temperature shut down | | <ul style="list-style-type: none"> • Check cooling restrictions • Prolonged over-temperature operation may damage the load electronics. |

D13 Chart Summarizing Day-to-Day Activities

| Day | Time (h) | Temp | Gas A | Gas C | Action | Notes |
|---------------|----------|-----------|----------------|----------------|---------------------------|--|
| 1 | 0.5 | RT | N/A | N/A | Set-up | Includes leak test, resistance, etc. At 0.55V |
| 1 | 3.5 | 80/80/73 | H ₂ | N ₂ | Humidify | |
| 1 | 3 | 80/80/73 | H ₂ | Air | Break-in | |
| 1 | 1 | | H ₂ | N ₂ | Cool down | |
| 8h | | | | | | |
| 2 | 0.5 | 25/25/25 | H ₂ | N ₂ | CO/CV | |
| 2 | 2.5 | 80/80/73 | H ₂ | Air | IV | |
| 2 | 2 | 80/80/73 | H ₂ | O ₂ | IV | |
| 2 | 2 | 80/80/73 | H ₂ | Air | IV | |
| 2 | 0.5 | 80/80/73 | H ₂ | N ₂ | CO/CV | |
| 7.5h | | | | | | |
| 3 | 2 | 80/80/73 | H ₂ | Air | IV | 1.5 atm |
| 3 | 2 | 80/80/73 | H ₂ | O ₂ | IV | 1.5 atm |
| 3 | 2 | 80/80/73 | H ₂ | Air | IV | 1.5 atm |
| 3 | 1 | 100/90/90 | H ₂ | N ₂ | CO/CV | 1.5 atm |
| 3 | 2 | 100/90/90 | H ₂ | Air | IV | 1.5 atm |
| 9h | | | | | | |
| 4 | 2 | 100/90/90 | H ₂ | O ₂ | IV | 1.5 atm |
| 4 | 2 | 100/90/90 | H ₂ | Air | IV | 1.5 atm |
| 4 | 1 | 120/90/90 | H ₂ | N ₂ | CO/CV | |
| 4 | 1 | 120/90/90 | H ₂ | Air | IV | 1.5 atm |
| 4 | 1.5 | 120/90/90 | H ₂ | O ₂ | IV | 1.5 atm |
| 4 | 1 | 120/90/90 | H ₂ | Air | IV | 1.5 atm |
| 8.5h | | | | | | |
| 5 | 0.5 | 100/90/90 | H ₂ | N ₂ | CO/CV | |
| 5 | 2 | 100/90/90 | H ₂ | Air | IV | 1.5 atm |
| 5 | 2 | 100/90/90 | H ₂ | O ₂ | IV | 1.5 atm |
| 5 | 2 | 100/90/90 | H ₂ | Air | IV | 1.5 atm |
| 5 | .25 | 100/90/90 | H ₂ | Air | Stability | 1.5 atm, 400 mA/cm ² |
| 6.75h | | | | | | |
| 6 | | 100/90/90 | H ₂ | Air | Stability | 1.5 atm, 400 mA/cm ² |
| 7 | | 100/90/90 | H ₂ | Air | Stability | 1.5 atm, 400 mA/cm ² |
| 60-70h | | | | | | |
| 8 | 0.5 | 100/90/90 | H ₂ | N ₂ | CO/CV | |
| 8 | 2 | 100/90/90 | H ₂ | Air | IV | 1.5 atm |
| 8 | 2 | 100/90/90 | H ₂ | O ₂ | IV | 1.5 atm |
| 8 | 2 | 100/90/90 | H ₂ | Air | IV | 1.5 atm |
| 8 | 1 | | H ₂ | N ₂ | Cool down | |
| 7.5h | | | | | | |
| 9 | 0.5 | 25/25/25 | H ₂ | N ₂ | CO/CV | |
| 9 | 1 | | | | Leak and Resistance Tests | |
| 9 | 0.25 | | | | Disassemble Cell | |

Appendix E: Fluoride Concentration Measurements Test Procedure

E1 Checking Electrode Operation

Note 1. Check Electrode Operation every day before beginning measurements

Note 2. At all times use a stirring plate and bar.

Note 3. Before and after each measurement rinse electrode in DI water

E1.1 Suggested Equipment

- i. 2- 250 mL beakers (1 for measurement and 1 to rinse electrode)
- ii. 1- 10 mL measuring cylinder
- iii. 2- 50 mL measuring cylinders (1 for DI water and one for TISAB II)
- iv. 1- 2000 mL beaker (for waste)
- v. 1- DI water wash bottle
- vi. 1- glass pipette with bulb

E1.2 Procedure

- i. Mix 50 mL of DI water with 50 ml of TISAB II.
- ii. Immerse Electrode into solution
- iii. Pipette 1 mL of either 0.1 M or 100 ppm F⁻ standard into the mixture. Record the equilibrated reading (mV).
- iv. Add 10 mL more of the same standard to them mixture and again record the equilibrated reading (mV).
- v. The difference between the two readings should be in the range of 54 – 60 mV/decade at 25 °C. If that is not the case refer to TROUBLESHOOTING.

E2 Fluoride Concentration Measurements

Note 1. At all times use a stirring plate and bar.

Note 2. Before and after each measurement rinse electrode in DI water

E2.1 Direct Calibration

Note 1. For sample with concentrations greater than 1 ppm.

Note 2. Calibrate every day before beginning measurements

Note 3. Use three standards that cover the whole range of the concentrations of the samples that are measured (e.g. 1, 10 and 100 ppm)

E2.1.1 Suggested Equipment

- i. 4-250 mL beakers (3 for standards and 1 to rinse electrode)
- ii. 2-50 mL measuring cylinders (1 for TISAB II and one for DI water)
- iii. 1-2000 mL beaker (for waste)

- iv. 1 DI water wash bottle

E2.1.2 Procedure

- i. Mix 50 mL of the most dilute standard and 50 mL of TISAB II (for 1, 2 and 100 ppm the standards are already mixed with TISAB II and are used as given)
- ii. Insert electrode into solution and press “*Calibrate*”. Wait for an equilibrated reading (in ppm) and set the value to the value of the standard (e.g. 1 ppm).
- iii. Insert electrode into next standard and press “*Calibrate*”. Wait for an equilibrated reading (in ppm) and set the value to the value of the standard (e.g. 10 ppm).
- iv. Repeat with final standard (e.g. 100 ppm)
- v. Press “*Measure*”. Record slope that is given (mV)

The electrode is now calibrated for the range of 1 to 100 ppm. Checking the electrode against the standards during measurement is recommended.

E2.2 Calibration Curve

Note 1. For samples with concentrations lesser than 1 ppm

Note 2. Make a calibration curve every day before beginning measurements

Note 3. Use low level TISAB (LL-TISAB, made in-house using procedure in E2.2.1) for all measurements

E2.2.1 Making LL-TISAB

Use very pure reagents – avoid metal ion contamination, especially aluminum and iron as they form complexes with fluoride

E2.2.1.1 Suggested Equipment

- i. 1-2000 mL beaker (for bulk solution)
- ii. 1-100 mL measuring cylinder
- iii. 1-calibrated pH electrode
- iv. 1-250 mL beaker (for rinsing electrode)
- v. 1-glass pipette with bulb
- vi. 1-DI water wash bottle
- vii. 1-2000 mL volumetric flask (for LL-TISAB)
- viii. 1-500 mL volumetric flask (for NaOH)

E2.2.1.2 Procedure

- i. Add ca. 1000 mL of DI water to the large beaker
- ii. Add 114 mL of pure glacial acetic acid
- iii. Add 116 g of pure sodium chloride and stir
- iv. Weigh 100 g of sodium hydroxide into volumetric flask and make up to 500 ml with water (cool in ice if necessary)
- v. Immerse electrode into large beaker and add 5M NaOH until a pH of 5.0 – 5.5 is achieved (make sure all solutions are at the same temperature)

- vi. Put LL-TISAB into volumetric flask and make up to 200 ml

E2.2.2 Making Standard

This standard is used to make the Calibration Curve

E2.2.2.1 Suggested Equipment:

- i. 1 400 mL beaker
- ii. 1 5 mL pipette with pump
- iii. 2 50 mL measuring cylinders

E2.2.2.2 Procedure:

- i. Measure 5 mL of 100 ppm F^- standard into a measuring cylinder and make up to 50 mL
- ii. Measure 50 mL of LL-TISAB and combine the two solutions in the beaker

E2.2.3 Making Calibration Curve

The Calibration Curve is needed to convert from mV to ppm

E2.2.3.1 Suggested Equipment:

- i. 2 - 250 mL beakers (1 for measurement and 1 to rinse electrode)
- ii. 1- 1 mL pipette with pump
- iii. 2 - 50 mL measuring cylinders (1 for LL-TISAB and one for DI water)

E2.2.3.2 Procedure:

- i. Measure 50 mL of DI water and 50 mL of LL-TISAB into a beaker
- ii. Rinse, immerse electrode into solution and record the reading (mV)
- iii. Add 1 mL of the standard, leave to equilibrate and record the reading (mV)
- iv. Repeat 3 until all expected concentrations of F^- are covered (at least 15 mL are recommended)
- v. Plot a semi-logarithmic graph of mV against ppm

Note. Addition of 1 mL of standard is ca. 0.1 ppm, 5 mL of standard is ca. 0.47 ppm and 10 mL of standard is 0.9 ppm

E2.3 Measuring Samples with Concentrations Greater than 1 ppm

Note. For samples with concentrations greater than 1 ppm, direct calibration of electrode is required. Use TISAB II for all measurements.

E2.3.1 Suggested Equipment:

- i. 1- 5 mL pipette with pump (for samples)

- ii. 2- 250 mL beakers (1 for measurement and 1 to rinse electrode)
- iii. 2 - 50 mL measuring cylinders (1 for DI water and one for TISAB II)
- iv. 1- 2000 mL beaker (for waste)
- v. 2 - 1000 mL beakers (1 for DI water and one for TISAB II)
- vi. 1- glass pipette with bulb (for TISAB II)
- vii. 1- DI water wash bottle

E2.3.2 Procedure:

- i. Measure 5 mL of sample into measuring cylinder and make up to 50 mL with DI water
- ii. Measure 50 mL of TISAB II and combine both cylinders in a beaker
- iii. Rinse electrode, immerse in beaker, leave to equilibrate and record the reading (ppm)
- iv. Repeat with other samples

Input all data into Excel, convert to $\mu\text{moles}/(\text{cm}^2 \text{ h})$ (equation: $\text{ppm} \times 2/(25 \times 19)$) and plot vs hours.

E2.4 Measuring Samples with Concentrations Less than 1 ppm

For samples with concentrations lesser than 1 ppm, use low level TISAB (LL-TISAB) for all measurements

E2.4.1 Suggested Equipment:

- ii. 1- 5 ml pipette with pump (for samples)
- iii. 2 - 250 mL beakers (1 for measurement and 1 to rinse electrode)
- iv. 2 - 50 mL measuring cylinders (1 for DI water and one for LL-TISAB)
- v. 1 - 2000 mL beaker (for waste)
- vi. 2- 1000 mL beakers (1 for DI water and one for LL-TISAB)
- vii. 1 - glass pipette with bulb (for LL-TISAB)
- viii. 1 - DI water wash bottle

E2.4.2 Procedure:

- i. Measure 5 mL of sample into measuring cylinder and make up to 50 mL with DI water
- ii. Measure 50 mL of LL-TISAB and combine both cylinders in a beaker
- iii. Rinse electrode, immerse in beaker, leave to equilibrate and record the reading (mV)
- iv. Repeat with other samples

Use Calibration Curve to determine the ppm values for each mV reading. Input all data into Excel, convert to $\mu\text{moles}/(\text{cm}^2 \text{ h})$ (equation: $\text{ppm} \times 2/(25 \times 19)$) and plot vs hours.

E3 Electrode Cleaning and Storage

E3.1 Electrode Cleaning I

Note. If electrode is not functioning properly, e.g. wrong slope or drifting of values, then it may help to clean the electrode

- i. Hold electrode in one hand and use thumb to press cap down, thereby draining the electrode
- ii. Rinse and drain with DI water and then refill to just below the hole with Electrode Filling Solution

E3.2 Electrode Cleaning II

If the above does not help this is known to help:

- i. Using a rubber glove or KimWipe rub the electrode for a minute with some ordinary toothpaste (acts as a slight abrasive)
- ii. Rinse thoroughly with DI water and soak in DI water for an hour
- iii. If desired, refill the filling solution as above

E3.3 Electrode Storage I

Note. This is for when the electrode will be used within a week

If the electrode will not be in use for a week or less, store the electrode in the lowest concentration standard (i.e. 1 ppm F⁻)

E3.4 Electrode Storage II

Note. This is for when the electrode will not be used within a week

- i. If the electrode will not be used for a longer period of time, drain and rinse the electrode with DI water as described in A1 and leave to dry.
- ii. Put cap on electrode and store dry in a safe place.

Appendix F: Safety Plan

Contract Number: DE-FC36-06GO16028

Prepared By: Darlene Slattery

Date: June 30, 2006

Modified: May 1, 2007

Approvals

James R. Fowler, FSEC CHO

Ali T-Raissi, HRD Division Director

James M. Fenton, FSEC Director

James Uhlir , UCF EH&S Director

Project Title: Lead Research and Development Activity for DOE's High Temperature, Low Relative Humidity Membrane Program

Contract Number: DE-FC36-06GO16028

Prepared By: Darlene Slattery

Date: June 30, 2006

Project Officer: David Peterson

F1 Scope of Work

Florida Solar Energy Center (FSEC), a research institute of the University of Central Florida, has been designated as the lead organization for DOE's High Temperature, Low Relative Humidity Membrane Program. As such, FSEC's research and development team will prepare and evaluate new polymeric electrolyte phosphotungstic acid composite membranes. Additionally, FSEC will develop standardized experimental methodologies to 1) measure conductivity (in plane and through-plane) 2) characterize mechanical, mass transport, and surface properties of the membranes and 3) predict durability of the membranes and their membrane electrode assemblies.

Work to stabilize and reduce particle size of the phosphotungstic acid that has led to the highest conductivity membranes and highest performance membrane electrode assemblies, which surpass Nafion[®], at 100 – 120 °C and low relative humidity will continue. Non-Nafion[®] based poly[perfluorosulfonic acids] (PFSA) of equivalent weight lower than 1100; sulfonated poly(ether ether ketone)s (SPEEKs) with various sulfonation degrees; sulfonated poly(ether ketone ketone) (SPEKK) as the proton-conducting component in a blend with either poly(ether sulfone) (PES) or SPEKK with different sulfonation levels will be fabricated into new composite membranes containing small particle stabilized phosphotungstic acid. This team, working with the fuel cell community, will develop standardized experimental methodologies to 1) measure conductivity as a function of relative humidity and mechanical properties of membranes, 2) characterize mechanical, mass transport, and surface properties of the membranes, and 3) predict durability of the membranes and their membrane electrode assemblies fabricated by the team for both the in-house research program and for membranes provided by the High Temperature, Low Relative Humidity Membrane Working Group (HTMWG) members. FSEC will provide the HTMWG with standardized tests and methodologies and short course education offerings on these test methodologies along with membrane electrode assembly fabrication techniques so that at the end of three years all research program members will be able to perform this work in their own facility. An easily-implemented protocol and rapid test apparatus for evaluating the through-thickness conductivity (or resistance) of membranes over a broad range of conditions will be developed. FSEC will use its experience in developing accredited standardized test methods for the solar thermal, photovoltaic and building energy efficiency industries to support this activity.

This program will be conducted within the Hydrogen R&D Division at the Florida Solar Energy Center, located in Cocoa, Florida. The facilities within the division include:

- Hydrogen Lab I - 2,000 square foot chemical lab
- Hydrogen Lab II - 1,800 square foot chemical lab
- Integrated Fuel Cell Test Bed (IFCT) facility – a 900 square foot fuel cell lab

Research activities in these facilities include:

- Alternative fuels
- Fuel cells
- High-pressure, high-temperature reactions
- Hydrogen energy systems
- Hydrogen membrane separation
- Hydrogen production and storage
- Material failure determination
- Material property determination
- Nano synthesis and materials development
- Photo and thermo-catalytic reaction and reactor engineering
- Photocatalytic and photoelectrochemical processes
- Pollutant detoxification
- Sensors and detectors
- Synthesis of metal hydrides and chemical hydrides
- Thermal imaging

The researchers in the Division are scientists and engineers with extensive backgrounds in all aspects of hydrogen research. Nine of the staff researchers have Ph.D.s and three have M.S. degrees. They are assisted by a number of students, which varies for any given semester.

Ultimate responsibility for meeting deliverables will rest with the project PI. However, he has appointed a Project Manager who will be responsible for oversight of the day to day tasks involved in the project. The Project Manager will work closely with the FSEC Chemical Hygiene Officer (CHO) to ensure that all tasks are carried out in a safe and responsible manner.

F2 Identification of Safety Vulnerabilities

The University of Central Florida has a “Chemical Hygiene Plan” that the Florida Solar Energy Center follows for all programs. Under that plan, the Department of Environmental Health and Safety Chemical Hygiene Officer (CHO) is tasked with reviewing contract proposals to determine the inclusion of hazardous chemicals or procedures. When they are identified, EH&S consults with the proposal’s Principal Investigator (PI) to ascertain the level of risk. It is the responsibility of the PI to choose the best method for project analysis. FSEC also has its own CHO who assists the FSEC PI’s with chemical hygiene and compliance, and who maintains FSEC’s chemical safety documentation.

To fulfill the requirements of this contract, researchers will prepare polymer solutions and modify them by the addition of phosphotungstic acid and/or via sulfonation. Membranes will be cast and tested. Tests will include in-plane and through-plane conductivity, swelling,

mechanical strength, chemical strength and glass transition temperature. Membranes deemed worthy of additional analysis will be catalyst coated and developed as a membrane electrode assembly, MEA.

Safety vulnerabilities are identified using a “What if” analysis. A process flow chart for the project was developed and each area was reviewed by the research team. Vulnerabilities thus identified include exposure to organic vapors during membrane fabrication; fire resulting from improper preparation or handling of catalysts; hydrogen leakage from a cylinder, regulator, or system; and loss of ventilation because of power failure in facility.

F3 Risk Mitigation Plan

F3.1 Organic Vapor Exposure

A number of solvents will be used in the preparation of the membranes. Because of the impact of humidity on the membranes, it was determined that membrane preparation was best carried out in the controlled environment of a glove box. A glove box dedicated to this process along with the appropriate personal protective equipment, PPE, will protect the membranes while ensuring that the scientist is not exposed to the solvents during the casting process.

F3.2 Catalyst Handling

The finely divided catalysts on support material are known to be flammable when an alcohol is added to the dry material (SOP). A procedure, that includes the addition of water before the alcoholic solvent is added, which has been verified in earlier programs, will be used. Additionally, catalyst spraying will be carried out in a fume hood to limit worker exposure. Organic materials, such as paper toweling, will not be used in the hood where catalysts are being sprayed.

F3.3 Hydrogen Leaks

Hydrogen leaks will be determined with both a hand held detector for checking systems and with an area detector in the area most prone to the presence of hydrogen. Hydrogen that flows through a fuel cell unreacted will be vented directly to a fume hood. In the event of a facility power failure that would result in a system shutdown, a valve will automatically shutdown the flow of hydrogen to the system. Gas cylinders not currently in use- hydrogen as well as other gases- will be capped and removed to the storage area that is separate from the laboratory. Before every procedure involving the use of hydrogen, the entire system will be checked for leaks using the hand held leak detector.

Maximum amounts of hydrogen to be used have been calculated on the premise that all test stands are simultaneously in use. In such an instance, there would be less than 8 L/min of hydrogen flowing. This was determined based upon the following:

1. Single Cell:

Assuming
25 cm² MEA

80 °C operation

H₂ Stoichiometry = 3

1.5 A/cm² – max

H₂ flow = 9.01 mL/min@ 1A x 25 x 3 x 1.5 = 1014 mL/min

2). Endurance Test Cell = 1014 mL/min – max

3). Diagnostic Test Cell = 1014 mL/min – max

4). 4-Cell Stack = 4 x 1014mL/min = 4056 mL/min – max

5). Conductivity Test Cell: 500 mL/min

Add 1) through 5) gives

= 1014 + 1014 +1014 + 4056 + 500 = 7598 mL/min = 7.958 litre/min

F3.4 Standard Operating Procedures

Before initiating any activity, a standard operating procedure (SOP) will be identified. If none has been identified for the particular activity, it will be developed in collaboration with the team member most familiar with the process. Once developed, the SOP will be approved by an internal HR&D Procedures Committee, headed by the Project Manager, before it is put into practice. Any changes to an SOP must be put in writing, with justifications and possible ramifications and approved by the committee. All SOPs will be signed by the developer, dated and, once approved by the Procedures Committee; a copy will be placed at the point of use. Additional copies will be available in the CHOs office and on CD in each laboratory.

Current SOPs include:

- Preparation of membrane solutions
- Manufacture of membranes
- Protonation of membranes
- Heat treatment of MEAs
- Membrane leak test
- PEMFC unit cell test
- Catalyst deposition

Additional SOPs currently under development include

- In-plane conductivity test
- Through-plane conductivity test
- Water uptake measurement
- Glass transition temperature determination

In addition to SOPs for individual tests, under the Chemical Hygiene Plan, all researchers in the laboratory are required to wear safety goggles, lab coats and, when handling chemicals, the

correct gloves. They are also required to wear closed toed shoes and no food or drink is allowed anywhere in the labs.

F3.5 Previous Experience with Hydrogen

All researchers in the Hydrogen R&D Division have extensive experience with hydrogen. While the junior members have as few as 3 – 5 years of hydrogen related experience, senior members have as much as 30 years of experience. In addition to experience in assembling and testing of fuel cells and fuel cell components, various researchers within the group are experts in cryogenics, hydrogen storage in metal hydrides, chemical hydrides and complex hydrides, hydrogen production via electrolysis, photocatalytic water splitting, and several reformation processes and separation processes using hydrides and membrane materials. As a result of this experience, all are qualified in the safe handling of hydrogen and systems, which use or produce hydrogen.

In the 17 years that FSEC's hydrogen laboratories have existed, only four incidents have occurred, three of which were very minor and none of which resulted in injuries. Only one was directly related to hydrogen and was the result of a failed O-ring in a system. All incidents were thoroughly reviewed, weaknesses identified and procedures modified. Each incident emphasized that safety precautions in place minimized damage to the surroundings.

F3.6 Safety Performance Measurement and Management of Change Reviews

Checklists have been developed and will be used to record accomplishment of the steps in a procedure. These checklists will be filed in binders kept at the location of each activity. Periodic monitoring of the checklists will be conducted by the project manager to ensure that procedures are being followed.

The FSEC CHO will make monthly laboratory inspections to determine the presence of any hazard. Identified hazards will be corrected in cooperation with the project PI, the researcher and the CHO and any necessary changes to procedures will be made. To minimize repeat hazards, remedial training in safe practices will be provided, if necessary.

A checklist of the inspection will be made and filed in a binder in the CHO's office. The checklists will be reviewed periodically to determine the need to make procedure changes to eliminate repeat hazards.

Before changes are made to any equipment or procedures and before any new activities are initiated, a review will be conducted with the researcher(s) involved, the project manager, the CHO and others as deemed necessary by the project manager.

F3.7 Employee Training

The University of Central Florida's Environmental Health and Safety Department, EH&S, requires all laboratory personnel to take a Chemical Safety and Environmental Management course. This course is given at the beginning of each term on the main campus of the university or can be requested at anytime for groups. In addition to the initial course for new employees, all employees are required to take a refresher course yearly.

In addition to the EH&S provided training, training videos on a variety of subjects have been obtained and made available to all employees. New employees are required to view the videos and complete the corresponding worksheet, which is filed by the CHO. Topics of these videos include general lab practices such as the use of glassware and balances, and a general safety video that includes use of fire extinguishers and handling of gas cylinders.

The topics of this course include basic lab practices, chemical hygiene, waste management, and fire safety. More specific training is provided by EH&S for other areas such as Radiation safety and Biohazard Safety. The CHO or the individual researcher is responsible for training on specific instruments or any specialized apparatus or equipment in the laboratory. The FSEC CHO and the PI train new personnel on the safe handling of hydrogen and other flammables to be used for a project. Training includes proper handling of gas cylinders, leak checking, proper venting and purging of systems and materials compatibility. Refresher training requirements are determined by the CHO or PI based upon employee performance. The CHO maintains electronic and hard copies of the training history of all laboratory personnel as part of the Chemical Hygiene Plan.

F3.8 Equipment Integrity

Much of the testing of the membranes will be accomplished using 850C Compact Test Stations from Scribner Associates. These test stations feature automatic shutdown and N₂ purge with under voltage, over current, over temperature, loss of supply pressures, low water, communications failure or external alarm. Additionally, there is a manual switch for emergency shutdown. The test stands were installed and qualified by Scribner Associates. Routine inspection of these units will be carried out by the researchers who will be using them. Any maintenance of the units will be done in consultation with Scribner Associates.

New equipment or systems that are installed will be reviewed by the CHO and Project Manager before being certified for usage. To be certified, a "What if" analysis will be conducted and any safety vulnerabilities identified and procedures developed to mitigate the vulnerabilities.

Routine maintenance of the laboratory safety equipment is conducted by the lab manager/CHO. Eye wash stations and showers are tested monthly and results recorded. The face velocity of fume hoods is verified semi-annually by UCF EH&S personnel and results indicated on each hood. Fire extinguishers are checked periodically by the State Fire Marshall, while sprinkler systems and fire alarms are checked by FSEC Operations personnel.

F3.9 Maintenance of Safety Documentation

The Chemical Hygiene Plan is maintained by the FSEC CHO and a copy is available to staff in his office, which is located within Hydrogen Lab I. It is additionally available online via the University EH&S website. See the reference section for details.

Material Safety Data Sheets, MSDS, are filed in a set of binders and are available for all substances currently in the inventory or used previously within the labs. The CHO is responsible for obtaining and filing the MSDS and they are continuously available in his office. Additionally, MSDS may be accessed from a general use computer in the laboratory.

F4 Communication Plan

F4.1 Emergency Response Plan

UCF has an Emergency Management Plan, available on the web at <http://www.ehs.ucf.edu/Emergency/EMP%202006-2007.pdf>. This plan outlines the procedure for minor emergencies, such as a spill of less than 4 L of a chemical that is not acutely hazardous or an unknown. In this Plan, employees are advised to notify the laboratory manager or the principal investigator, PI, and use the available spill kits to clean up under the supervision of the PI.

In the event of a major emergency, such as a fire, explosion or a spill of more than 4 L or one that involves a chemical that is acutely hazardous, FSEC personnel have been instructed to leave the lab and dial 911 immediately from a safe location. In addition, Brevard County (where FSEC is located) has a Special Response Team for hazardous materials that is available 24/7 and is trained to deal with chemical spills or other incidents. Their number is posted by laboratory telephones but they may also be summoned by requests to 911.

Each laboratory has posted on the door a list of phone numbers of individuals to call in the event of an incident within that laboratory. Non-emergency issues are reported to these contact individuals. Laboratories have spill kits, fire extinguishers, fire alarms, safety showers and eye wash stations. Most FSEC labs also have glass view panels in the walls for quick visual assessment of physical conditions in the lab.

F4.2 Incident Reporting and Lessons Learned

All incidents are reported to the individual supervisor and FSEC's laboratory manager/CHO. Incidents that result in damage or injury must be written up for submission to the Environmental Health and Safety Department on the main campus. If damage or injury has occurred, the incident is presented to and is discussed at the next FSEC Executive Committee Meeting and a decision is made on further action.

At a minimum, the individual involved and his/her supervisor reviews the activities leading up to the incident, determines a probable cause and modifies procedures and/or the set-up in order to avoid a recurrence

As required in Section 4.3 of the Communications Plan of the DOE "Guidance for Safety Aspects of Proposed Hydrogen Projects," all incidents and near-misses must be reported to DOE.

F5 Sample Handling and Transport

The materials that are expected to be transported will be membranes fabricated by other members of the HTMWG and shipped to us for testing. These are not considered hazardous materials and, therefore, their transport is not an issue.

UCF and FSEC have procedures for shipping materials that are considered hazardous. A member of the EH&S Department is qualified to package such materials for shipment. When the need arises for such shipments, packing materials that meet DOT guidelines are obtained and the EH&S employee comes to FSEC and prepares the package.

F6 References

UCF Environmental Health & Safety
Chemical Safety Information, CHP
Emergency Information, UCF
City of Cocoa, Florida (Police & Fire)
Brevard County, Florida (Hazmat)

www.ehs.ucf.edu

www.ehs.ucf.edu/chemical/main.html

www.ehs.ucf.edu/emergency/main.html

www.cocoaf1.org/City%20Services/

www.brevardcounty.us/fire_rescue/

Appendix G

Facilities and Instrumentation

Facilities exist at FSEC and the University of Central Florida's Material Characterization Facility Advanced Materials Processing and Analysis Center to allow complete fabrication and characterization of membranes and membrane electrode assemblies.

G1 Membrane Fabrication

- i. Controlled temperature and humidity fabrication stations containing several levelling tables
- ii. Oven equipped with a vacuum (Isotemp Vacuum Oven Model 281A)
- iii. Balances

G2 Membrane Electrode Assembly Fabrication

- i. Ultrasonic blender (Fisher® Scientific Gen 1000 Homogenizer)
- ii. X-Z Spray system
- iii. Screen-printing Machine
- iv. Hot press (Carver Accustamp)
- v. Oven (Fisher Scientific Isotemp Oven)

G3 Electrochemical Characterization

- i. 4 test stations (Scribner Associates Fuel Cell Test Systems 850C (3) and 890 (1)), with MEADS coming soon (Scribner Associates 8 cell test station)
- ii. 3 Potentiostats (Princeton Applied Research 263A (2) and 273 (1))

G4 Materials Characterization

- i. X-Ray Diffraction
- ii. Transmission Electron Microscopy
- iii. Scanning Electron Microscopy (with Energy Dispersive X-ray Analysis)
- iv. X-Ray Photoelectric Spectroscopy
- v. Auger Spectroscopy
- vi. Differential Scanning Calorimetry
- vii. Thermal Gravimetric Analysis
- viii. Fourier Transform Infrared Spectroscopy with Attenuated Total Reflectance
- ix. Mechanical Tensile Strength Testing
- x. Fluoride Ion Electrode Analysis
- xi. Ion Chromatography
- xii. Gas Chromatography with Mass Spectrometry