# FSEC's MEA Test Protocol

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# Outline

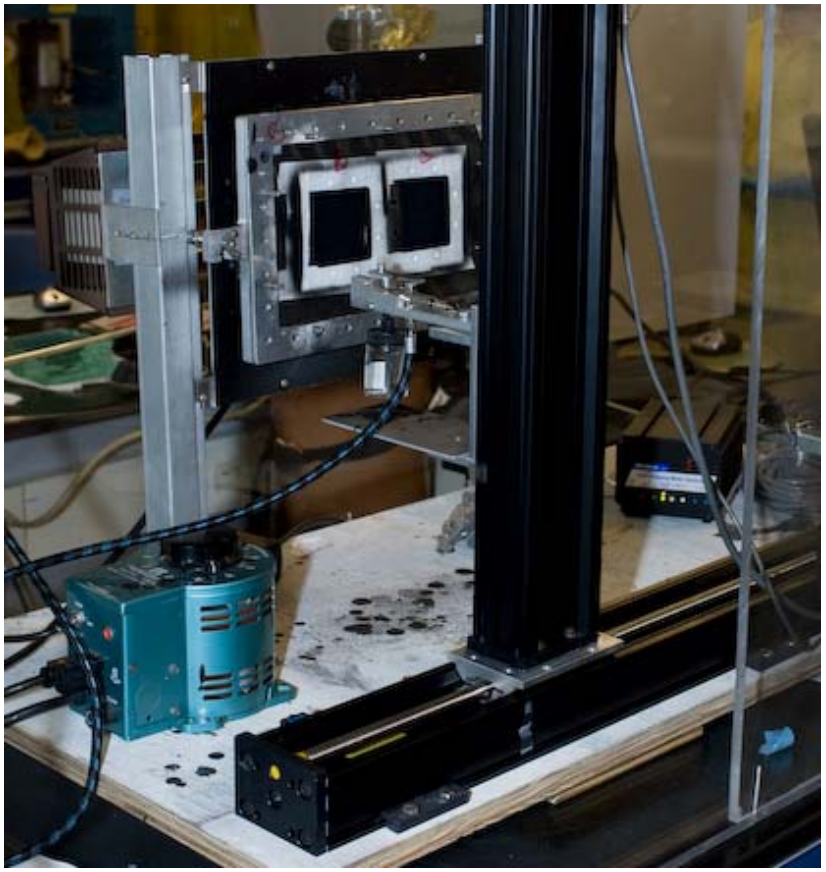
- Goals
- Test Components
  - Materials Used, Tests Run
- Day to Day MEA Test Sequence
  - Days 1-9
- Cell Report
- Summary

# Goals

- Define test procedures used at FSEC to conduct screening tests under FSEC's Task 5 "Characterize performance of MEA" of the DOE program DE-PS36-6095020
- Assess merits of membranes submitted by Task 1 team members
- Show current capability of membranes rather than simply provide Go/No Go rating against Program Requirements
- Clearly define operating conditions
  - Allows details to be understood by the DOE
  - Each team member can set limits of processing and operation based on capabilities of their membranes
  - All membranes tested to same protocol, so results provide a fair and accurate evaluation of performance capabilities
  - Impact of fabrication history, characteristics of cell components, cell assembly procedures, cell operating history, normalized by using identical procedures on all cells

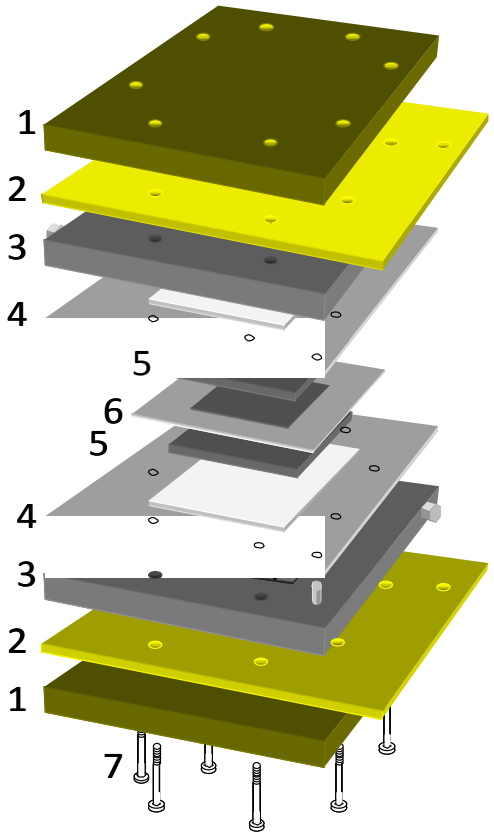
# Materials - CCM

- CCM from membranes provided by Task 1 team members
  - Pieces of membrane sent for conductivity testing and for SEM/EDAX characterization
  - Membrane pairs coated with:
    - 0.4 mg/cm$^2$ TEC10E50E -- Pt 50 wt% catalyst supported on HSAC (SA = 800 m$^2$/g) supplied by TKK
    - Catalyst mixed with 30% 1100EW Nafion® then sprayed using a spray gun, which is carried by a numerically-controlled X/Z plotter
    - CCM dried at 80 $^o$C, hot-pressed at 136 $^o$C and 75 psi, and protonated
  - Second CCM made at the same time, with identical processing returned to membrane fabricator untested for evaluation and analysis



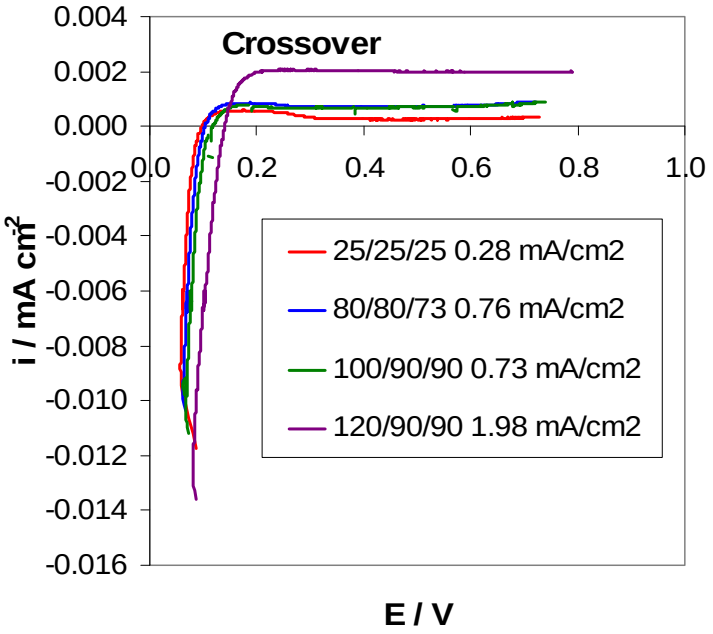
# Cell Details and Assembly

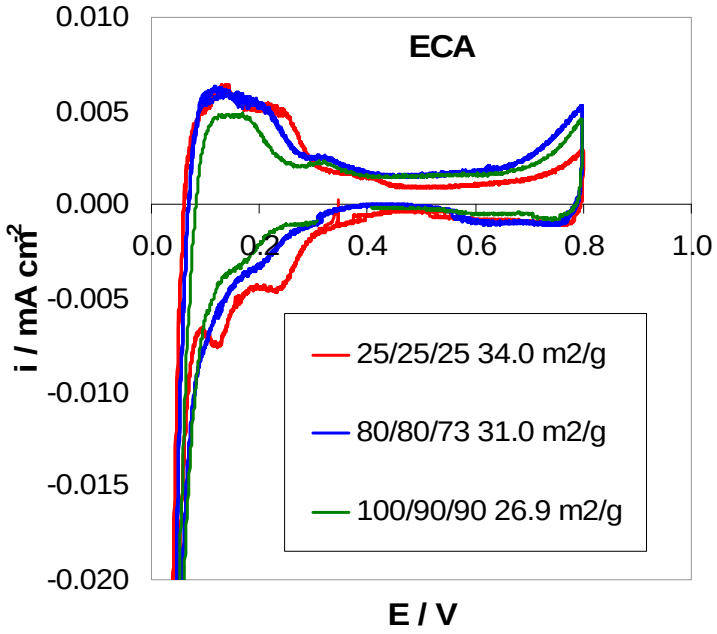
- Single cell hardware (25 $cm^2$) from Fuel Cells Technologies Incorporated (FCT)
  - Graphite bipolar plates with serpentine flow pattern, gold current collector plates, aluminum pressure plates
  - Anode bipolar plate flow field rotated 90° to give cross flow pattern and dimensions of serpentine flow fields modified to decrease pressure drop
- All MEAs use 10BB Gas diffusion layers (SGL Carbon, Sigracettm)
  - Through-plane permeability tested prior to use
- Cell seals made of Teflon® gaskets
  - thickness of gaskets thinner than cell assembly giving "pinch" of 0.009 to 0.010 inches
  - Pinch = $T_{GDL\ (Anode)} + T_{GDL\ (Cathode)} + T_{CCM} - T_{gaskets}$
- Then externally leak test, internally leak test, and electrically isolation test




1. End Plate
2. Gold coated copper plate
3. Bipolar plate
4. Teflon gasket
5. Gas diffusion layer
6. MEA
7. M6 Bolt

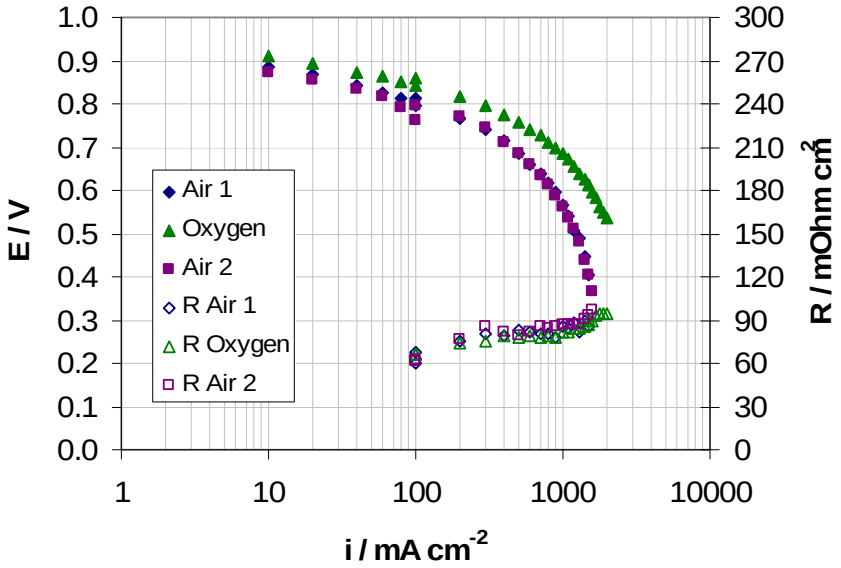
# Linear Sweep Voltammetry (LSV) and Cyclic Voltammetry (CV)




- Prior to performance evaluation, LSV and CV tests are performed
- Cathode is working electrode and anode is counter and reference electrode
- 0.4 liters/min of hydrogen (anode) and nitrogen (cathode)
- CV scan rate = 30 mV/s, 0 - 0.8 V
- LSV scan rate = 4 mV/s, 0 - 0.8 V
- LSV shows level of gas diffusion through membrane and electrical shorts
- CV provides a measure of ECA of the cathode

# Cell Conditioning (Day 1)

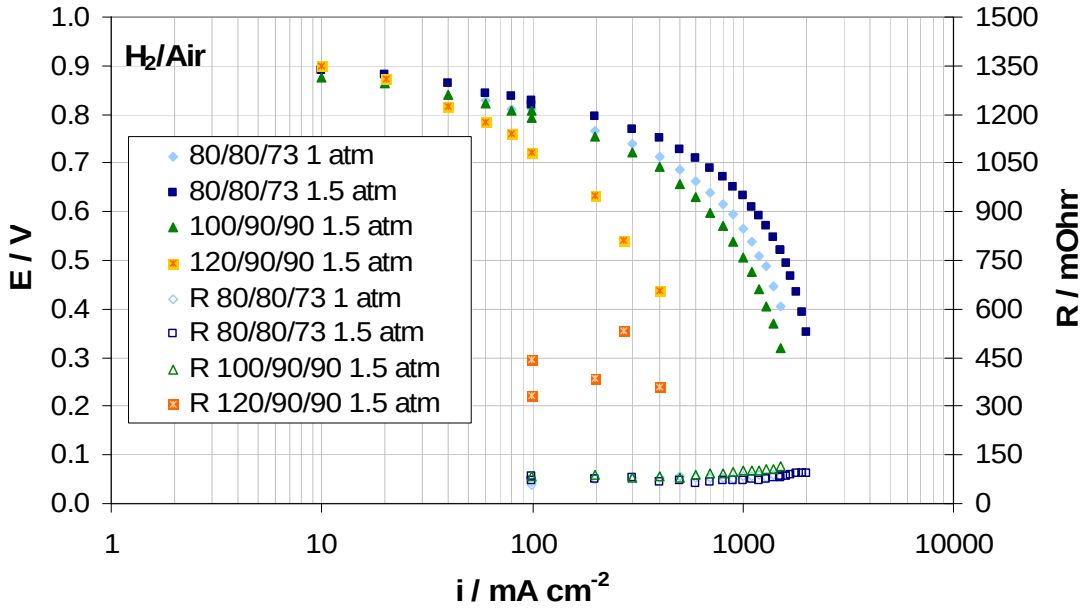



- After initial CO/ECA test, cell heated to 80 °C, anode to 80 °C, and cathode to 73 °C
  - Cell is conditioned for operation by adding water to the electrolyte
    - as a vapor from saturated gases ($H_2$ on anode and $N_2$ on cathode, 3 h)
    - by operation of the cell at 0.55 V (3 h)
- After "wet-up", performance curves run at ambient pressure and high stoichiometry, at near 100% RH and 80 °C.
- Performance sweeps are taken on $H_2$/Air, $H_2$/$O_2$, and $H_2$/Air

# Performance Verification

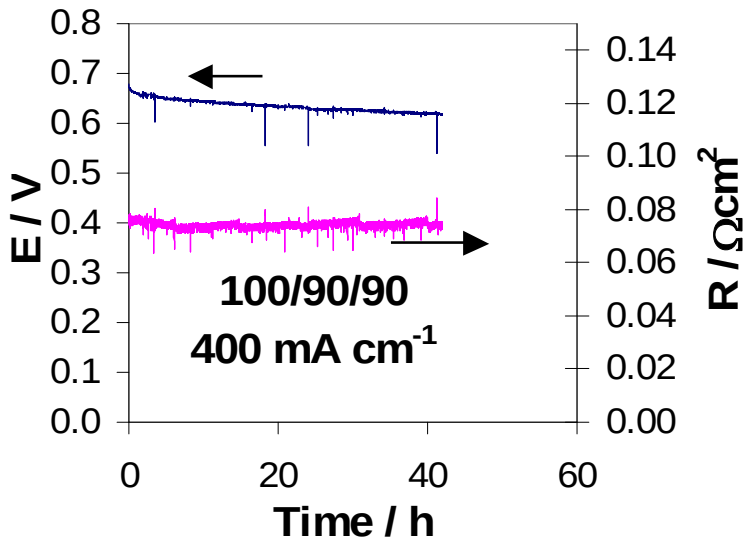
- Provide data on cells operating at conditions that are at the DOE goals or that represent progress toward those goals and show the relative merits of each of the candidate membranes at those conditions
- Started without shutting down.
- Cell pressurized to 1.5 atmospheres and run $H_2$/Air, $H_2/O_2$, $H_2$/Air performance sweeps
  - Stoichiometry above 3 to minimize the effects of reactant utilization

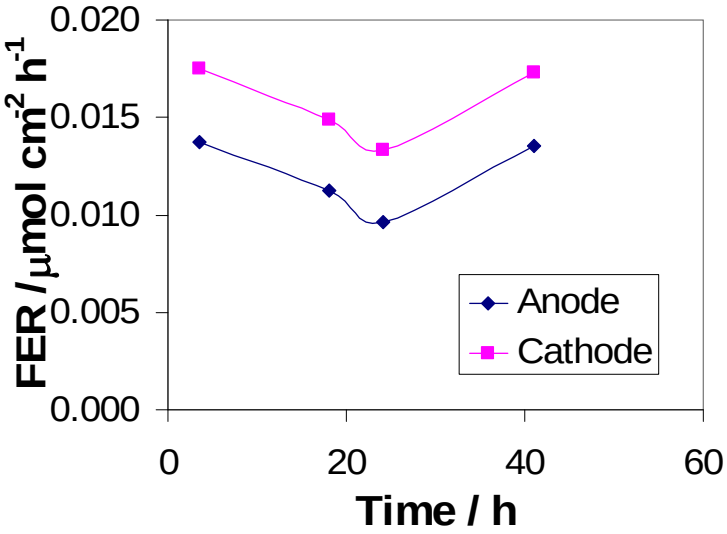
# Polarization Curve




- Typical performance for load calibration sweep, with 3 characteristic regions:
  - low current density region dominated by activation overpotential losses
  - mid current density region dominated by resistance losses
  - high current density region dominated by mass transport losses
- Analysis of curves and changes to the various regions of curves provides insight into the performance and stability of the membrane, catalyst, catalyst interface
- Fuel Utilization and Air Utilization impact the shape of these curves.
  - utilizations used are higher than goal, but data is valuable in comparing performance with lower fuel and air flows

# Stability Test



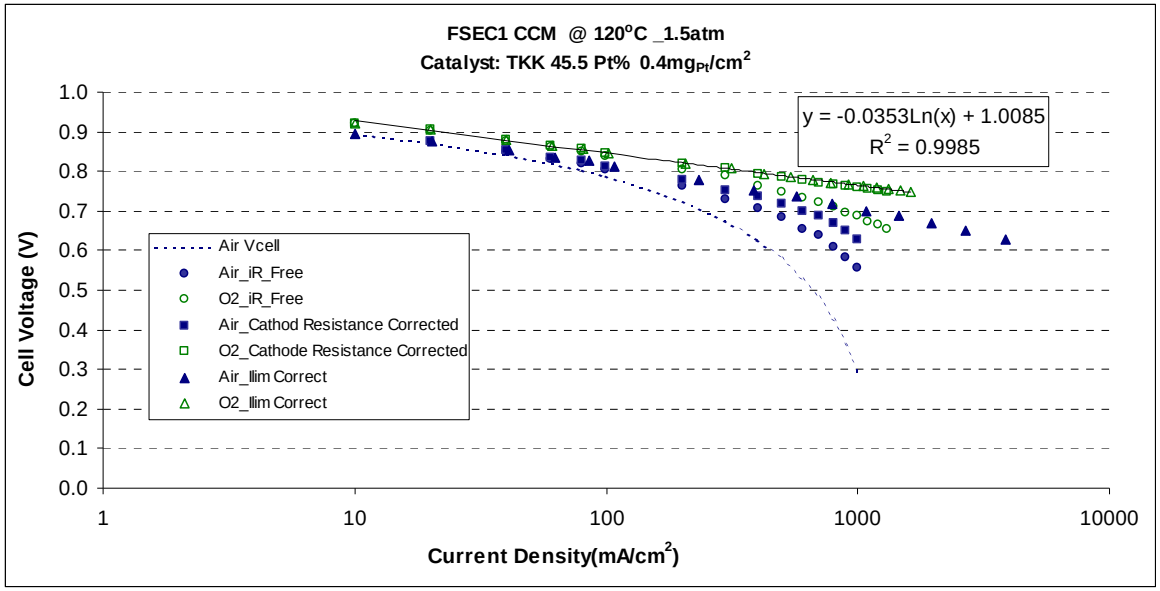




- ~60-hour endurance test
- Cell voltage and resistance are measured
- Initial and final ECA, $H_2$ CO, and performance are determined at cell operating temperature
- Water from the exit streams is condensed, collected, and analyzed for fluoride ion concentration
- Any change in performance or resistance is a result of MEA degradation due to chemical and mechanical stress

# Post-Test Integrity Test

- Before cell is removed from test stand
  - external leak test of cell and stand
  - resistance tests of cell and stand
- Bolt torque is measured
- To determine the changes in the cell integrity as a result of the protocol testing
- 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

# Cell Analysis




- FSEC has developed and verified a process to evaluate sources of polarization in $H_2$/air PEMFCs and uses it to analyze cell performance and guide development efforts.
  - Six sources of polarization
    - Non-electrode ohmic overpotential
    - Eectrode ohmic overpotential
    - Non-electrode concentration overpotential
    - Activation overpotential from catalyst activity
    - Activation overpotential from the Tafel slope
    - Electrode concentration overpotential
- Useful for diagnosing main sources of loss in MEA especially where several sources of loss are present simultaneously

# Day by Day PEM MEA Test Sequence – Days 1 and 2

- Day 1
  - Assemble the cell
  - Measure internal and external leakage
    - < 1 mA eq and 20 mA eq, respectively
  - Measure cell resistance
    - Anode to cathode > 30 Ohm
  - Room temp. CO/ECA
  - Wet up
    - 80/80/73
    - $H_2/N_2$, 3 h, no load
    - $H_2$/Air, ~3 h, 0.55 V
  - Cool down

- Day 2
  - Heat to 80/80/73
  - Measure and record OCV
  - Run V/I curve
    - Stoichiometry: Anode 3, Cathode 3.6
    - $H_2$/Air, $H_2/O_2$, $H_2$/Air
  - CO/ECA at 80/80/73
  - Hold overnight at 400 mA/cm$^2$

# Day by Day PEM MEA Test Sequence – Days 3 and 4

- Day 3
  - 80/80/73
  - Pressurize to 1.5 atm
  - Measure and record OCV
  - Run V/I curve
    - Stoich.: Anode 3, Cathode 3.6
    - $H_2$/Air, $H_2$/$O_2$, $H_2$/Air
  - Heat to 100/90/90
  - Release pressure and measure CO/ECA
  - Pressurize to 1.5 atm
  - Measure and record OCV
  - Run V/I curve
    - Stoich.: Anode 3, Cathode 3.6
    - $H_2$/Air
  - Hold overnight at 400 mA/cm$^2$

- Day 4
  - Run V/I curve at 100/90/90
    - Stoich.: Anode 3, Cathode 3.6
    - $H_2$/$O_2$, $H_2$/Air
  - Heat to 120/90/90
  - Release pressure and measure CO/ECA
  - Pressurize to 1.5 atm
  - Measure and record OCV
  - Run V/I curve
    - Stoich.: Anode 3, Cathode 3.6
    - $H_2$/Air, $H_2$/$O_2$, $H_2$/Air
  - Cool to 100/90/90
  - Hold overnight at 400 mA/cm$^2$

# Day by Day PEM MEA Test Sequence – Days 5-8

- Days 5, 6, 7
  - Release pressure and measure CO/ECA at 100/90/90
  - Measure and record OCV
  - Run V/I curve
    - Stoich.: Anode 3, Cathode 3.6
    - $H_2$/Air, $H_2$/$O_2$, $H_2$/Air
  - Start stability test
    - 1.5 atm, $H_2$/Air
    - 400 mA/cm$^2$
    - 100/90/90
    - Stoich.: Anode 3, Cathode 3.6
    - Monitor voltage and resistance
    - [$F^-$] of $H_2O$ from anode/cathode

- Day 8
  - Release pressure and measure CO/ECA at 100/90/90
  - Measure and record OCV
  - Run V/I curve
    - Stoich.: Anode 3, Cathode 3.6
    - $H_2$/Air, $H_2$/$O_2$, $H_2$/Air
  - Cool down to room temperature
    - $H_2$/$N_2$

# Day by Day PEM MEA Test Sequence – Day 9

- Measure CO/ECA at 25/25/25
- Measure cell resistance
- Measure internal and external leakage
- Measure bolt load
- Disassemble cell
- Visual Inspection
- Bag and seal components

- Data analysis
  - Δ CO
  - Δ ECA
  - Δ Activation
  - Δ Tafel
  - Δ Diffusion
  - Δ $i_{lim}$
  - Δ Conductivity
  - FER

# Cell Data Report-Day 1-Cell Build

- Membrane Description
  - Membrane BPP-1-115
  - Thickness = 25µm ± 1µm
  - Composition: 1100 EW PSFA/Tetratek/15 wt% PTA
  - Appearance: clear and colorless
- Catalyst Loading= 0.39 mg/cm$^2$
- Cell Description

GDL Thickness

| | | |
|---|---|---|
| 15.25 | 15.65 | 15.75 |
| 15.25 | 15.55 | 14.9 |
| 15.4 | 15.4 | 14.95 |

Cathode
GN=0.055 L/min/cm $H_2O/cm^2$

| | | |
|---|---|---|
| 15.65 | 16.25 | 15.10 |
| 15.4 | 15.05 | 14.9 |
| 15.3 | 15 | 14.95 |

Anode
GN=0.055 L/min/cm $H_2O/cm^2$

CCM Thickness

| | | |
|---|---|---|
| 2.45 | 2.5 | 2.65 |
| 2.65 | 2.85 | 2.8 |
| 2.45 | 2.65 | 2.6 |

Pinch

| | | |
|---|---|---|
| 9 | 10.05 | 9.15 |
| 8.95 | 9. 1 | 8.25 |
| 8.8 | 8.7 | 8.15 |

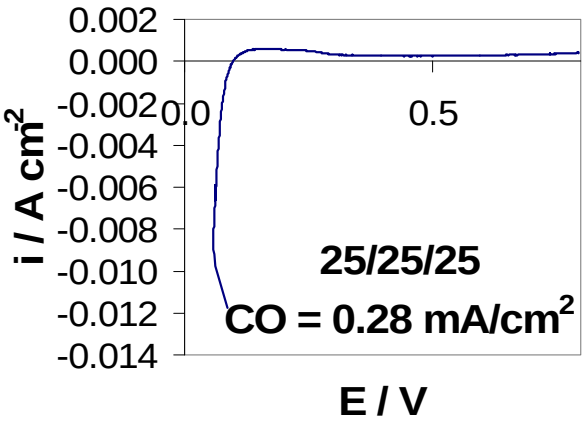
All numbers are in mil.

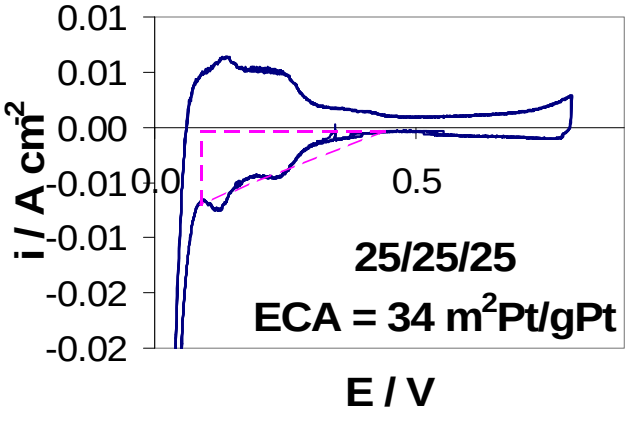
The gaskets were 11.65 (Cathode) and 12.7 (Anode) mil

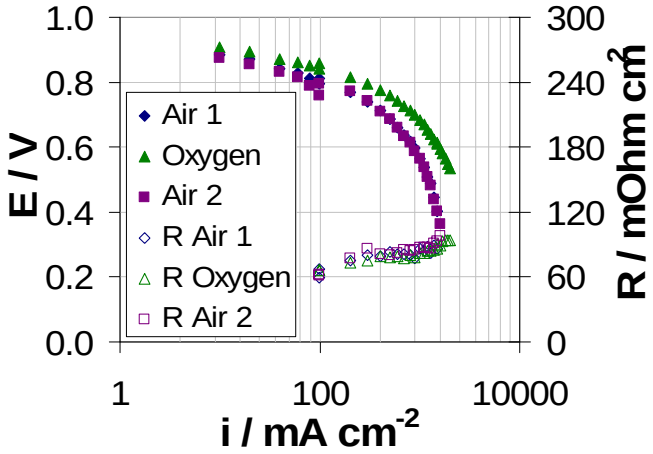
# Cell Report-Day 1- Cell Build Verification

- Cell build verification test data
  - Electronic resistance for short determination- 60 Ohm
  - External leakage rate – 0 mL/min
  - Internal leakage rate – 0 mL/min
- Current at 0.55 V (Break-in)
  - 0 h = 19.86 A
  - 1 h = 22.44 A
  - 2 h = 24.96 A
  - 3 h = 25.23 A

# Cell Report - Data From Start-Up – Day 2



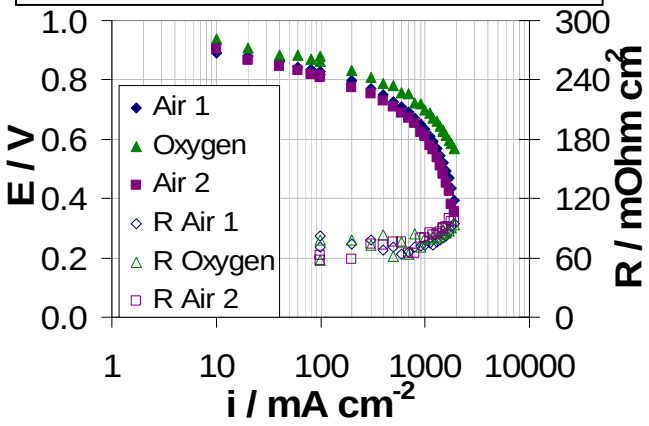



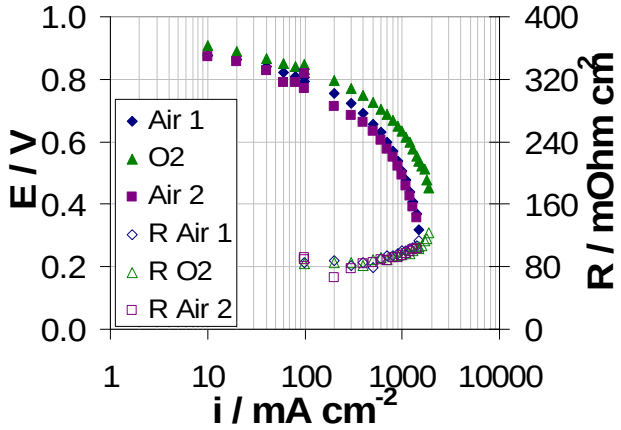

- Cell characterization at start-up
  - Cyclic voltammetry at 25 °C
  - Linear sweep voltammetry at 25 °C
- Performance test data at 80/80/73, 1 atm

# Cell Data Report-Performance Test Data

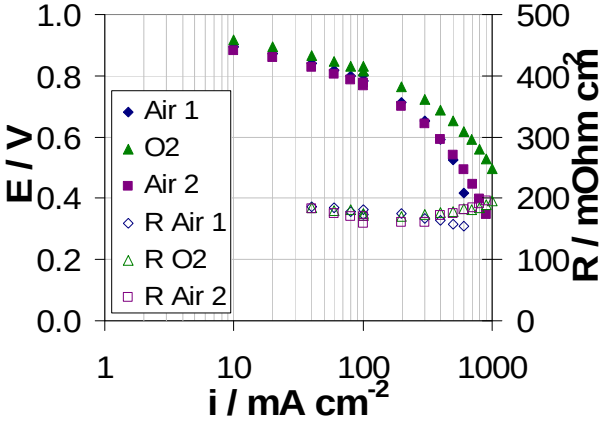
80 °C, 100% RH, 1.5 atm




100 °C, 69% RH, 1.5 atm




120 °C, 35% RH, 1.5 atm




- Cell voltage and cell resistance (OCV to 0.3V) at the following conditions:
  - $H_2/O_2$ and $H_2$/Air reactants at 150 kPa, 80°C/80°C/73°C (100% RH)
  - $H_2/O_2$ and $H_2$/Air reactants at 150 kPa, 100°C/90°C/90°C (69% RH)
  - $H_2/O_2$ and $H_2$/Air reactants at 150 kPa, 120°C/90°C/90°C (35% RH)

# Cell Data Report-Tables for Rapid Membrane Comparisons

– Current density and resistance using $H_2$/Air at 0.7 V

| Condition ($T_{cell}/T_{anode}/T_{cathode}$, P) | i, Air ($mA/cm^2$) | i, $O_2$ ($mA/cm^2$) | R, Air (Ohm $cm^2$) | R, $O_2$ (Ohm $cm^2$) |
|---|---|---|---|---|
| 80/80/73, 1 atm | 450 | 1000 | 0.061 | 0.057 |
| 80/80/73, 1.5 atm | 650 | 1300 | 0.065 | 0.060 |
| 100/90/90, 1.5 atm | 500 | 800 | 0.076 | 0.081 |
| 120/90/90, 1.5 atm | 220 | 360 | 0.17 | 0.18 |

– Polarization losses at 100°C, 69% RH, 1.5 atm, 400 $mA/cm^2$ using $H_2$/air reactants. The theoretical open circuit voltage (OCV) in this case is 1.167V.

– 3rd column shows cell performance on $H_2$/air at 400 $mA/cm^2$ as performance is reduced by each one of the polarization losses stepwise from the OCV.

| Source of Polarization | Polarization Loss (V) | Performance with Cumulative Loss (V) |
|---|---|---|
| Cathode Activation | 0.40 | 0.77 |
| Internal Cathode Diffusion | 0.02 | 0.75 |
| External Cathode Diffusion | 0.00 | 0.75 |
| Internal Cathode Resistance | 0.016 | 0.73 |
| Cell Resistance from Current Interrupt | 0.03 | 0.70 |

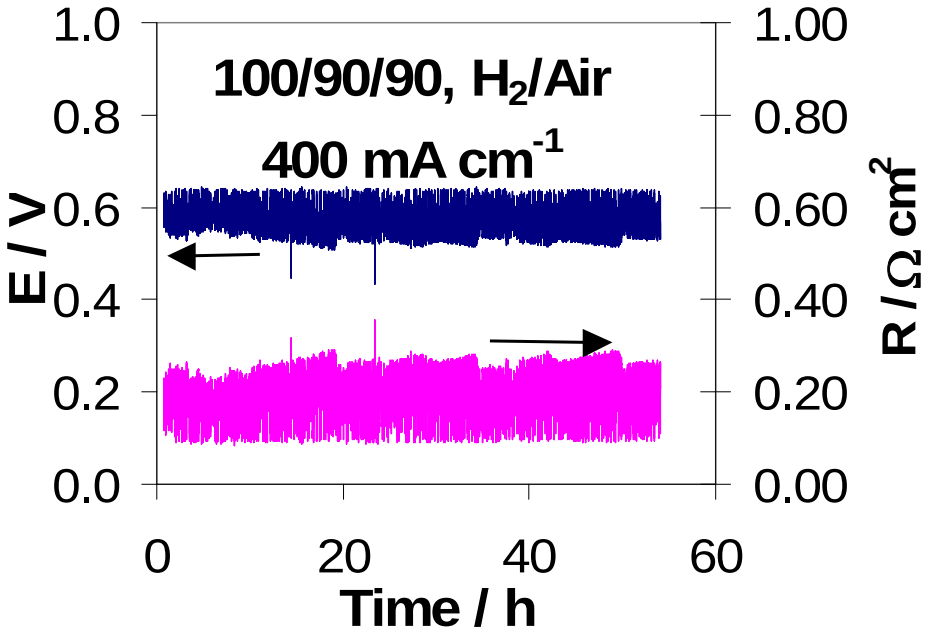
# Cell Data Report-Tables for Rapid Membrane Comparisons

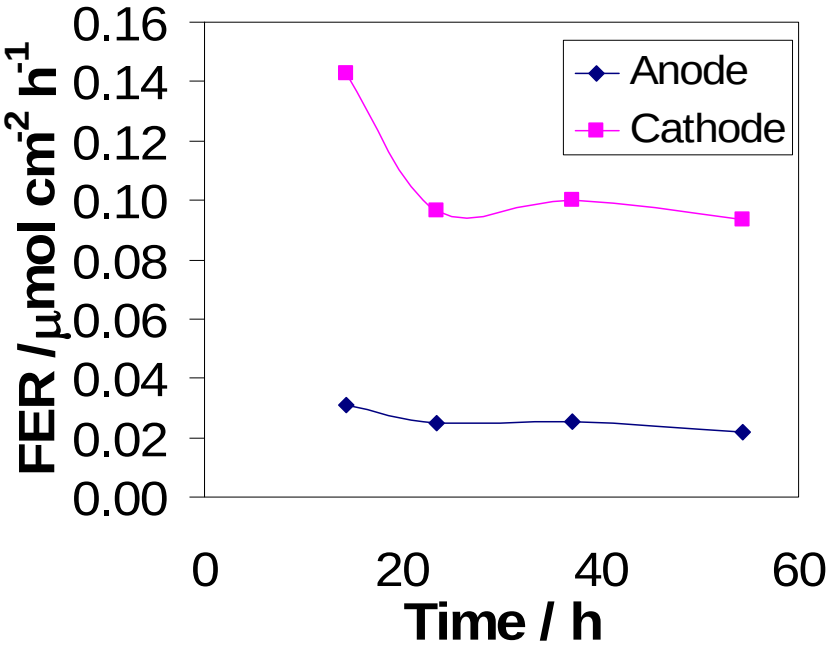
| | | Oxygen | Air |
|---|---|---|---|
| R-nonelectrode | ohm-cm$^2$ | 0.08 | |
| Current ratio | At 400mA/cm$^2$ | | |
| Relectrode | ohm-cm$^2$ | 0.04 | |
| Non-electrode concentration | $i_{Lim}$ (mA/cm$^2$) in O2/Air | 10000 | 2100 |
| Activation Tafel | Kinetic Tafel slope B | 73 | |
| Concentration electrode | b'(mV/dec) | 185 | |
| Catalyst activity | mA/mg Pt at 0.3V QOverpotential | 164 | |

- Diagnostic parameters using the approach of M. Williams, et al.*

*M.V. Williams, H.R., Kunz, and J.M. Fenton, Jl. Electrochem. Soc., **152**, A635-A644 (2005)

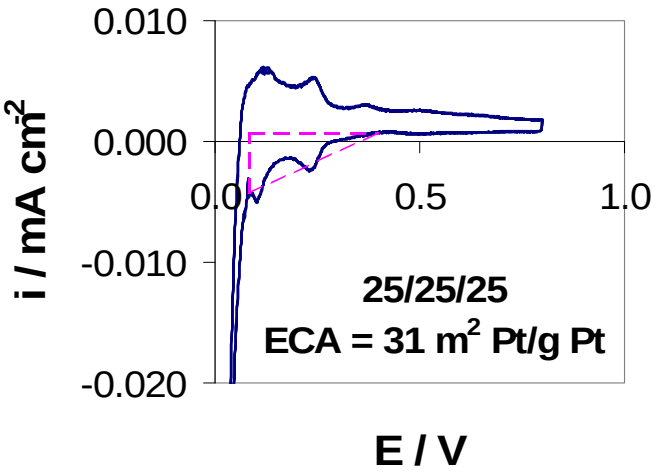
# Cell Data Report-Endurance Data

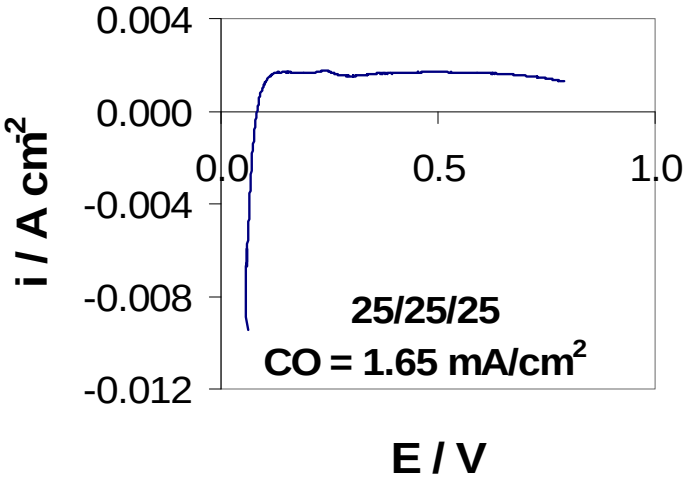


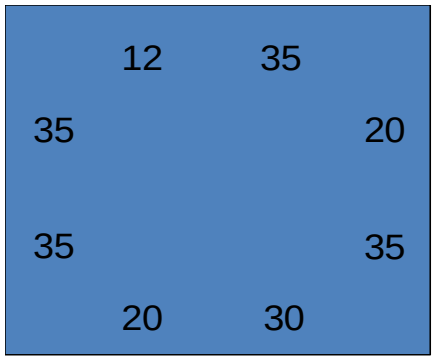




- Endurance test data
  - E and R for ~60 hours with $H_2$/Air at 400 mA/cm$^2$, 1.5 atm, 100 °C, 69% RH
  - Fluoride present in exhaust $H_2$ and air

# Cell Data Report – Post-Test Evaluation of Cell




- Cyclic Voltammetry at 25/25/25
- LSV at 25/25/25
- Electronic resistance for short determination- 65 Ohm
- External leakage rate – 0 mL/min
- Internal leakage rate – 0 mL/min
- Bolt Load (in lb)

# Summary

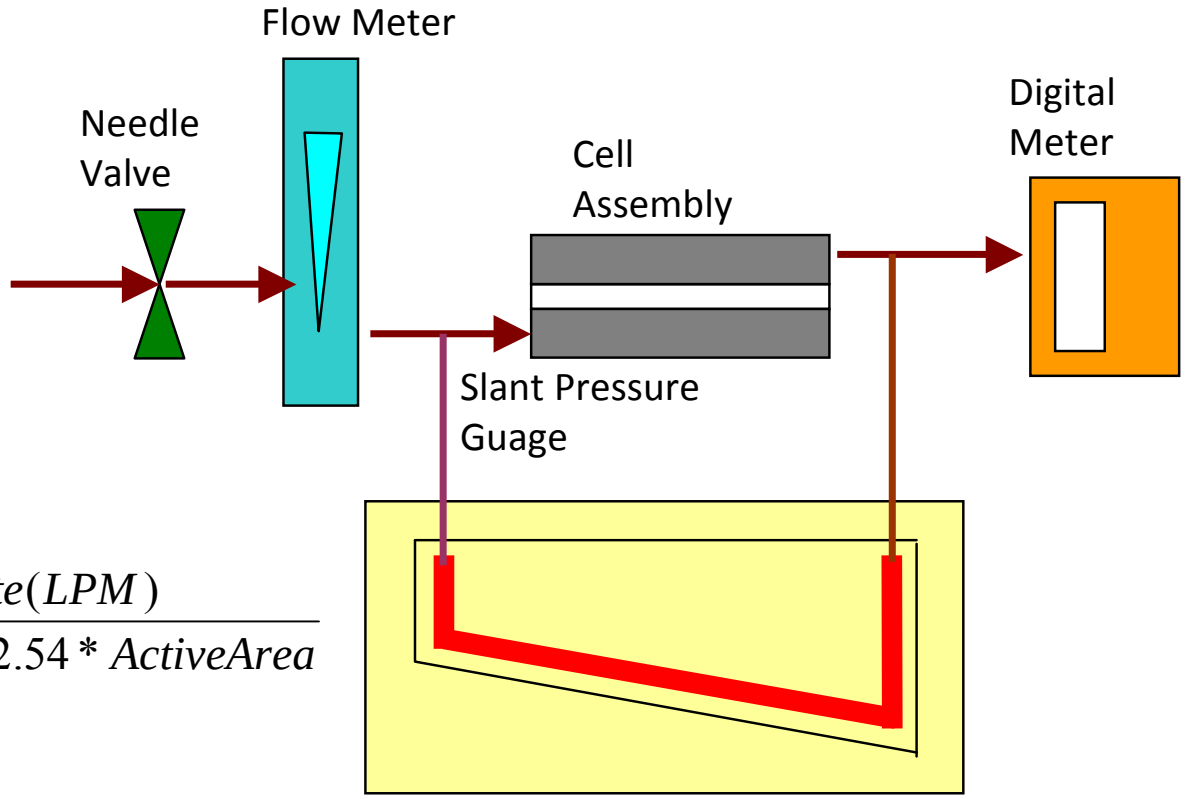
- 9-day MEA test protocol has been developed
- Includes performance tests at 80 $^{o}$C/100% RH, 100 $^{o}$C/69% RH, and 120 $^{o}$C/35% RH at 1.5 atm
- Because membranes tested to same protocol, results are a fair and accurate evaluation of performance capabilities


# Acknowledgement

**U.S. Department of Energy**

**Contract # DE-FC36-06GO16028**

# Gurley Number Test




$$GurleyNumber = \frac{FlowRate(LPM)}{\Pr essureDrop * 2.54 * ActiveArea}$$

- Gurley Number (GN) measures gas permeation rate through the gas diffusion backing samples
- GN represents gas flow rate (in LPM) for a Gas Diffusion Layer at fixed pressure difference (in cm of $H_2O$) through a fixed area of sample ($cm^2$)