

**USCAR FUEL CELL TECH TEAM**  
**CELL COMPONENT ACCELERATED STRESS TEST PROTOCOLS**  
**FOR PEM FUEL CELLS**

(Electrocatalysts, Supports, Membranes, and Membrane Electrode Assemblies)

**Revised May 26, 2010**

Fuel cells, especially for automotive propulsion, must operate over a wide range of operating and cyclic conditions. The desired operating range encompasses temperatures from below the freezing point to well above the boiling point of water, humidity from ambient to saturated, and half-cell potentials from 0 to >1.5 volts. Furthermore, the anode side of the cell may be exposed to hydrogen and air during different parts of the driving and startup/shutdown cycles.

The severity in operating conditions is greatly exacerbated by the transient and cyclic nature of the operating conditions. The cell/stack conditions cycle, sometimes quite rapidly, between high and low voltages, temperatures, humidities, and gas compositions. The cycling results in physical and chemical changes, sometimes with catastrophic results.

This document describes test protocols to assess the performance and durability of fuel cell components intended for automotive propulsion applications. The goal of this testing is to gain a measure of component durability and performance of electrocatalysts and supports, membranes, and membrane electrode assemblies (MEAs) for comparison against 2010 DOE targets contained in **Reference 1**. The resulting data may also help to model the performance of the fuel cell under variable load conditions and the effects of ageing on performance.

These protocols are intended to establish a common approach for determining and projecting the durability of polymer electrolyte membrane (PEM) fuel cell components under simulated automotive drive cycle conditions.

This document is not intended to be comprehensive as there are many issues critical to a vehicular fuel cell (e.g., freeze/thaw cycles) that are not addressed at this time. Additional issues will be addressed in the future. Furthermore, it is recognized that the cycles specified herein have not been fully correlated with data from stacks and systems operated under actual drive cycles. Therefore, additional tests to correlate these results to real world lifetimes is needed, including actual driving, start/stop, and freeze/thaw cycles.

The durability of catalysts can be compromised by platinum (Pt) particle growth and dissolution, especially at high electrode potentials; this sintering/dissolution is accelerated under load-cycling. Durability of catalyst supports is another technical barrier for stationary and transportation applications of PEM fuel cells. Corrosion of high-surface area carbon supports poses significant concerns at high electrode potentials and is accelerated during start/stop cycles and during higher temperature operation (>100°C).

Membranes are another critical component of the fuel cell stack and must be durable and tolerate a wide range of operating conditions including low humidity (20 to 100% RH) and high temperature (-40 to 120°C for transportation applications and >120°C for stationary applications). The low operating temperature and the humidity requirements of current membranes add complexity to the fuel cell system that impacts the system cost and durability. Improved membranes are needed that perform better and are less expensive than the current generation of polymer membranes.

The associated testing protocols and performance metrics are defined in Table 1 for electrocatalysts, Table 2 for catalyst supports, Table 3 for membrane/MEA chemical stability, and Table 4 for membrane/MEA mechanical durability, respectively, as derived from References 2, 3, and 4.

The specific conditions and cycles are intended to isolate effects and failure modes and are based on assumed, but widely accepted, mechanisms. For example, the electrocatalyst cycle is different from the support cycle because they suffer from different degradation mechanisms under different conditions. Similarly, membrane/MEA chemical degradation is distinguished from mechanical degradation.

Durability screening at conditions and under cycles different from those presented here-in are acceptable provided that the developer can provide:

- conclusive/convincing evidence that the cycle/conditions do not compromise separation/isolation of degradation mechanisms
- degradation rates extrapolated to the conditions/cycles prescribed here-in.

Data to be reported, if applicable, at each point on the polarization curves and during steady-state and variable load operation include, but are not limited to:

- |  |                                    |
|--|------------------------------------|
| ➤ Ambient temperature and pressure                           | ➤ Fuel inlet dew point             |
| ➤ Cell voltage   | ➤ Air inlet and outlet temperature |
| ➤ Cell current and current density                           | ➤ Air flow rate                    |
| ➤ Cell temperature   | ➤ Air inlet and outlet pressure    |
| ➤ Cell resistance, if available (along with test conditions) | ➤ Air inlet dew point              |
| ➤ Fuel inlet and outlet temperature                          | ➤ Fuel and air quality             |
| ➤ Fuel flow rate   | ➤ Coolant inlet temperature        |
| ➤ Fuel inlet and outlet pressure                             | ➤ Coolant outlet temperature       |
|  | ➤ Coolant flow rate                |

Pre-test and post-test characterization of cell and stack components should be performed according to developer's established protocols. At the discretion of the developer, tests should be terminated when hydrogen crossover exceeds safe levels.

## **References**

1. Hydrogen, Fuel Cells & Infrastructure Technologies Program Multi-Year Research, Development and Demonstration Plan, August 2006 (<http://www1.eere.energy.gov/hydrogenandfuelcells/mypp/>)
2. Appendix D of DOE Solicitation **DE-PS36-06GO96017**
3. Mathias, M., et al, "Two Fuel Cells in Every Garage?" Interface Vol. 14, No 3, Fall 2005.
4. Mathias, M., et al, "Can Available Membranes and Catalysts Meet Automotive PEFC Requirements?" Presentation at ACS Meeting, Philadelphia, August 2004.

**Table 1**  
**Electrocatalyst Cycle and Metrics**  
**Table revised March 2, 2010**

<b>Cycle</b>	Triangle sweep cycle: 50 mV/s between 0.6 V and 1.0 V. Single cell 25-50 cm <sup>2</sup>	
<b>Number</b>	30,000 cycles	
<b>Cycle time</b>	16 s	
<b>Temperature</b>	80°C	
<b>Relative Humidity</b>	Anode/Cathode 100/100%	
<b>Fuel/Oxidant</b>	Hydrogen/N <sub>2</sub> (H <sub>2</sub> at 200 sccm and N <sub>2</sub> at 75 sccm for a 50 cm <sup>2</sup> cell)	
<b>Pressure</b>	Atmospheric pressure	
<b>Metric</b>	<b>Frequency</b>	<b>Target</b>
<b>Catalytic Mass Activity*</b>	At Beginning and End of Test minimum	≤40% loss of initial catalytic activity
<b>Polarization curve from 0 to ≥1.5 A/cm<sup>2</sup>**</b>	After 0, 1k, 5k, 10k, and 30k cycles	≤30 mV loss at 0.8 A/cm <sup>2</sup>
<b>ECSA/Cyclic Voltammetry***</b>	After 10, 100, 1k, 3k, 10k, 20k and 30k cycles	≤40% loss of initial area

\* Mass activity in A/mg @ 150 kPa abs backpressure at 857 mV iR-corrected on 6% H<sub>2</sub> (bal N<sub>2</sub>)/O<sub>2</sub> {or equivalent thermodynamic potential}, 100%RH, 80°C normalized to initial mass of catalyst and measured before and after test.

\*\* Polarization curve per Fuel Cell Tech Team Polarization Protocol in Appendix 1

\*\*\* Sweep from 0.05 to 0.6V at 20mV/s, 80°C, 100% RH.

**Table 2**  
**Catalyst Support Cycle and Metrics**  
**Table revised May 26, 2010**

<b>Cycle</b>	Hold at 1.2 V for 24 h; run polarization curve and ECSA; repeat for total 400 h. Single cell 25-50 cm <sup>2</sup>	
<b>Total time</b>	Continuous operation for 400 h	
<b>Diagnostic frequency</b>	24 h	
<b>Temperature</b>	80°C	
<b>Relative Humidity</b>	Anode/Cathode 100/100%	
<b>Fuel/Oxidant</b>	Hydrogen/Nitrogen	
<b>Pressure</b>	150 kPa absolute	
<b>Metric</b>	<b>Frequency</b>	<b>Target</b>
<b>Catalytic Activity*</b>	Every 24 h	≤40% loss of initial catalytic activity
<b>Polarization curve from 0 to ≥1.5 A/cm<sup>2</sup>**</b>	Every 24 h	≤30 mV loss at 1.5 A/cm <sup>2</sup> or rated power
<b>ECSA/Cyclic Voltammetry***</b>	Every 24 h	≤40% loss of initial area

\* Mass activity in A/mg @ 150 kPa abs backpressure at 857 mV iR-corrected on 6% H<sub>2</sub> (bal N<sub>2</sub>)/O<sub>2</sub> {or equivalent thermodynamic potential}, 100%RH, 80°C normalized to initial mass of catalyst and measured before and after test.

\*\* Polarization curve per Fuel Cell Tech Team Polarization Protocol in Appendix 1

\*\*\* Sweep from 0.05 to 0.6V at 20mV/s, 80°C, 100% RH.

**Table 3**  
**MEA Chemical Stability and Metrics**

Table revised December 10, 2009

<b>Test Condition</b>	<b>Steady state OCV, single cell 25-50 cm<sup>2</sup></b>	
<b>Total time</b>	500 h	
<b>Temperature</b>	90°C	
<b>Relative Humidity</b>	Anode/Cathode 30/30%	
<b>Fuel/Oxidant</b>	Hydrogen/Air at stoics of 10/10 at 0.2 A/cm <sup>2</sup> equivalent flow	
<b>Pressure, inlet kPa abs (bara)</b>	Anode 150 (1.5), Cathode 150 (1.5)	
<b>Metric</b>	<b>Frequency</b>	<b>Target</b>
<b>F<sup>-</sup> release or equivalent for non-fluorine membranes</b>	At least every 24 h	No target – for monitoring
<b>Hydrogen Crossover (mA/cm<sup>2</sup>)*</b>	Every 24 h	≤2 mA/cm <sup>2</sup>
<b>OCV</b>	Continuous	≤20% loss in OCV
<b>High-frequency resistance</b>	Every 24 h at 0.2 A/cm <sup>2</sup>	No target – for monitoring
<b>Shorting resistance**</b>	Every 24 h	>1,000 ohm cm <sup>2</sup>

\* Crossover current per USFCC “Single Cell Test Protocol” Section A3-2, electrochemical hydrogen crossover method.

\*\* Measured at 0.5V applied potential, 80°C and 100% RH N<sub>2</sub>/N<sub>2</sub>. Compression to 20% strain on the GDL.

**Table 4**  
**Membrane Mechanical Cycle and Metrics**  
**(Test using a MEA)**  
**Table revised December 10, 2009**

<b>Cycle</b>	<b>Cycle 0% RH (2 min) to 90°C dewpoint (2 min), single cell 25-50 cm<sup>2</sup></b>	
<b>Total time</b>	Until crossover >2 mA/cm <sup>2</sup> or 20,000 cycles	
<b>Temperature</b>	80°C	
<b>Relative Humidity</b>	Cycle from 0% RH (2 min) to 90°C dewpoint (2 min)	
<b>Fuel/Oxidant</b>	Air/Air at 2 SLPM on both sides	
<b>Pressure</b>	Ambient or no back-pressure	
<b>Metric</b>	<b>Frequency</b>	<b>Target</b>
<b>Crossover*</b>	Every 24 h	≤2 mA/cm <sup>2</sup>
<b>Shorting resistance**</b>	Every 24 h	>1,000 ohm cm <sup>2</sup>

\* Crossover current per USFCC “Single Cell Test Protocol” Section A3-2, electrochemical hydrogen crossover method.

\*\* Measured at 0.5 V applied potential, 80°C and 100% RH N<sub>2</sub>/N<sub>2</sub>. Compression to 20% strain on the GDL.

# Appendix 1

## Fuel Cell Tech Team Polarization Protocol

Test Point #	Current Density [A/cm <sup>2</sup> ]	Anode Inlet H2% (balance N2) inlet/dry	Anode H2 Stoich [-]	Anode Dewpoint Temp [°C]	Anode Inlet Temp [°C]	Anode Pressure outlet [kPaabs]	Cathode Inlet O2% inlet/dry	Cathode Inlet N2% inlet/dry	Cathode O2 Stoich [-]	Cathode Dewpoint Temp [°C]	Cathode Inlet Temp [°C]	Cathode Pressure Outlet [kPaabs]	Cell/Stack control Temp [°C]	Test pt. Run Time min	Set Point Transit time s
Break-in															
B1	0.6	100%	1.5	59	80	150	21%	79%	1.8	56	80	150	80	20	0
Reduction															
R1	0	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	1 Until V> 0.1V	0
R2	0	100%	1.5	59	80	150	0%	100%	1.8	59	80	150	80		0
Polarization curve															
P1	0.2	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P2	0.4	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P3	0.6	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P4	0.8	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P5	1	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P6	1.2	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P7	1.4	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P7	1.6	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P8	1.8	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P9	2	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P10	1.8	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P11	1.6	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P12	1.4	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P13	1.2	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P14	1	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P15	0.8	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P16	0.6	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P17	0.4	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P18	0.2	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P19	0.1	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P20	0.05	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P21	0.02	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P22	0.05	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P23	0.1	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0
P24	0.2	100%	1.5	59	80	150	21%	79%	1.8	59	80	150	80	3	0

Stoichs for points below 0.2A/cm2 at 0.2A/cm2 equivalent flow