Appendix A: FCTT AST and Polarization Curve Protocols for PEMFCs

U.S. DRIVE Fuel Cell Tech Team
Cell Component Accelerated Stress Test and Polarization Curve Protocols for PEM Fuel Cells

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

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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 start-up/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 DOE and U.S. DRIVE targets**.** The resulting data may also help to model the performance of the fuel cell under variable load conditions and the effects of aging 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 fuel cell stacks and systems operated under actual drive cycles. Therefore, additional tests to correlate these results to real-world lifetimes are 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 humidity ranging from 20% to 100% relative humidity (RH) and temperatures ranging from -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 A-1 for electrocatalysts, Table A-2 for catalyst supports, Table A-3 for membrane/MEA chemical stability, and Table A-4 for membrane/MEA mechanical durability, respectively, as derived from References 1 and 2.

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 these two cycles 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 herein are acceptable if the developer can provide convincing evidence that the cycle/conditions does not compromise the separation/isolation of degradation mechanisms.

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, the following:

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

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

Table A-5 contains the polarization curve protocols referenced in Tables 1 and 2 of this document. Table A-6 contains the protocol for determining cell/stack durability corresponding to the 5,000-hour U.S. DRIVE Fuel Cell Tech Team durability target.

References

1. Mathias, Mark, et al., "Two Fuel Cells in Every Garage?" *Interface* 14.3 (Fall 2005): 24–35, http://electrochem.org/dl/interface/fal/fal05/IF8-05_Pg24-35.pdf.
2. Mathias, Mark, et al. "Can Available Membranes and Catalysts Meet Automotive PEMFC Requirements?" *Prepr. Pap.-Am Chem. Soc., Div. Fuel Chem.,* 49.2 (2004): 471-474, http://web.anl.gov/PCS/acsfuel/preprint%20archive/Files/49_2_Philadelphia_10-04_1010.pdf.

Table A 1. Electrocatalyst Cycle and Metrics

Table revised March 2, 2010

Cycle	Triangle sweep cycle: 50 mV/s between 0.6 V and 1.0 V. Single cell 25–50 cm^2
Number	30,000 cycles
Cycle time	16 seconds
Temperature	80ºC
Relative humidity	Anode/cathode 100/100%
Fuel/oxidant	Hydrogen/N$_2$ (H$_2$ at 200 sccm and N$_2$ at 75 sccm for a 50 cm^2 cell)
Pressure	Atmospheric pressure

Metric	Frequency	Target
Catalytic mass activity*	At beginning and end of test minimum	≤40% loss of initial catalytic activity
Polarization curve from 0 to ≥1.5 A/cm^2**	After 0, 1k, 5k, 10k, and 30k cycles	≤30 mV loss at 0.8 A/cm^2
ECSA/cyclic voltammetry***	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$_2$ (bal N$_2$)/O$_2$ {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 Table A-5.

*** Sweep from 0.05 to 0.60 V at 20 mV/s, 80ºC, and 100% RH.

Table A 2. Catalyst Support Cycle and Metrics

Table revised January 14, 2013

Cycle	Triangle sweep cycle: 500 mV/s between 1.0 V and 1.5 V; run polarization curve and ECSA; repeat for total 400 h. Single cell 25–50 cm^2
Number	5000 cycles
Cycle time	2 seconds
Temperature	80ºC
Relative humidity	Anode/cathode 100/100%
Fuel/oxidant	Hydrogen/nitrogen
Pressure	Atmospheric

Metric	Frequency	Target
Catalytic activity*	At beginning and end of test, minimum	≤40% loss of initial catalytic activity
Polarization curve from 0 to ≥1.5 A/cm^2**	After 0, 10, 100, 200, 500, 1k, 2k, and 5k cycles	≤30 mV loss at 1.5 A/cm^2 or rated power
ECSA/cyclic voltammetry***	After 0, 10, 100, 200, 500, 1k, 2k, and 5k cycles	≤40% loss of initial area

* Mass activity in A/mg @ 150 kPa abs, backpressure at 857 mV iR-corrected on 6% H_2 (bal N_2)/O_2 {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 Table A-5.

*** Sweep from 0.05 to 0.6 V at 20 mV/s, 80ºC, and 100% RH.

Table A 3. MEA Chemical Stability and Metrics

Table revised December 10, 2009

Test condition	Steady-state OCV, single cell 25–50 cm^2
Total time	500 hours
Temperature	90°C
Relative humidity	Anode/cathode 30/30%
Fuel/oxidant	Hydrogen/air at stoics of 10/10 at 0.2 A/cm^2 equivalent flow
Pressure, inlet kPa abs (bara)	Anode 150 (1.5), cathode 150 (1.5)

Metric	**Frequency**	**Target**
F$^-$ release or equivalent for non-fluorine membranes	At least every 24 hours	No target – for monitoring
Hydrogen crossover (mA/cm^2)*	Every 24 hours	$\leq$2 mA/cm^2
OCV	Continuous	$\leq$20% loss in OCV
High-frequency resistance	Every 24 hours at 0.2 A/cm^2	No target – for monitoring
Shorting resistance**	Every 24 hours	>1,000 ohm cm^2

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

** Measured at 0.5 V applied potential, 80ºC, and 100% RH N_2/N_2. Compression to 20% strain on the GDL.

Table A 4. Membrane Mechanical Cycle and Metrics
(Test using an MEA)

Table revised December 10, 2009

Cycle	Cycle 0% RH (2 min) to 90^{o}C dewpoint (2 min), single cell 25–50 cm^2
Total time	Until crossover >2 mA/cm^2 or 20,000 cycles
Temperature	80ºC
Relative humidity	Cycle from 0% RH (2 min) to 90^{o}C dewpoint (2 min)
Fuel/oxidant	Air/air at 2 SLPM on both sides
Pressure	Ambient or no back-pressure

Metric	Frequency	Target
Crossover*	Every 24 hours	$\leq$2 mA/cm^2
Shorting resistance**	Every 24 hours	>1,000 ohm cm^2

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

** Measured at 0.5 V applied potential, 80ºC, and 100% RH N_2/N_2. Compression to 20% strain on the GDL.

Table A-5. Fuel Cell Tech Team Polarization Protocol

Test Point #	Current Density [A/cm^2]	Anode Inlet H_2% (balance N_2) inlet/dry	Anode H_2 Stoich [ ]	Anode Dewpoint Temp [°C]	Anode Inlet Temp [°C]	Anode Pressure Outlet [kPaabs]	Cathode Inlet O_2% inlet/dry	Cathode Inlet N_2% inlet/dry	Cathode O_2 Stoich [ ]	Cathode Dewpoint Temp [°C]	Cathode Inlet Temp [°C]	Cathode Pressure Outlet [kPaabs]	Cell/Stack Control Temp [°C]	Temp 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	0
R2	0	100%	1.5	59	80	150	0%	100%	1.8	59	80	150	80	Until V>0.1V	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/cm^2 at 0.2A/cm^2 equivalent flow

Table A-6. Protocol for Determining Cell/Stack Durability

Test Point #	Current Density [A/cm^2]	Anode Inlet H_2% (balance N_2) inlet/dry	Anode H_2 Stoich [ ]	Anode Dew point Temp [C]	Anode Inlet Temp [C]	Anode Pressure outlet [kPaabs]	Cathode Inlet O_2% inlet/dry	Cathode Inlet N_2% inlet/dry	Cathode O_2 Stoich [ ]	Cathode Dew point Temp [C]	Cathode Inlet Temp [C]	Cathode Pressure Outlet [kPaabs]	Cell/ Stack control Temp [C]	Test pt. Run Time min	Set Point Transition time s	Worst Case Response Transition Time s
Wet w/load cycling																
RH1	0.02	80%	96	83°	85°	101.3	21%	79%	108	83°	85°	101.3	80	0.5	0	2
RH2	1.2	80%	1.6	83°	85°	101.3	21%	79%	1.8	83°	85°	101.3	80	0.5	0	2
RH3	0.02	80%	96	83°	85°	101.3	21%	79%	108	83°	85°	101.3	80	0.5	0	2
RH4	1.2	80%	1.6	83°	85°	101.3	21%	79%	1.8	83°	85°	101.3	80	0.5	0	2
RH5	0.02	80%	96	83°	85°	101.3	21%	79%	108	83°	85°	101.3	80	0.5	0	2
RH6	1.2	80%	1.6	83°	85°	101.3	21%	79%	1.8	83°	85°	101.3	80	0.5	0	2
RH7	0.02	80%	96	83°	85°	101.3	21%	79%	108	83°	85°	101.3	80	0.5	0	2
RH8	1.2	80%	1.6	83°	85°	101.3	21%	79%	1.8	83°	85°	101.3	80	0.5	0	2
RH9	0.02	80%	96	83°	85°	101.3	21%	79%	108	83°	85°	101.3	80	0.5	0	2
RH10	1.2	80%	1.6	83°	85°	101.3	21%	79%	1.8	83°	85°	101.3	80	0.5	0	2
Trans 1	0.6	80%	2	70°	80°	101.3	21%	79%	2	70°	80°	101.3	80	2	0	30 (dew point)
Dry w/load cycling																
RH11	0.1	80%	5	53°	80°	101.3	21%	79%	5	53°	80°	101.3	80	0.5	0	30 (dew point)
RH12	0.02	80%	25	53°	80°	101.3	21%	79%	25	53°	80°	101.3	80	0.5	0	2
RH13	0.1	80%	5	53°	80°	101.3	21%	79%	5	53°	80°	101.3	80	0.5	0	2
RH14	0.02	80%	25	53°	80°	101.3	21%	79%	25	53°	80°	101.3	80	0.5	0	2
RH15	0.1	80%	5	53°	80°	101.3	21%	79%	5	53°	80°	101.3	80	0.5	0	2
RH16	0.02	80%	25	53°	80°	101.3	21%	79%	25	53°	80°	101.3	80	0.5	0	2
RH17	0.1	80%	5	53°	80°	101.3	21%	79%	5	53°	80°	101.3	80	0.5	0	2
RH18	0.02	80%	25	53°	80°	101.3	21%	79%	25	53°	80°	101.3	80	0.5	0	2
RH19	0.1	80%	5	53°	80°	101.3	21%	79%	5	53°	80°	101.3	80	0.5	0	2
RH20	0.02	80%	25	53°	80°	101.3	21%	79%	25	53°	80°	101.3	80	5	0	2