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Release Date
July 13, 2006

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Table of Contents

1 Introduction & Scope

This procedure was developed by consensus of members of the US Fuel Cell Council Materials and Components Working Group, Single Cell Testing Task Force. The main intent of this procedure is to highlight key issues affecting fuel cell data quality and to present best small-scale test practices in a practical, specific form. It is envisioned that the procedure might serve as a template which can be adapted and applied in the context of data exchange or joint testing between material suppliers and fuel cell developers.

The procedure was developed in conjunction with a series of inter-lab round-robin tests managed by the Single Cell Testing Task Force. In each round of testing, the four sites tested five single cells to determine

adequacy of the procedure, repeatability of test cells, and reproducibility of data between test sites. In addition to providing an experimental basis for this test procedure, the round-robin results will be published separately as a representative sample of the state of single-cell testing in the industry.

The Task Force is not necessarily recommending the use of this specific hardware configuration as a standard for testing materials since many studies require specific cell configurations. However, the procedure and hardware may be useful in cases where adopting an existing, non-proprietary protocol would expedite inter-lab data qualification as a basis for other data exchange. As such, this procedure will also be effective in validating data being obtained to support testing efforts of the Materials and Components Working Group and the Transportation Working Group, Joint Hydrogen Quality Task Force.

The main body of this document is organized to present background information and important technical considerations for each area of testing, along with a representative test protocol. Appendix A provides cell material specifications, assembly and diagnostic methods, and specific test sequences used in the round-robin tests managed by the Single Cell Testing Task Force.

2 Document Revisions

° Draft 14Mar05. Added general safety considerations, basic data quality checks, and statistical considerations; added clarifying text to most sections; expanded section on break-in procedure; re-wrote introduction, polarization curve, shutdown, and data reporting sections.

° Draft 15Mar05. Edited general safety considerations for single cell testing; section 11, paragraph 3 was “cell voltage should”; appendix A, section 2, 2) was a), 3) was a) “The Teflon “picture frame””, 4) was a) “The active area gasket”, 7) b) was “coated side facing the membrane”; appendix A, section 6, changed first polarization curve to section 6.1, added section 6.2 (was removed from previous protocol).

° Draft 20May05. Edited number of cells per test site to five, rather than 4 in Intro and Scope section; Added oxygen as potential combustible gas in Section 3; Added Doc 04-007 as reference to section 5.1;

° Draft 16Jun05. Added reference to leak check procedure in round-robin procedure appendix; Added test schedule, along with test plan per site as Appendix B. Added component specifications as Appendix C; Added time at each point of polarization curve back from previous revision; Changed order of polarization curves in round robin procedure, Appendix A.

° Draft 10Aug05: Added “at least” to section 8.2 first sentence for minimum data collection requirements and changed to 2 data points per minute; Section 8.2, 9 – deleted the sentence to change the data acquisition rate to 2 points per minute and hold at OCV for 5 minutes - changed time period to hold at OCV from 5 minutes to 30 seconds and to capture at least 10 points; Section 7 – Changed warmup to occur with purge gas. Then enable reactant gas flows prior to initiating load sequence. Section 8 – Protocol – changed the sequence such that the purge gas is used first while warming up the cell. Then the reactant gases applied and load. Changed the last OCV step to 30 seconds from 5 minutes. Appendix A – Revised the Leak Procedure to make the pressure test mandatory and the Cyclic Voltammetry optional. Amended the cyclic voltammetry procedure based on comments from T. Reward. Section A5 – Removed the 0.94V setting during break-in. Appendix B – changed the Actual months to Month 1 through 12 on the testing schedule, removed the participants for confidentiality upon release.

° Draft 17Feb06: Updated section A5 such that break-in load sequence is conducted at 80C; Amended Section A6 to include instructions for cell condition to be held between success polarization curves, added note to run successive polarization curves until the cell is stable and then record the next 3 polarization curves as reportable data

° Draft 05Jun06: Added the revisions prior to Rev A back into the document; Deleted Section 7.2, which was a representative break-in; Amended Section 8.2 7) to include specific instructions on setting the back pressure (set once at high flow rate and controlled at the outlet); Added step 8) to Section 8.2.

° Draft 26Jun06: Edited the product code for the GDE in Section A1 to A-13 and added Reference Grade in front of the description

° Draft 13July06: Edited A2)5) to read 7.04 x 7.04 rather than 7.1 x 7.1 to exactly match the dimensions of the material provided.

3 General Safety Considerations

This procedure contains general recommendations for single-cell fuel cell testing. Operating a fuel cell involves combustible gases (hydrogen and oxygen (depending on the test requirements)) and reactants at high temperature and pressure. Operators must be trained and experienced in the operation of complex engineering test systems and specifically in safe procedures involving electrical equipment and combustible gases.

Fuel cell test personnel are responsible for obtaining and following all applicable safety codes and generally accepted engineering practices related to their fuel cell test system, facility, fuel cell reactants (with particular attention to compressed hydrogen gas), exhaust products, and high electrical voltage and current. In summary, safely operating a fuel cell test station requires appropriate technical training and experience as well as safe facilities and equipment, all of which are outside the scope of this document.

4 Required Test Equipment

4.1 Test Station

The test station should meet the standards established in Fuel Cell Test Station Requirements and Verification Procedure, USFCC Document Number 04-011.

4.2 Supply Gases and Water

Fuel cells are very sensitive to contaminants in the reactant streams. The following quality standards have been found to be adequate for fuel cell testing:

a) Hydrogen gases should be at least 99.999% purity.

b) Air should be dry, oil free, and filtered to remove particulates greater than 1 micron. Air purity shall be equal to or better than CGA-7.1 Grade J Air, which has oxygen concentration of 20.5 ± 2% O2, ≤ 0.5ppmv CO2, ≤ 1.0 ppmv CO, ≤ 0.2 ppmv nitrogen oxides, ≤ 0.1ppmv sulfur oxides, ≤ 0.5ppmv hydrocarbons, ≤1ppmv water vapor.

c) De-ionized water for humidification should have a minimum resistivity of 250 kOhm-cm_2.

5 Cell Materials and Assembly

5.1 Material Definition

Cell materials and assembly procedures will be driven by the specific investigation at hand, and the focus material (e.g., the membrane-electrode assembly) will often be the only aspect of the fuel cell to be evaluated. Parties to the testing should specify and document all materials in the fuel cell test article. This specification should include a full definition with serial and lot numbers or, in the case of proprietary information, a unique material identification code. Such specification should be made for at least the following cell components:

a) Membrane

b) Diffusion media

c) Catalyst

d) Seals

e) Flowfield

f) Endplate/hardware

Refer to USFCC Document Number 04-007, Protocol on Fuel Cell Component Testing, for further assistance with determining required data to capture for materials definition.

5.2 Cell Assembly

Cell assembly procedures have large impact on the repeatability of fuel cell data. Specific assembly procedures should be documented for at least the following assembly operations:

a) Membrane alignment, including identification of anode and cathode sides.

b) Diffusion media alignment, including identification of anode and cathode parts, as well as the sides to be placed facing the membrane and flowfield.

c) Seal placement.

d) Alignment fixtures or jigs to be used, if any.

e) Compression procedures and specifications, which may include diffusion media compression values, bolt tightening order, progressive tightening procedures, and final torque specifications.

6 Leak Testing

The fuel cell should be tested for crossover and overboard reactant gas leaks in accordance with Leak Check Procedure, Single Cell, USFCC Document Number 04-070. Parties to the testing should specify and document acceptable leak rate thresholds. Measured leak rates must be documented within the project test report. Appendix A specifies the leak check procedures used in the USFCC single-cell round-robin testing program.

7 Cell Break-In

7.1 Technical Considerations

Many different break-in protocols for new materials exist within the fuel cell industry, with variations in duration and load cycle as well as the cell conditions. The main consideration is to perform a repeatable break-in procedure which brings the cell materials to a stable level of performance for subsequent testing. Since the break-in procedure has a lasting effect on the fuel cell materials, it is also possible to permanently bias test results with inappropriate conditions early in the fuel cell life. Three main possibilities exist for the definition of break-in protocols within the arena of testing for which this document is intended:

a) Procedures could be specified by a material supplier in consideration of unique material properties.

b) Procedures could be specified by a fuel cell developer in order to test the candidate material under conditions consistent with other program testing.

c) Procedures could be jointly developed by the material supplier and customer.

Regardless of the approach, the break-in procedure must be reviewed and approved by both parties to the testing in order to avoid unfavorable conditions and to agree on the baseline for performance testing.

Break-in protocols are typically applied only to a newly assembled cell; however, reconditioning procedures may also be used upon restarting a cell before each round of testing. Such procedures involve a stabilization period at a specified load and cell conditions in order to bring the cell to a predictable state of hydration before testing is resumed.

8 Polarization Curve Protocol

8.1 Technical Considerations

Fuel cell testing may take many forms in the context of evaluating cell materials. Some examples of specific test protocols are: steady-state testing at singular, critical operating points; parametric sensitivity testing centered on key operating conditions; very low power stability; and high current density operation. The polarization curve is perhaps the most familiar test scenario, involving a sweep of electrical loads in order to generate a characteristic cell voltage vs. load curve for a specific set of operating conditions.

Some considerations for designing a successful test protocol include the following:

a) When increasing load, first set reactant flows to the higher flow rates. When decreasing load, drop the load first, then adjust reactant flows to the lower flow rates.

b) Choose a stabilization time at each test point that includes the equilibration time for the test stand plus the fuel cell under test. Typical fuel cell test stations will require 5 – 10 minutes to stabilize at new test points, depending on the type of controls and quality of process calibration. Changes in temperature and humidification typically take the longest to re-stabilize. However, even fairly quick changes in flow and pressure can trigger longer term deviations in temperature and dew point as a result of changed heat transfer coefficients and mass of water being vaporized for humidification. Consider that the fuel cell will require a period of time to equilibrate after the test conditions are stable. Voltage hysteresis will be evident with shorter test points; the cell voltage at any test point will be affected to some extent by previous test conditions since cell hydration changes rather slowly.

c) Choose a data acquisition rate which is fast enough to observe trends in test conditions and cell voltage.

8.2 Protocol

A representative polarization curve protocol is described below, using nominal 20 minute test points with at least 2 data points recorded per minute:

1) Set the test station warning and shutdown safety limits. These should include at least the following:

   a) Maximum cell temperature.

   b) Maximum anode-cathode differential pressure.

   c) Minimum cell voltage.

2) Set purge gas flows corresponding to the lowest load step in the polarization curve.

3) Wait 1 minute for gas flow equilibration.

4) Set cell temperature for polarization curve conditions.

5) Set the data acquisition rate to 1 data point per minute.

6) Warm up the cell using the cell heater. During warmup, maintain gas dewpoint and inlet temperatures so that $T_{dewpoint} < T_{gas\ inlet} < T_{cell}$ at all times in order to prevent water condensation in the cell (a nominal 5 C temperature differential may be used to prevent condensation).

7) Set the anode and cathode flow rate to the maximum flow rate setting reached in the polarization curve (highest load step). Increase the back pressure on the anode and cathode until the appropriate outlet pressure setting is achieved, ensuring that the differential between the anode and cathode is kept below 35 kPa. The back pressure should not be adjusted after the initial setting, noting that the outlet pressure could be lower than the set point during the initial steps of the polarization curve.

8) Reduce the anode and cathode flow rates back to the lowest load step in the polarization curve.

9) Set the load box to the current specified in the sequence.

10) Disengage the electrical load and record the initial true OCV (must be true open circuit). Report the average voltage for the 30 second period OCV period capturing at least 10 points. If high frequency resistance is available on the test station, record the value at a 1 kHz frequency.

11) Reset the load box to the lowest load step in the polarization curve and hold for the specified stabilization period. Reset the data acquisition rate to 2 data points per minute for the last 5 minutes of the test period. Report the average voltage for 5 minute period.

12) Increase flows to values corresponding to the next highest load step in the polarization curve.

13) Increase the load to the next highest load step in the polarization curve and hold for the specified stabilization period. Report the average voltage for 5 minute period.

14) Repeat steps 12) and 13) above for the remaining steps of the sequence. If the cell is unable to run at a given current without falling below the minimum specified cell voltage, terminate the polarization curve and move to the next test.

15) After finishing the test sequence, maintain gas flows at the last setting, disengage the electrical load, and hold for 30 seconds. Report the average voltage for the OCV period.

16) Re-equilibrate cell at a standard condition prior to changing to new polarization curve conditions.

Appendix A includes the specific polarization curve load steps used in the USFCC single-cell round-robin testing program.

9 Shutdown

9.1 Technical Considerations

Different methods of cell shutdown may be applied, depending on specific test objectives or material considerations. The general purpose of a shutdown procedure is to cool the cell to ambient temperature while returning the cell to a non-condensing level of hydration. Gas temperatures and humidification must be controlled during the cool-down period to avoid both condensation and extreme membrane drying (which would occur if pressure and gas relative humidity were quickly reduced to ambient conditions while the cell is at an elevated temperature). An additional benefit is to eliminate any vacuum which would be drawn if flows were simply stopped and the remaining reactants cooled down in a sealed cell; in this situation, reactants are also consumed to some degree due to gas crossover.

9.2 Protocol

A representative shutdown protocol is described below:

1) Disengage the electrical load.

2) Purge anode and cathode with humidified nitrogen at flows corresponding to the lowest polarization curve load step until the cell cools to ambient temperature. During cool-down, maintain gas dew point and inlet temperatures so that $T_{dew\ point} < T_{gas\ inlet} < T_{cell}$ at all times in order to prevent water condensation in the cell (a nominal 5 C temperature differential may be used to prevent condensation).

3) After the cell has reached ambient temperature, switch to dry nitrogen flows on the anode and cathode for 5-10 minutes (this step is optional, depending on the final level of membrane hydration desired).

4) Disconnect the cell and tightly cap the anode and cathode inlets and outlets.

10 Data Content & Format

As described in the sample procedure above, cell data should be logged at 2 data points per minute. The 5 minute time-averaged value should be reported as the voltage for each test point.

Data files should be named according to a convention agreed upon by the parties to the testing, in a manner that clearly identifies the data for archival purposes. A text header within the data file should include all relevant cell hardware descriptions as well as the test conditions under which the cell was run. These conditions include:

a) Cell temperature.

b) Anode and cathode stoichiometry.

c) Anode and cathode inlet relative humidities, referenced to cell temperature.

d) Anode and cathode inlet pressures.

The data file should also log at least the following quantities on each time line of the file, with one column per data quantity. Site and cell identifiers and date are included redundantly on each line to facilitate data sorting when files are combined.

a) Site identifier

b) Cell identifier

c) Date

d) Time

e) Sequence step number

f) Cell load

g) Cell voltage

h) Cell temperature

i) Anode dew point temperature

j) Cathode dew point temperature

k) Anode outlet pressure

l) Cathode outlet pressure

m) Anode hydrogen flow rate

n) Cathode air flow rate

Appendix A includes the specific data file definition and naming convention used in the USFCC single-cell round-robin testing program.

11 Basic Data Quality Checks

Reviewing and checking two aspects of recorded data can eliminate the most basic sources of poor data quality: a) Instrument accuracy, b) Stability of test conditions and operator set points, and b) stability of cell.

Test conditions should be examined at each test point to ensure that the feedback signals for inlet parameters correspond to the desired set points. Discrepancies here can reveal errors in scripting automated sequences, operator errors, or test stand operational problems.

Cell voltage during the averaging period should be analyzed for stability. An average voltage has little meaning if the cell voltage is varying to an excessive degree. Some typical fault conditions are described below:

a) Cell voltage rising or falling with an overall trend, indicating that the cell is not equilibrated at the

test conditions.

b) Cell voltage varying randomly to an excessive level, indicating possible cell flooding or perhaps water droplets from the humidifiers.

c) Cell voltage varying cyclically, indicating unstable control of flows, temperatures, or dew points. These control parameters may be then plotted against time to detect any faults which may be driving the cell voltage.

12 Statistical Considerations

Depending on cell configuration and test conditions, important part-to-part differences in cell voltage may be detected. It is generally not recommended to make conclusions about cell performance based on a sample size of 1 cell per configuration under test. A true replication would involve a completely separate cell build, installation on the test station, and completed test sequence.

Once data are in hand for replicated cell tests, statistical tests may be used as a guideline for detecting significant differences. The simplest approach to comparing two cell configurations is to construct confidence intervals for the mean value of each configuration of interest (e.g., cell design A, B, C; or test station A, B, C) and conclude that two configurations are significantly different only if their confidence intervals do not overlap. An equivalent test is to perform a 2-sample *t*-test.

The confidence levels for such tests may be chosen depending on the type of decision being made. A 95% confidence level is common for final material choices or other decisions which require a high level of evidence to support the conclusion of a true difference. On the other hand, an 80% confidence level may be appropriate for screening situations where follow-up is intended. Examples of this include initial screening of potential design factors, or for detecting evidence of site-to-site differences which should be checked in the interest of data quality. Using a lower confidence level will result in a higher chance of a "false-positive," i.e., concluding there is a difference when one does not truly exist. However, this is consistent with a screening scenario where there is more risk in missing an important (or a test station out of calibration) than there is by falsely concluding there is a difference.

Choosing a sample size is always a tradeoff between resource costs and gaining sufficient information for good decisions. Tables 1 and 2 give some rough guidelines for the statistical tests described above. In each table the minimum detectable voltage difference (in mV) is provided as a function of sample size and the standard deviation of voltage for individual cells tested. Table 1 is constructed for a 95% confidence level and Table 2 for an 80% level. Both tables assume a constant 95% "power" for the tests, meaning that there is approximately a 5% chance of missing a significant result when it truly exists.

As an example in Table 1, if it is important to detect a minimum of 15 mV difference in mean voltage with 95% confidence and the standard deviation is estimated at 5 mV from previous testing, a sample size of 4 is necessary for each configuration tested. The required sample size jumps to 12 if the standard deviation is 10 mV. It is not uncommon for engineers to conclude that there is insufficient time, material, or cash resources to allow replications beyond 3 to 5; in that case, it is still important to realize the extent to which the test program may fall short of the sample size required for full confidence, and make appropriately conservative decisions.

	Standard Deviation		
Sample Size	**5 mV**	**10 mV**	**15 mV**
3	20	40	60
4	15	31	46
5	13	26	39

8	10	19	29
10	9	17	26
12	8	15	23

Table 1: Minimum detectable voltage difference vs. sample size and standard deviation, with a 5% chance of a false positive (95% confidence) or false negative (95% power).

	Standard Deviation		
Sample Size	**5 mV**	**10 mV**	**15 mV**
3	14	27	41
4	11	22	34
5	10	20	29
8	8	15	23
10	7	13	20
12	6	12	18

Table 2: Minimum detectable voltage difference vs. sample size and standard deviation, with a 20% chance of a false positive (80% confidence) or false negative (95% power).

Appendix A: Round-Robin Test Specifications & Methods

A1) Cell Materials and Assembly

a) Cell Materials

1) Hardware:
   a) Manufacturer: Teledyne Energy Systems, Inc
   b) Part Number: MEDUSA CH-50
   c) Collector Plate Material: POCO Graphite
   d) Flow Field Configuration: Triple Serpentine, Coflow
   e) Machined Active Area: 46 cm2
2) Sealing Gasket:
   a) Vendor : Saint-Gobain Performance Plastics, 14 McCafferty St., Hoosick Falls, NY, Tel: 518-686-6280 or 800-962-2666
   b) Material: Silicone Coated Fabric, 10 mil
   c) Part Number: 1010
3) Active Area Gasket:
   a) Vendor: PLASTIC SUPPLY, INC @ 800-235-1533 or 505-884-0507
   b) Description: TEFLON FEP gasket, 1-mil thickness (Comes in 12” or 24” width).

4) Membrane-and-Electrode:

a) Membrane:

i) Manufacturer: DuPont

ii) Part Number: NE-1135

iii) Description: Nafion®1135, 3.5 mil thick membrane, dry, "as shipped" form, no pre-conditioning.

b) Polyester Sheet

i) 2-mil

c) Gas Diffusion Electrode:

i) Vendor: E-TEK Div. of De Nora N. A., Inc., 39 Veronica Ave, Somerset, NJ 08873-6800 USA, Phone: (732) 545-5100 Fax: (732) 545-5170, www.etek-inc.com

ii) Product Code: A-13, under custom products

iii) Description: Reference Grade ELAT/Std/SS/V2.1 Single Sided Hand-Fabricated ELAT Electrode; 20% Pt on Vulcan XC-72, 0.5mg/cm2 Pt loading; with 0.6-0.8mg/cm2 Nafion application over catalyst coating.

A2) Cell Assembly

Perform the following steps first using the polyester sheet as the membrane. This will help to compress the GDL and GDE and prevent the fibrous materials from developing holes in the membrane.

1) Cell Hardware Preparation

a) Clean the machined graphite flow fields with water or isopropanol.

b) Dry with compressed nitrogen or dry, filtered, oil free air.

2) Prepare Material

Cut gaskets, gas diffusion media and membrane incorporating locating holes in the membrane. The Sealing Gasket (used for cell sealing) and Gas Diffusion Media are cut oversized to the cell active

area. The required active area is defined by the Active Area Gasket (used to prevent holes in the membrane at the fuel inlet and outlet).

3) Active Area Gasket

The Teflon active area gasket has a ~46 cm2 opening (active area) and outside dimensions of 10 cm by 10 cm.

4) Sealing Gasket

The sealing gasket has an outside dimension of 10.2 cm by 10.2 cm, with center cutout dimensions of 7.1 cm by 7.1 cm to accommodate the GDE.

5) GDE

The GDE dimensions are 7.04 cm by 7.04 cm, matching the inside dimensions of the silicone sealing gasket.

6) Membrane

a) The membrane is cut to 10 cm by 10 cm.

b) The membrane does not require any heat lamination step before fuel cell assembly and testing.

7) Assemble Soft Goods into Cell Hardware

a) Center the Sealing Gasket on the flow field plate of the test cell.

b) Position the GDE within the Sealing Gasket's center opening, such that the Nafion® Polymer Solution coated-side is facing the membrane.

c) Position the Active Area Gasket such that the opening is centered on the GDE, bridging any gap between the GDE and Sealing Gasket.

d) Center the membrane on the assembly.

e) Place another Active Area Gasket, GDE, and Sealing Gasket on the assembly.

f) Place the matching flow field plate atop the assembly.

8) Cell Torque Requirements

a) Lubricate the test cell bolts with bolt lubrication prior to assembly.

b) Torque the test cell bolts in a diagonal pattern per Figure A1. Use incremental torque settings of 25 in-lb increments until the final recommended torque is reached. The recommended final torque amount for the test cell design described in this protocol is a maximum 150 in-lbs to achieve a compression of 150 to 200 psi in the active area of the cell. Gasket thickness (10+1 mil) per side helps to achieve a ~15-20% compression of the GDE on each side.

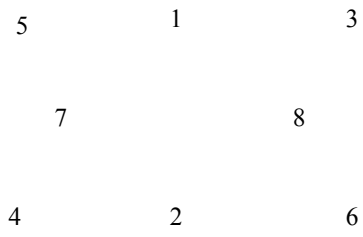

Figure A1: Cell Torque Diagram

A3) Leak Testing

Step one, Pressure Test Method is required. The Electrochemical Hydrogen Crossover Method, Step 2 can be performed in addition to Step 1 for comparison, but is not required.

1) Pressure Test Method: Refer to Leak Check Procedure, Single Cell, USFCC Document Number 04-070 using the pressure settings outlined below,

a) External leaks:

i) Check for sealing leaks using equal 25 psig N2 on the anode and cathode.

ii) With cell pressurized and gas inlet/exits blocked, a leak is determined when gas pressure drops 1 psi over 10 minutes.

b) Crossover leaks:

i) Check for crossover leaks using 3 psig N2 on the anode and cathode.

2) Electrochemical Hydrogen Crossover Method:

The testing procedure for hydrogen crossover is based on an electrochemical detection of the molecular hydrogen passing through the membrane. For that purpose, the assembled cell is purged with hydrogen at the anode side and with nitrogen at the cathode side. In this mode, the fuel cell anode serves as reference and counter electrode and the fuel cell cathode acts as the working electrode (three-electrode arrangement). Under these conditions, a voltammogram is recorded. The detected current resulting from the oxidation of molecular hydrogen at the fuel cell cathode is determined.

a) Recommended Equipment:

i) The following equipment is recommended. Equivalent may be used. Record manufacturer, model and settings for actual equipment used.

ii) Princeton Applied Research Potentiostat/Galvanostat, Model 273

b) Assembled Cell Operating Condition:

i) Cell temperature approximately 24°C (room temperature)

ii) Gases at 100% relative humidity

iii) Stoichiometry Hydrogen = 1.5 @ 1 A/cm

iv) (Alternative: 4% Hydrogen/Nitrogen, 500 cc/min)

v) Nitrogen = 30 nl/h

vi) Pressure = 1 bar at sea level (adjusted to 3.7 psig at LANL)

c) Testing Procedure for Probing the Cathode:

i) Purge the anode with hydrogen and the cathode with nitrogen for at least 30 minutes to equilibrate the cell.

ii) Set the range on the potentiostat to 0.1 to 0.4V and the scan rate to 2.0 mV/s.

iii) Run the scan and wait 10 minutes

iv) Repeat the Sweep Voltammetry.

v) The crossover current is determined from the steady state value at 300 mV. The recorded value is then translated into a hydrogen crossover in terms of ml/min-cm_2.

vi) Repeat the procedure twice to verify the result.

d) Potentiostat settings:

i) Current range:	2 Amps
ii) 1_{st} potential:	100 mV
iii) 2_{nd} potential:	400 mV
iv) Scan speed:	2 mV/s

e) Reference Values

i) The typical acceptable value for hydrogen crossover for Nafion 1135 membrane is 0.014 mL/min-cm_2, which is equivalent to 2 mA/cm_2; see Figure A2.

ii) A typical voltammogram for a one mil membrane is shown in Figure A3.

Figure A2: Typical Hydrogen Crossover for Nafion NF-1135

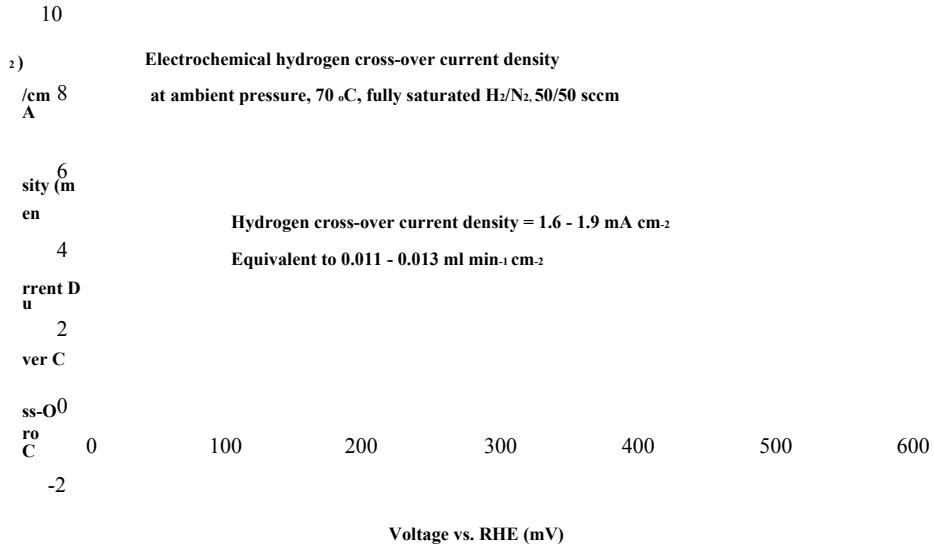

Figure A3: Typical Results for Electrochemical Crossover Data for 1 mil Membrane

A4) Cell Connections

1) Operate the test cell in vertical orientation.

2) The gas flow should be connected as coflow.

3) Connect load cables capable of handling at least 70 A.

A5) Cell Break-In and Re-Conditioning

a) Break-in Load Sequence

The cell break-in procedure is to be completed only by the location that assembles the cells. Table A1 defines the break-in load cycle.

Test Condition	Step Time (min)	Cumulative Time (hr)
Initial Start-up	As required to warm up to 80 C	
Cycling step 1 (Perform once):		
o 0.60 V	60	1.0
Cycling step 2 (Perform 9 times):		
o 0.70 V	20	
o 0.50 V	20	7.0
Constant current operation:		
o 10 amps	720	19.0

Table A1: Cell Break-in Load Sequence

Verify break-in status by repeating the polarization curve sequence in Table A2 three times, or as necessary, to ensure that the cell is broken-in. Remain at each sequence step for 20 minutes. The cell is considered broken in when less than 5 mV deviation from the previous polarization curve is recorded at 40 amps. A wait period of 10 minutes should be observed between polarization curves. During this period, return the gas flow rates to the equivalent of 10 stoich at 10 amps and set the current to 40 amps.

Fuel: Hydrogen, 1.2 Stoich, 100 % RH

Oxidant: Air, 2.0 Stoich, 100 % RH

Temperature (C): 80

Pressures (psig): 25

Sequence Step	Current Density (mA/cm2)	Current (A)	H_2 Flow Rate (slpm)	Air Flow Rate (slpm)
0	0	0	0.042	0.166
1	100	5	0.042	0.166
2	200	10	0.084	0.332
3	400	20	0.167	0.663
4	600	30	0.251	0.995
5	800	40	0.334	1.327
6	1000	50	0.418	1.658
7	1200	60	0.501	1.99

Table A2: Polarization Curve to Verify Break-in

b) Cell Re-Conditioning

Cell re-conditioning is to be completed at each test facility to ensure that the cell is properly humidified for the polarization curves.

1) Flow fully-humidified gases at a stoichiometry of 2 / 2 through the cell for one hour while heating to 80 C, with a current setting of 20 amps.

2) Run cell at these conditions for four hours; the cell is conditioned when the voltage is equilibrated.

A6) Polarization Curve Conditions and Load Sequence

a) General Procedure

i) Perform Polarization Curve 1 three times. Then perform Polarization Curve 2 three times.

ii) Table A3 specifies the polarization curve load sequence, to be followed in the order presented.

iii) Maintain conditions for each sequence step for 20 minutes.

iv) A wait period of 10 minutes should be observed between polarization curves. During this period, return the gas flow rates to the equivalent of 10 stoich at 10 amps and set the current to 40 amps.

v) Conduct subsequent polarization curves until repeatable results are observed (within 5mV deviation of the previous polarization curve at 40A). Then record the next three polarization curves as reportable data.

Sequence Step	Current Density (mA/cm2)	Current (A)	H_2 Flow Rate (slpm)	Air Flow Rate (slpm)
0	0	0	0.042	0.166
1	100	5	0.042	0.166
2	200	10	0.084	0.332
3	400	20	0.167	0.663
4	600	30	0.251	0.995
5	800	40	0.334	1.327
6	1000	50	0.418	1.658
7	1200	60	0.501	1.99

Table A3: Round-Robin Polarization Curve Sequence

b) Polarization Curve 1

i) Polarization Curve 1 test conditions are listed below.

Fuel: Hydrogen, 1.2 Stoich, 100 % RH

Oxidant: Air, 2.0 Stoich, 100 % RH

Temperature (C): 80

Pressures (psig): 25

c) Polarization Curve 2

Polarization test conditions are listed below.

Fuel: Hydrogen, 1.2 Stoich, 100 % RH

Oxidant: Air, 2.0 Stoich, 100 % RH

Temperature (C): 60

Pressures (psig): 0 at outlet (~3psig back-pressure at full flow)

A7) Data File Content & Naming

a) File Naming Convention

A common file format and naming convention would also be helpful: Data files should be saved in Excel format, named according to the following convention:

1) USFCC.SCRR*n*.Site*n*.Set*n*.Cell*n*.xls, where *n* refers to specific test identifiers. For example for the Single Cell Round Robin 1, Site 2, first test set of tests, Cell3, the filename would be "USFCC.SCRR1.Site2.Set1.Cell3.xls. "

2) The "Set" identifier is necessary since a site sometimes performs a repeat set of runs, such as to close the round-robin loop at the same site. In this case, increment to Set2, etc.

b) Data File Content

Data Quantity	Column Label	Units / Format / Notes
Site number	SiteNum	1, 2, 3, 4,…N
Cell number	CellNum	1, 2, 3, 4,…N
Date	Date	mm/dd/yy
Time	Time	hh:mm:ss 24-hour format – i.e., 13:30:55
Sequence step	Step	0, 1, 2, … 7
Cell load	Load	Amps 1 decimal place
Cell voltage	Voltage	Volts 3 decimal places
Cell temperature	T.Cell	deg C 1 decimal place
Anode dew point temperature	T.An.DP	deg C 1 decimal place Measured as saturator water temperature
Cathode dew point temperature	T.Ca.DP	deg C 1 decimal place Measured as saturator water temperature
Anode outlet pressure	P.An.Out	kPa absolute 1 decimal place
Cathode outlet pressure	P.Ca.Out	kPa absolute 1 decimal place
Anode hydrogen flow rate	Flow.An	SLPM 3 decimal places Referenced to 0 C, 100 kPa
Cathode air flow rate	Flow.Ca	SLPM 3 decimal places Referenced to 0 C, 100 kPa

A8) Shipping

1) For shipping, use appropriate packing provided to avoid damage to plates and hardware.

2) Ship with “do not freeze” specification.

Appendix B: Round Robin Logistics

Participant	M1	M2	M3	M4	M5	M6	M7	M8	M9	M10	M11	M12
Cell Assembly												
Test Site 1												
Test Site 2												
Test Site 3												
Test Site 4												
Test Site 1 Repeated												

Site Specific Procedure Requirements

Cell Assembly:

Perform Cell Assembly, Leak Check, and Break-in as specified in the protocol.

Test Site 1:

Perform Cell Reconditioning and Polarization Curves.

Test Sites 2 – 4 and Repeat:

Perform Leak Check - Pressure Test Method, Cell Reconditioning, and Polarization Curves.

Test Site 1 Repeated:

Perform Leak Check – Pressure Test Method after all testing completed.

Appendix C: Component Specifications

C1) DuPont™ Nafion® PFSA Membranes

DuPont™ Nafion® PFSA Membranes NE-1135, N-115, N-117, NE-1110

Description

DuPont™ Nafion® PFSA membranes are non-reinforced films based on Nafion® PFSA polymer, a perfluorosulfonic acid/PTFE copolymer in the acid (H^+) form. Nafion® PFSA membranes are widely used for Proton Exchange Membrane (PEM) fuel cells and water electrolyzers. The membrane performs as a separator and solid electrolyte in a variety of electrochemical cells that require the membrane to selectively transport cations across the cell junction. The polymer is chemically resistant and durable.

Order and Packaging Information

Membrane dimensions are based on dry product conditioned at 23 °C and 50% Relative Humidity before cutting. The membrane's water content will affect its dimensions, and the change may not be symmetrical in the length, width, and thickness directions. In addition, certain conditioning steps performed by the customer also may affect the dimensions. Customers may wish to review their membrane treatment steps and dimensional requirements with a Nafion® Technical Representative before establishing membrane shipping dimensions.

Standard dry product dimensions for individual pieces include:

- Width: 0.30 m (min.) to 1.22 m (max.)

- Length: 0.30 m (min.) to 1.22 m (max.)

The membrane delivery package for cut pieces will depend on the size and quantity of the membrane order. Smaller-sized membranes are shipped flat, while longer lengths of individual pieces are shipped on a roll. The membranes are protected with a polyethylene wrap and inner packaging, then placed in shipping containers.

Standard dry product dimensions for roll goods include:

- Width: 12-in (0.305-m) and 24-in (0.610-m) standard roll widths, and roll widths from 0.20-m (min.) up to 1.22-m (max.) on special order. Intermediate widths available in increments of 0.125-in.
- Length: 50-meter standard roll length

There is a 100 m_2 minimum order requirement for non-standard roll widths and lengths. Membrane pieces or rolls can be cut to custom sizes, and special packaging provided at additional cost and/or delivery time. Please contact Nafion® Customer Service for details.

Properties of Nafion® PFSA Membrane

[1]

A. Thickness and Basis Weight Properties

Membrane Type	Typical Thickness (microns)	Basis Weight (g/m_2)
NE-1135	89	190
N-115	127	250
N-117	183	360
NE-1110	254	500

B. Physical and Other Properties

[2]

Property	Typical Value	Test Method
Physical Properties		
Tensile Modulus, MPa (kpsi)		
50% RH, 23 °C	249 (36)	ASTM D 882
water soaked, 23 °C	114 (16)	ASTM D 882
water soaked, 100 °C	64 (9.4)	ASTM D 882
Tensile Strength, maximum, MPa (kpsi)		
50% RH, 23 °C	43 (6.2) in MD, 32 (4.6) in TD	ASTM D 882

water soaked, 23 °C	34 (4.9) in MD, 26 (3.8) in TD	ASTM D 882
water soaked, 100 °C	25 (3.6) in MD, 24 (3.5) in TD	ASTM D 882
Elongation at Break, %		
50% RH, 23 °C	225 in MD, 310 in TD	ASTM D 882
water soaked, 23 °C	200 in MD, 275 in TD	ASTM D 882
water soaked, 100 °C	180 in MD, 240 in TD	ASTM D 882
Tear Resistance - Initial, g/mm		
50% RH, 23 °C	6000 in MD, TD	ASTM D 1004
water soaked, 23 °C	3500 in MD, TD	ASTM D 1004
water soaked, 100 °C	3000 in MD, TD	ASTM D 1004
Tear Resistance[3] - Propagating, g/mm		
50% RH, 23 °C	>100 in MD, >150 in TD	ASTM D 1922
water soaked, 23 °C	92 in MD, 104 in TD	ASTM D 1922
water soaked, 100 °C	74 in MD, 85 in TD	ASTM D 1922
Specific Gravity	1.98	
Other Properties		
Conductivity, S/cm	0.10 min	see footnote[4]
Available Acid Capacity, meq/g	0.92 min	see footnote[5]
Total Acid Capacity, meq/g	0.95 to 1.01	see footnote[5]

1 Measurements taken with membrane conditioned to 23 °C, 50% relative humidity (RH).

2 Physical Properties measured for N-115. Where specified, MD - machine direction, TD - transverse direction. Conditioning state of membrane given. Measurements taken at 23 °C, 50% RH.

3 Tear resistance (g/mm) of dry membrane increases with thickness. Values given measured using 50 micron membrane.

4 Conductivity measurement as described by Zawodzinski, et.al, *J. Phys. Chem.*, 95 (15), 6040 (1991). Membrane conditioned in 100 °C water for 1 hour. Measurement cell submersed in 25 °C D.I. water during experiment. Membrane impedance (real) taken at zero imaginary impedance.

5 A base titration procedure measures the equivalents of sulfonic acid in the polymer, and uses the measurement to calculate the acid capacity or equivalent weight of the membrane.

Properties of Nafion® PFSA Membrane

C. Hydrolytic Properties

Property	Typical Value	Test Method
Hydrolytic Properties		
Water content, % water[6]	5	ASTM D 570
Water uptake, % water[8]	38	ASTM D 570
Thickness change, % increase		
from 50% RH, 23 °C to water soaked, 23 °C	10	ASTM D 756
from 50% RH, 23 °C to water soaked, 100 °C	14	ASTM D 756
Linear expansion, % increase[9]		

from 50% RH, 23 °C to water soaked, 23 °C	10	ASTM D 756
from 50% RH, 23 °C to water soaked, 100 °C	15	ASTM D 756

[7] Water content of membrane conditioned to 23 °C, 50% relative humidity (RH), compared to dry weight basis.

[8] Water uptake from dry membrane to water soaked at 100 °C for 1 hour (dry weight basis).

[9] Typical MD and TD values. MD expansion is slightly less than TD.

Recommended Roll Storage Conditions

Unopened roll packages of Nafion® PFSA membrane should be stored in the original shipping box, out of direct sunlight, and in a climate-controlled environment, maintained at 10 to 30°C, and 30 to 70% relative humidity. Before opening the package, pre-condition the membrane roll to the processing area temperature for 24 hours.

Once opened and exposed to the environment, the membrane will equilibrate to the ambient relative humidity, and change in dimensions accordingly. Membrane order dimensions are specified and measured at 23°C and 50% Relative Humidity.

Handling Practices

Ventilation should be provided for safe handling and processing of Nafion® PFSA membrane. The amount of local exhaust necessary for processing Nafion® PFSA membrane at elevated temperatures will depend on the combined factors of membrane quantity, temperature, and exposure time.

Scrap Disposal

Preferred disposal options are (1) recycling and (2) landfill. Incinerate only if incinerator is capable of scrubbing-out hydrogen fluoride and other acidic combustion products. Treatment, storage, transportation, and disposal must be in accordance with applicable federal, state/provincial and local regulations.

Safe Handling and Use of Nafion® PFSA Membranes

The following information should be reviewed before handling and processing Nafion® PFSA Membranes:

- DuPont Material Safety Data Sheet for Nafion® PFSA Membranes NE-1135, N-115, N-117 and N-1110
- Nafion® Technical Information "Safe Handling and Use"

• "Guide to Safe Handling of Fluoropolymer Resins", Third Edition, June 1998, Published by the Fluoropolymers Division of the Society of the Plastics Industry, Inc.

For more information about Nafion® contact:

DuPont Fuel Cells	Telephone: (910) 678-1380
Nafion® Global Customer Service	Domestic U.S.A. only: (800) 436-1336
22828 NC Highway 87 W	Overseas: (910) 678-1337
Fayetteville, NC 28306, U.S.A.	Fax: (910) 678-1342

DuPont Fuel Cells Web Site: http://www.dupont.com/fuelcells

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The data listed here fall within the normal range of product properties, but they should not be used to establish specification limits nor used alone as the basis of design. This information is based on technical data that DuPont believes to be reliable. It is intended for use by persons having technical skill and at their own discretion and risk. This information is given with the understanding that those using it will satisfy themselves that their particular conditions of use present no health or safety hazards. Because conditions of product use are outside our control, DuPont makes no warranties, express or implied, and assumes no obligation or liability in connection with any use of this information or for results obtained in reliance thereon. The disclosure of the information is not a license to operate under or a recommendation to infringe any patent of DuPont or others.

Caution: Do not use in medical applications involving permanent implantation in the human body. For other medical applications, see "DuPont Medical Caution Statement", H-50102.

Revised: 12Sep2005

C2) Saint Gobain Performance Plastics COATED FABRIC 1010

PRODUCT DESCRIPTION

COHRlastic® Coated Fabric 1010 is a general purpose silicone rubber coated fiberglass fabric featuring abrasion resistance, excellent electrical properties and an operating temperature range of -85°F to +500°F. Typical applications include electrical gasketing, heater covering, conveyor belts, shrink tunnel curtains, release or separator fabrics.

APPLICATION ADVANTAGES

- Combination of silicone coating and fiberglass is flexible and conformable.
- Resistant to wear and tear.
- Can be bonded to itself with silicone RTV.
- Excellent release surface.
- Excellent chemical resistance.

*PHYSICAL PROPERTIES**

Overall Thickness	(in.)	.010
Fiberglass Thickness	(in.)	.0038
Break Strength	(lbs/in.)	Warp 175 Fill 150
Tear Strength	(gr.)	Warp 6,000 Fill 4,000
Elongation	(%)	<10
Burst, Diaphragm	(psi)	300
Temperature Range	(ºF)	-85 - +500
Color		White
Weight	(oz/yd²) avg.	11

*Values are typical properties and should not be used for writing specifications.

AVAILABILITY

CF 1010 is sold by the yard in continuous lengths, 36" wide.

® REGISTERED TRADEMARK

March '97

SAINT-GOBAIN PERFORMANCE PLASTICS

14 McCaffrey Street

Hoosick Falls, NY 12090

Phone: 518.686.7301 (800.962.2666)

Fax: 518.686.4840 (800.526.8479)

C3) Denora E-TEK Solid Polymer Electrolyte Electrode ELAT® Reference

Solid Polymer Electrolyte Electrode ELAT® Reference Grade (ELAT-rg) Single Sided Coating

General Information: Gas diffusion electrodes are used in a wide variety of applications such as fuel cells, metal/air batteries, industrial electrolytic processes, gas sensors, and, of course, in research and development activities. While many of E-TEK's assemblies are used as standards, the ELAT-rg is applied as a reference component for polymer electrolyte fuel cell test stands.

SUGGESTED USES

In research, development, and monitoring of production, calibration and certification of fuel cell test stations serves a vital function. These fabrications have been designed with the highest piece-to-piece reproducibility in order to reduce data scatter due to component variation. Typical applications are for use in standard single-cell test methods such as that developed by the United States Fuel Cell Council (USFCC) www.usfcc.com

DESCRIPTION

SS-ELAT-rg 20% Pt 0.5mg/cm2 0.75mg/cm2 Nafion®

SPECIFICATIONS

SS-ELAT-rg 20% Pt 0.5mg/cm2 0.75mg/cm2 Nafion

With each lot of ELAT-rg we provide documentation providing a lot average (for orders of three or more pieces) and individual piece specifications including:

- actual Pt loading, determined via XRF
- thickness, measured at 7psi
- basis weight (g/m2)

OPTIONS

Non-standard piece sizes available as a custom order (maximum size 250mm x 250mm).

SIZE

Intended for use with a standard machined quadruple serpentine flow fields: fuel cell active area 50 cm2. Actual piece size is 70.4mm x 70.4mm (2.77\" x 2.77\").

C4) TELEDYNE MEDUSA™ CH Single Cell Test Hardware Series

MEDUSA CH-5, CH-25, CH-50

Teledyne Energy Systems, Inc. (TESI) is now offering 5, 25 and 50 cm2 single cell test hardware designed for quality, ease of assembly, and ease of operation. Features of TESI's fuel cell test hardware include:

- Durable stainless steel compression endplates provide a pure medium for gas delivery with machine bolts located conveniently around the perimeter to provide even compression of the MEA;
- Swagelok® fittings for gas inlet and outlet connections;
- Endplates heated using durable cartridge heaters, with a thermocouple well in the cathode endplate for accurate temperature control;
- Gold plated copper bus plates, electrically insulated from the endplates;
- Connections for each bus plate on opposite sides with the remaining half of the plate unexposed, preventing shorts between the two collector plates;
- Graphite collector plates, precision machined at TESI's manufacturing facility with a serpentine flow field for optimum flow of reactants; and,
- Bus plates affixed to the respective endplates, for ease of assembly.

The single cell test hardware is delivered with everything for immediate testing, with exception to the fuel cell cables. Cables are provided with the MEDUSA RD Fuel Cell Test Station. Custom flow fields and active areas are available. Please contact TESI for details.

Teledyne Energy Systems Web Site: http://www.teledynees.com