Application Note – Repeatability of In-Plane Fuel Cell Membrane Conductivity Measurements

A reasonable question one may ask is "*How repeatable are in-plane membrane conductivity measurements when using the BekkTech Conductivity Cell?*"

To answer this question, we performed replicate in-plane conductivity measurements on commercially-available Nafion™ NR-212 at 80 °C using a BT-710 In-Plane Conductivity Cell and a Scribner 740 Membrane Test System. Ten unique samples from the same production lot of Nafion were used.

The relative humidity (RH) profile consisted of a 60 min conditioning step at 80% RH, followed by an RH sweep from 95% to 10% RH with a 30 min RH-step duration. All measurements were conducted using ultra-high purity (99.999%) H_2. and ultra-pure humidifier water (18 MΩ -cm). Sample temperature and humidity control were better than ±0.3 °C and ±0.2% RH.

At the end of each 30 min RH step, the membrane resistance was obtained via a linear voltage sweep (0 V → -0.1 V → +0.1 V → 0 V *vs*. OCV) at 10 mV/sec scan rate using a Solartron 1287 potentiostat (Ametek) and CorrWare® (Scribner Associates).

The slope of Voltage vs. Current response was used as the resistance (R in Ω) in the conductivity calculation,

$$\sigma\ (S/cm) = \frac{L}{R \cdot W \cdot T}$$

Sample width (W) and the distance between the two V-sense leads (L) were obtained by optical analysis of calibrated images of the samples. The as-received nominal thickness of Nafion NR-212 ($T_{nominal}$ = 50.8 μm) and measured thickness ($T_{measured}$) were used in calculation of the membrane conductivity. Measured thickness was determined as the average of five measurements for each membrane sample using a low-force precision thickness meter Model MP-1VF (Brunswick Instruments, Inc.).

In this study, ten Nafion NR-212 samples were tested at 80 °C (N = 10). Results of the calculated membrane conductivity as a function RH are shown in Table 1 and Table 2 based on the nominal and measured thickness, respectively.

Statistical parameters that can be used to express measurement repeatability include the range (= Maximum – Minimum), range expressed as a percent of the mean ($\bar{X}$), standard deviation (s), and relative standard deviation (RSD),

$$RSD\ (\%) = s/\bar{X}$$

Each of these parameters are presented in the Tables.

The range was ~ 10-15% of the mean while the standard deviation was approximately 1/3 that amount, *i.e.*, RSD ~ 3-5%. This level of reproducibility is consistent with the degree of reproducibility of the through-plane method for the 740 MTS (unpublished work).

rev. A, 4/2020

Table 1. Nafion NR-212 in-plane conductivity at 80 °C based on nominal thickness, $T_{nominal}$ = 50.8 μm.

Nominal RH	Conductivity @ $T_{nominal}$ (N = 10)						
	$\bar{X}$	Max.	Min.	s	Range	Range / $\bar{X}$	RSD
%	mS/cm	mS/cm	mS/cm	mS/cm	mS/cm	%	%
80	102.4	111.8	96.1	4.8	15.7	15.4	4.7
95	168.4	178.3	160.2	6.6	18.1	10.8	3.9
90	141.6	152.6	134.5	5.8	18.1	12.8	4.1
80	104.0	112.5	98.3	4.8	14.2	13.7	4.6
60	57.5	61.8	53.7	2.6	8.1	14.1	4.5
50	40.1	43.7	37.9	1.8	5.8	14.5	4.5
40	25.7	27.2	24.2	1.0	3.1	11.9	3.8
20	6.5	6.9	6.0	0.3	0.9	14.4	4.3
10	1.7	1.8	1.5	0.1	0.3	18.9	5.8
80	101.2	108.9	93.9	4.4	15.0	14.8	4.3

Table 2. Nafion NR-212 in-plane conductivity at 80 °C based on measured thickness, $T_{measured}$.

Nominal RH	Conductivity @ $T_{measured}$ (N = 10)						
	$\bar{X}$	Max.	Min.	s	Range	Range / $\bar{X}$	RSD
%	mS/cm	mS/cm	mS/cm	mS/cm	mS/cm	%	%
80	97.5	104.9	91.8	4.0	13.1	13.4	4.1
95	160.4	170.6	152.3	5.7	18.4	11.4	3.5
90	134.8	143.1	127.7	5.0	15.4	11.4	3.7
80	99.0	105.5	92.8	4.1	12.8	12.9	4.1
60	54.8	58.8	51.5	2.3	7.3	13.4	4.1
50	38.1	41.0	36.2	1.6	4.7	12.4	4.3
40	24.5	26.1	23.2	0.9	3.0	12.1	3.6
20	6.2	6.6	5.8	0.2	0.8	12.9	3.8
10	1.6	1.7	1.4	0.1	0.3	20.3	5.6
80	96.3	103.3	91.4	3.7	11.8	12.3	3.9

Figure 1 shows conductivity as a function of RH on logarithmic (top) and linear (bottom) scale. The latter highlights the error bars which are ±½-range in both plots, *i.e.*, the full height of the error bar equals the range.

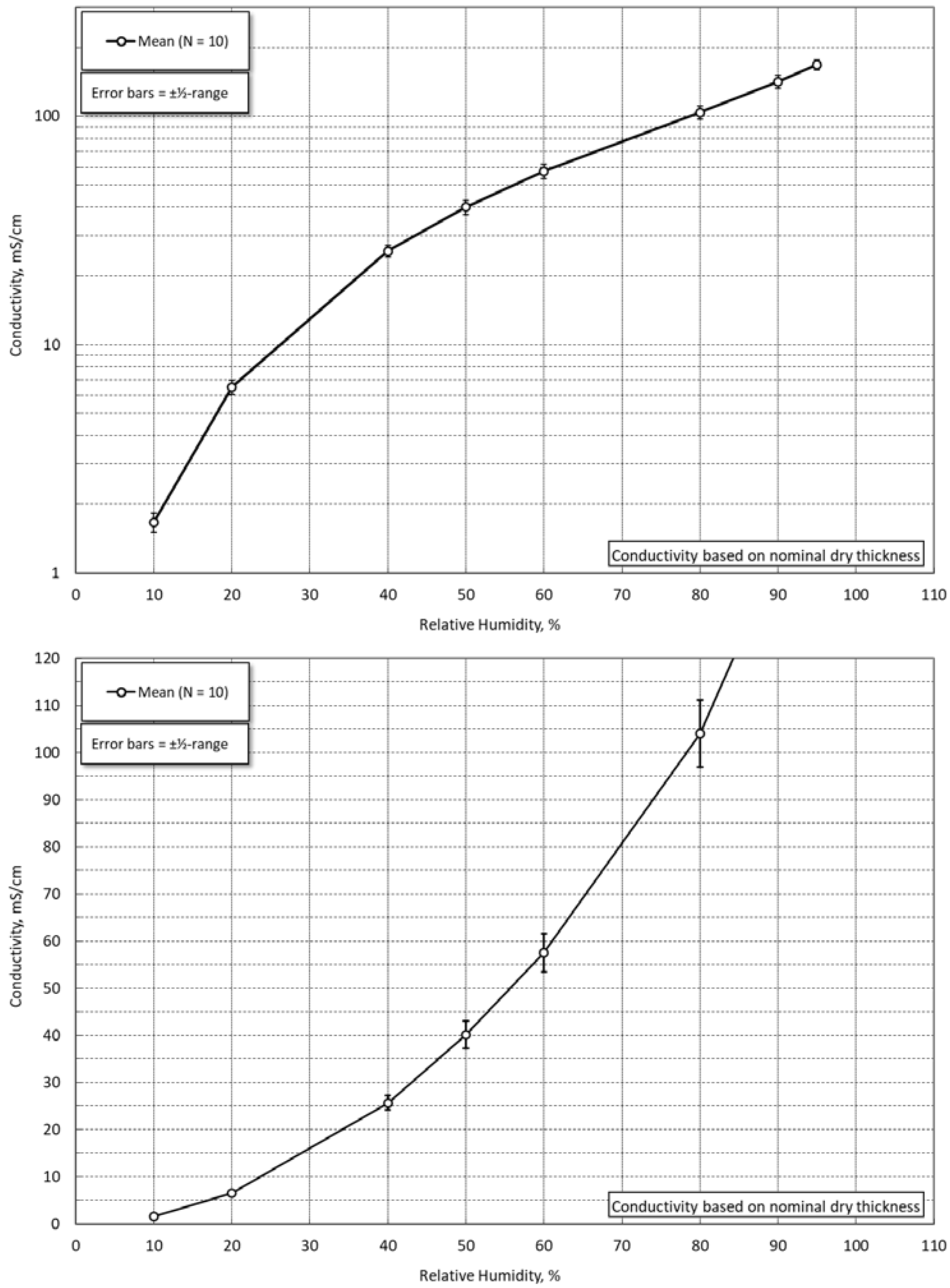

Figure 1. In-plane conductivity of Nafion NR-212 as a function of RH at 80 °C. Top: logarithmic scale; bottom: linear scale.

Factors Affecting Repeatability

1. Accuracy of sample dimension measurements are significant factors. Relevant dimensions including sample width (W) and thickness (T) as well as the distance between the V-sense leads (L). The nominal distance between the V-sense leads for the BekkTech cell is 0.45 cm. In the study, however, we observed that L varied by as much as 0.033 cm between samples, or ~ 7% of the nominal value. These results suggest that repeatability may be improved by measuring L and membrane dimensions for each sample for example, by analysis of calibrated images of the sample pre- and post-test.

With reference to sample dimensions, we note that reproducibility improved slightly when the measured thickness was used in place of the nominal thickness in the conductivity calculation.

2. Reproducibility of the test conditions, specifically the test temperature, but more importantly relative humidity also play a significant role is the observed measurement reproducibility. Because of the very strong sensitivity of the resistivity of PFSA-based materials to water content, test-to-test variation in RH can have a significant impact on observed membrane conductivity. The 740 MTS control software has the ability to perform dynamic RH control, which gives excellent RH reproducibility (±0.2% RH) and therefore likely represents the best-case-scenario with respect to overall reproducibility of the environmental conditions.

Nafion™ is a trademark of The Chemours Company FC, LLC.