

Standard Operating Procedure (SOP)

Water Uptake Measurement

Test ID # LTE-P-20

Rev 3

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Prepared By:

04/09/2019
Date

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Date: 09/24//2019

Revision History

This page documents the revisions over time to the SOP. The most recent iteration should be listed in the row space, with consecutive versions following.

Date of Revision	Page(s)/Section(s) Revised	Revision Explanation
04/09/2019	All	First Release
09/24/2019	All	Formatting, minor edits
03/04/2020	All	Revised procedure with edits Distributed for review

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1. Procedures

- a. Scope and Applicability – Determine the percent of water content of membrane
- b. Summary of Method – This procedure applies to the receiving inspection for incoming membrane
- c. Definitions – Not applicable for this SOP
- d. Health & Safety Warning – Not applicable for this SOP
- e. Cautions – When hydrating the membrane, do not allow the water to boil away. Exposure of the membrane to high temperature without water coverage may thermally decompose the samples, altering results. Materials used should be compatible with the membrane, so as not to introduce contaminants that could alter results.
- f. Interferences – Environmental conditions of the room should be measured during the water uptake measurements. Change from day to day in temperature and humidity can impact measurement results.
- g. Personnel Qualifications / Responsibilities – The user of this procedure should have basic laboratory safety training per the organization’s requirements.
- h. Equipment and Supplies –
 1. Membrane (Confirm that the membrane has a “date received” and that the date of use is not beyond the expiration date.)
 2. 500 ml Pyrex beaker
 3. Three (3) 100 ml Pyrex beakers
 4. Watch glass
 5. Deionized water (DI)
 6. Teflon coated tweezers
- i. Step by Step Procedure
 - Instrument or Method Calibration and Standardization – The user should have a calibrated scale available, as well as a calibrated temperature probe for use in DI water and material compatibility with the membrane sample
 1. Cut three samples of the film, each approx. 0.1 g. (*You can also use a consistent area for the sample instead of a weight*)

2. Put samples into a beaker (500 ml capacity) and add 250 ml of DI water, cover with the watch glass, heat and maintain at 80°C for about 3 hours.
3. Remove the samples and place each into a beaker (100 ml capacity). Add 60 ml of fresh DI water and let them cool down for about 1 hour.
4. Blot each membrane sample with lint-free tissue and then weigh the membrane (analytically on a watch glass). Repeat this step two more times while allowing the membranes to equilibrate in DI water for at least 5 minutes between drying/weighing. Calculate the average of three wet weight measurements for each membrane.
5. Obtain the mass of the dry membrane as described in the procedure i(3) of SOP Test ID # LTE-P-3: The membrane sample is blotted with lint-free tissue followed by 1 h air dry at room temperature. The membrane is further dried for at least 12 h in vacuum without heating (see Figure 1A for recommended vacuum dry set up; heating under vacuum causes change in color of Nafion™ 212 sample as shown in Figure 1B). During vacuum dry process, it is recommended to keep the membrane sample inside a loosely capped polypropylene bottle (see Figure 1A). Polypropylene bottle, which is hydrophobic in nature, can minimize the percentage error that could be caused by the absorption of the moisture if glass bottles are used. After drying for 12 h, the desiccator is filled with nitrogen and the polypropylene bottle is immediately closed tightly. The bottle is then weighed. The membrane is removed, and the empty bottle is reweighed to determine the mass of the dry membrane. An analytical balance with tolerance of 0.1 mg and capacity of >1 g is used for measurement.
6. Calculate the water content using the following formula as an average of three replicates noting the standard deviation and record the results.

j. Data and Records Management –

$$\text{Weight Percent Water} = \frac{\text{Wet weight} - \text{Dry weight}}{\text{Dry weight}} \times 100\%$$

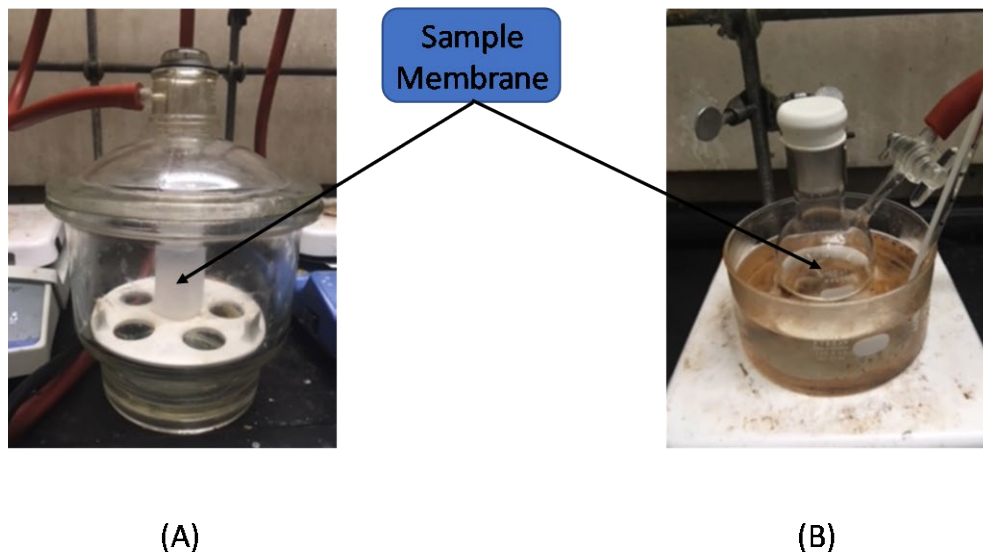


Figure 1: (A) Vacuum dry set up without heating, and (B) Vacuum dry set up with heating at 80 °C.

4. Quality Control and Quality Assurance Section

All instrumentation used for measurements should be calibrated annually. These include at least the following:

- Thermometer/Temperature probe
- Weighing scale
- Drying oven/desiccator
- Room temperature gauge
- Room humidity sensor

5. Validation of the SOP using Nafion™ 212

Using Nafion™ 212 (Reported Water Uptake = 50 ± 5%)

Nafion™ 212 Sample	Dry Mass (mg)	Wet Mass* (mg)	Measured Water Uptake (%)	Average Water Uptake (%)
1	133.5	197.5	64	57 ± 6
2	135.7	193.7	58	
3	144.8	195.8	51	

* average of three measurements

6. Reference Section

- R1. 40 CFR 30, Code of Federal Regulations, “Grants and Agreements With Institutions of Higher Education, Hospitals, and Other Non-Profit Organizations.”
- R2. ANSI/ASQC E4-1994, *Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs*, American National Standard, January 1995.
- R3. U. S. Environmental Protection Agency, 2000a. *Guidance for Data Quality Assessment: Practical Methods for Data Analysis (QA/G-9)*, EPA/600/R-96/084, Office of Environmental Information.
- R4. <https://www.fuelcellsetc.com/store/N212>