Standard Operating Procedure (SOP)

Conductivity Measurements for Cation Exchange Membrane in Electrolyte

Test ID #PEC-P-8

Rev 1

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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
04/16/2019	All	First Release

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3. Procedures

a. Scope and Applicability – This SOP details procedures for performing conductivity measurements on cation exchange membranes (CEMs) in electrolytes.

b. Summary of Method – A piece of pre-treated CEM is installed between the cathode and anode chamber in an electrochemical cell with flowing fresh electrolyte. Two reference electrodes are pointed on each side of the membrane with an angle. Increasing multi-steps current densities are applied by a potentiostat between the two metal electrode plates. The voltage between the two reference electrodes is monitored by a Keithley voltage meter. The resistance of the thin electrolyte layers and the CEM is determined by the slope of the current density and voltage curve. The tip distance of the two reference electrodes are moved further from each other step by step, and the previous procedures for measuring the resistance are repeated. A straight curve of resistance as a function of the tip distance is fitted, and the resistance of the membrane is determined by the intercept of the curve, assuming that the resistance of the thin electrolyte layers is excluded (distance = 0). The conductivity of the membrane (mS cm^{-1}) is calculated by the resistance divided by the thickness of the membrane.

c. Definitions –

- CEM – Cation exchange membrane
- WE – Working electrode
- RE – Reference electrode
- CE – Counter electrode

d. Health & Safety Warning – Acidic electrolytes present both acute and chronic exposure hazards to eyes and skin, and proper PPE gloved should be worn to minimize exposure. Main routes of exposure are inhalation, skin contact and eye contact.

e. Cautions – Impact-resistant safety goggles, face shield, heavy gloves, and clothing providing full skin coverage must be worn when handling the acidic electrolyte.

Inhalation: Not expected to be an inhalation hazard unless heated or misted. Very toxic, can even cause death. Can cause severe irritation of the nose and throat. Can cause life-threatening accumulation of fluid in the lungs (pulmonary edema). Symptoms may include coughing, shortness of breath, difficult breathing and tightness in the chest. Long-term damage may result from a severe short-term exposure.

Skin contact: Corrosive. Contact can cause pain, redness, burns, and blistering. Permanent scarring can result. A severe exposure can cause death.

Eye contact: Corrosive. Contact causes severe burns with redness, swelling, pain and blurred vision. Permanent damage including blindness can result.
Ingestion: Can burn the lips, tongue, throat and stomach. Symptoms may include nausea, vomiting, stomach cramps and diarrhea. Permanent damage can result. Can cause death.

Effects of long-term (chronic) exposure: At low concentrations: Can cause dry, red, cracked skin (dermatitis) following skin contact. At high concentrations: May wear away tooth enamel when breathed in. May harm the respiratory system. Can irritate and inflame the airways.

Carcinogenicity: Not known to cause cancer. Strong inorganic mists containing sulfuric acid are carcinogenic to humans. Has been associated with: cancer of the larynx, lung cancer.

f. Interferences – The CEM must be boiled in hot water and soak in the electrolyte for conditioning before measurement. The averaged voltage between the two reference electrodes has to be accurately determined each step to ensure an accurate resistance. The tip distance between the two reference electrodes has to be measured accurately.

g. Personnel Qualifications / Responsibilities – Training with laboratory safety regarding chemicals is required. Familiarity with potentiostat and Keithley voltage meter is needed.

h. Equipment and Supplies –

- Electrochemical stacked cell
- Two reference electrodes
- Metal (stainless steel 316L for example) electrodes
- Potentiostat
- Keithley voltage meter (or another channel from a potentiostat)
- Vernier caliper (or ruler)

i. Step by Step Procedure <u>(LIST ALL PERTINENT STEPS, IN ORDER, AND THE MATERIALS NEEDED TO ACCOMPLISH)</u>

- Instrument or Method Calibration and Standardization – This SOP needs a potentiostat to supply the stepped voltage for the two electrodes, and need a Keithley voltage meter (or another channel in a potentiostat) to monitor the voltage with time.

- Sample Collection – Not applicable for this SOP.

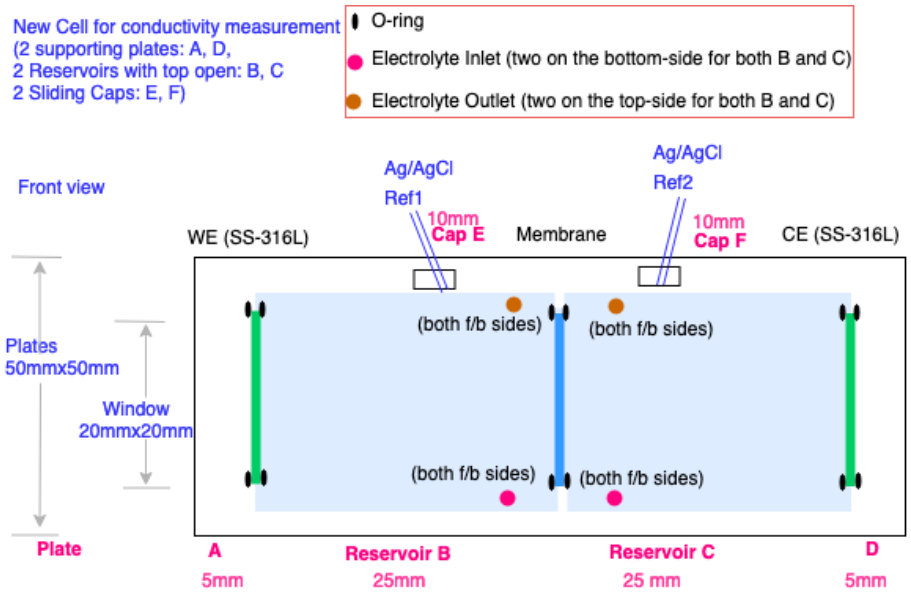
- Sample Handling and Preservation – Not applicable for this SOP.

- Sample Preparation and Analysis –

Peel off the plastic cover of the CEM (if applicable) and boil the CEM in DI water (70 ^{o}C) for ~20 min.

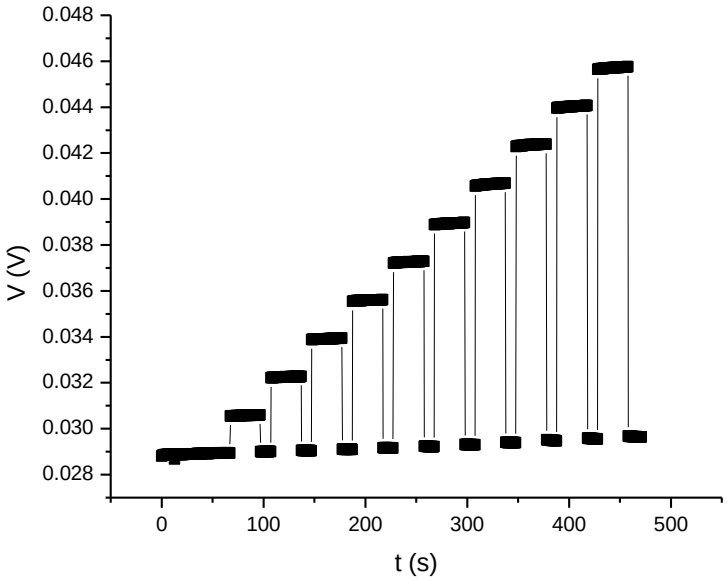
Soak the CEM in the electrolyte (the same electrolyte used in the measurement) for 2 hours for conditioning.

Install the CEM between the cathode and anode chamber in an electrochemical cell. Two reference electrodes are pointed on each side of the membrane (as close as possible) with an angle.



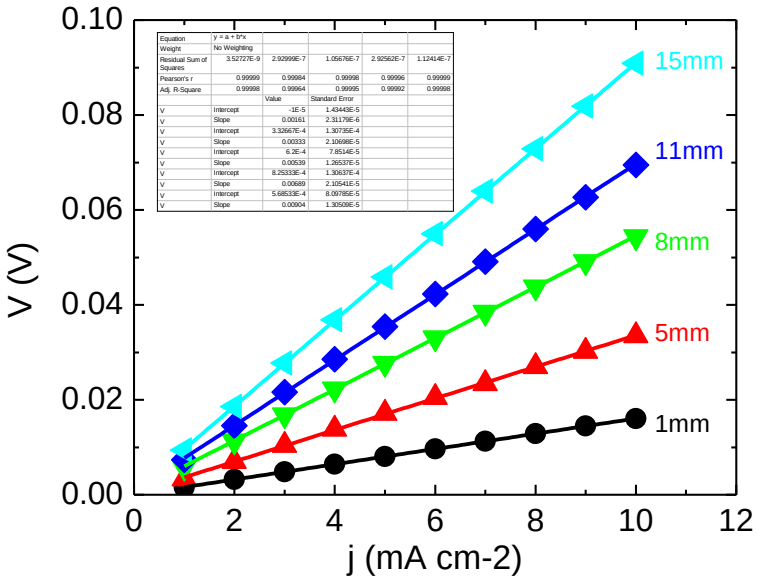
Fill in the cathode and anode chamber by flowing fresh electrolyte (0.5 M NaCl or 0.5 M H_2SO_4) using a peristaltic pump. Continuously flow the fresh electrolyte through the inlets on the bottom sides of the chamber to the outlets on the top sides of the chamber during the measurement.

Increasing multi-steps current density (1, 2, 3,…10 mA cm^{-2} for 30 second in each step with a 10-second-step 0 mA cm^{-2} for equilibrium) are applied by a potentiostat between the two metal electrode plates (stainless steel 316L). Meanwhile, the voltage between the two Ag/AgCl reference electrodes is monitored by a Keithley voltage meter.

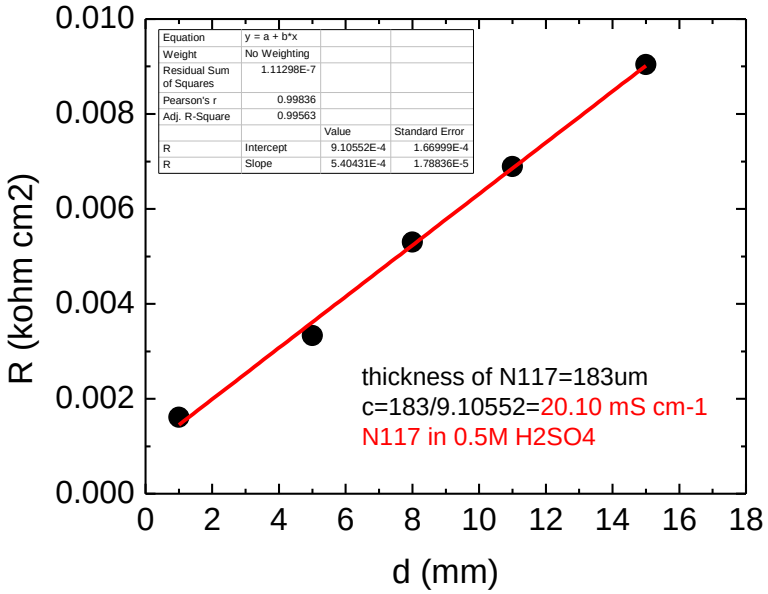


The resistance of the thin electrolyte layers and the CEM is determined by the slope of the current density and voltage curve.

The tip distance of the two reference electrodes are moved further from each other step by step (1, 5, 8, 11 and 15 mm, for example), and the previous procedures for measuring the resistance are repeated.



A straight curve of resistance as a function of the tip distance is fitted, and the resistance of the membrane is determined by the intercept of the curve, assuming that the resistance of the thin electrolyte layers is excluded (distance = 0).



The conductivity of the membrane (mS cm^{-1}) is calculated by the resistance divided by the thickness of the membrane.

- Troubleshooting – Sometimes the monitored voltages are not in a well-defined steps shape. The following steps should be checked:

  The tilted angles for the reference electrodes should be kept constant.
  The electrolyte should flow fast enough to ensure fresh enough in the chambers.
  The metal electrodes should not be oxidized on the surface.

- Data Acquisition, Calculations & Data Reduction Requirements –
  The monitoring voltage data is acquired by the IV software with an interval of 0.5 s.
  In each voltage step, the averaged difference between the voltage with (1, 2, 3… 10 mA cm^{-2}) and without (0 mA cm^{-2}) applied current is adopted.

- Computer Hardware & Software –
  Data processing software such as Excel, Matlab, Origin can be used for processing, fitting and plotting the data.

j. Data and Records Management – Not applicable for this SOP.

4. Quality Control and Quality Assurance Section

   Not applicable for this SOP.

5. Reference Section