

**Submission ID #: 71248**

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**Project Page Link: <https://review.jove.com/account/file-uploader?src=21730688>**

**Title: Measuring Proton Conductivity in MOF-Based Mixed Matrix Membranes by Electrochemical Impedance Spectroscopy**

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## **Author Questionnaire**

- 1. Microscopy:** Does your protocol require the use of a dissecting or stereomicroscope for performing a complex dissection, microinjection technique, or something similar? **No**
- 2. Software:** Does the part of your protocol being filmed include step-by-step descriptions of software usage? **Yes**
- 3. Filming location:** Will the filming need to take place in multiple locations? **No**
- 4. Testimonials (optional):** Would you be open to filming two short testimonial statements **live during your JoVE shoot**? These will **not appear in your JoVE video** but may be used in JoVE's promotional materials. **No**

### **Current Protocol Length**

Number of Steps: 16

Number of Shots: 34

# Interviews

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*Videographer: Obtain headshots for all authors available at the filming location.*

## INTRODUCTION:

~~REQUIRED: Summarize your research focus and the main questions it aims to answer.~~

- 1.1. **Ermias A. Mideksa**: Our protocol focuses on measuring the in-plane proton conductivity of MOF-polymer mixed-matrix membranes using Electrochemical Impedance spectroscopy under controlled humidity and temperature conditions.
  - 1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

~~Describe the research contexts or systems in which this protocol can be applied.~~

- 1.2. **Ermias. A. Mideksa**: This protocol can be applied to polymer or MOF-based mixed-matrix membranes for proton exchange membrane fuel cell applications.
  - 1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

## CONCLUSION:

~~Describe the most important consideration or challenge when performing this protocol.~~

- 1.3. **Ermias. A. Mideksa**: The most important considerations are proper membrane placement, workstation connection, and membrane equilibration at the specified humidity and temperature.
  - 1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

~~Are there any additional methods or analyses that can be performed following this procedure?~~

- 1.4. **Ermias. A. Mideksa**: Additional methods that can be performed following this procedure include combining in-plane and through-plane conductivity measurements to reliably evaluate proton conductivity.
  - 1.4.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

~~How can future studies build on this work? Describe the questions or research directions that can be investigated next.~~

- 1.5. **Ermias. A. Mideksa**: Future studies can build on this work by testing membrane performance in membrane electrode assemblies and fuel cell stacks under real operating conditions.

1.5.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera.

**Videographer's Notes: 1.6.1 is an added intro that the author wanted to film.**

***Videographer: Obtain headshots for all authors available at the filming location.***

# Protocol

*Videographer: Please always record an audio slate while filming each shot mentioned in the shotlist.*

## 2. In-Plane Proton Conductivity Measurements

**Demonstrator: Ermias A. Mideksa**

NOTE: SCREEN timestamps for protocol were added at the postshoot stage. Please contact the postshoot note integrator (Sri Lakshmi Priya) for queries regarding lab media.

- 2.1. To begin, obtain the previously prepared polyvinyl alcohol or PVA (*P-V-A*) and PVA mixed-matrix membranes containing MIP-207 (*M-I-P-two-zero-seven*) sulfonic acid membrane samples [1].
  - 2.1.1. WIDE: Talent placing the previously prepared membranes on the workstation. *Videographer: Please obtain CU shot as well for this shot.*
- 2.2. Cut the fabricated membrane samples according to the in-plane measurement cell geometry [1]. Using a micrometer, measure the sample dimensions and thickness at several positions [2]. Calculate and record the average thickness [3].
  - 2.2.1. Talent cutting the fabricated membrane samples according to the in-plane measurement cell geometry. **Videographer's Notes: 2.2.1.1 Cutting other membrane sample**
  - 2.2.2. Talent measuring the sample dimensions and thickness at several positions using a micrometer. **Videographer's Notes: Measuring size of membranes**
  - 2.2.3. Talent recording the calculated average thickness. **Videographer's Notes: Measuring thickness and recording in notebook**
- 2.3. Assemble the conductivity cell by placing the membrane between 2 electrodes with a known geometric contact area,  $A$  [1]. Then, sandwich the assembly between 2 Teflon cells [2] and gently tighten it with screws and hex nuts to ensure good interfacial contact [3].
  - 2.3.1. Talent placing the membrane between 2 electrodes.
  - 2.3.2. Talent placing the assembly between 2 Teflon cells.
  - 2.3.3. Talent tightening the assembly with screws and hex nuts.
- 2.4. Place the assembled Teflon cells inside a climatic humidity chamber directly connected to a potentiostat [1]. Set the chamber to the desired temperature and RH (*R-H*) conditions [2].
  - 2.4.1. Talent placing the assembled Teflon cells inside the climatic humidity chamber.

Videographer's Notes: 2.4.1.1 Close up shot of 2.4.1

2.4.2. Talent setting the desired temperature and relative humidity conditions in the climatic humidity chamber. Videographer's Notes: Talent in front of computer

2.5. Program the temperature and relative humidity set points directly on the programmable humidity chamber [1]. Allow the set points to change automatically over time without manual intervention [2].

2.5.1. SCREEN: 71248\_How-to-program-a-climatic-chamber\_Pimenta.mov: 01:37-01:54, 02:37-02:49

2.5.2. SCREEN: 71248\_How-to-program-a-climatic-chamber\_Pimenta.mov: 04:19-04:34

2.6. Connect the in-plane conductivity cell to the potentiostat equipped with a frequency response analyzer [1]. Place the setup at the selected temperature and RH condition [2]. Allow the membrane to equilibrate for 1 hour before measurement [3].

2.6.1. Talent connecting the in-plane conductivity cell to the potentiostat.

2.6.2. Talent placing the setup at the selected temperature and RH condition.

2.6.3. Shot of the membrane setup equilibrating in the selected condition.

2.7. Specify a frequency range using the electrochemical impedance spectroscopy or EIS (*E-I-S*) software from 1 megahertz to 1 hertz with at least 2 measurements per frequency [1]. Set the alternating current or AC (*A-C*) perturbation amplitude to 20 millivolts [2]. Start the measurement and record the impedance spectrum for subsequent analysis [3].

2.7.1. SCREEN: Show the electrochemical impedance spectroscopy software while entering the frequency range from 1 megahertz to 1 hertz. Videographer's Notes: 2.7.1-2.7.3, and 2.8.1 are recordings of the screen. (problem putting OBS on other computer)

2.7.2. SCREEN: Show the alternating current perturbation amplitude set to 20 millivolts.

2.7.3. SCREEN: Show the measurement starting and the impedance spectrum being recorded.

2.8. Save the impedance spectrum for subsequent analysis after the measurement is complete [1].

2.8.1. SCREEN: Show the completed impedance spectrum being saved for subsequent analysis. Author's Notes: Comes immediately after 2.7.2

NOTES: The videographer's and author's notes don't match in 2.8 and 2.9. Go with the author's changes.

Skip Videographer's Notes: 2.8.2 is an added shot of the chamber at 60 degrees

Videographer's Notes: 2.8.3 also added shot showing the ending of the experiment on computer

- 2.9. Verify that the humidity chamber is set to 60 degrees Celsius and 95 percent RH [1]. Allow it to cool for 1 hour until the temperature is below 30 degrees Celsius [2]. Remove the measurement cells from the humidity chamber [3]. Turn the humidity chamber off [4]. Disassemble the measurement cells [5], and confirm that the membrane remains mechanically intact with no visible wear or tear, after the proton conductivity measurement conditions [6]. Notes: VO has been updated for the added shot and revised to align with the moved shots.

Added Shot: Talent shows humidity chamber at 60 degrees Celsius and 95% RH

- 2.9.2 Shot of the humidity chamber cooling until the temperature is below 30 degrees Celsius. Videographer's Notes: 2.9.1-2.9.3 are one shot, 2.9.2 extra shot close up taking sample out of chamber.

Notes: 2.9.2 and 2.9.3 are moved before 2.9.1 as per the author's request

- 2.9.3 Talent removing the measurement cells from the humidity chamber.

- 2.9.1. Talent turning the humidity chamber off. Videographer's Notes: 2.9.1 and 2.9.2 are flipped. First remove samples from chamber, then turn the machine off

~~2.9.2. Shot of the humidity chamber cooling until the temperature is below 30 degrees Celsius.~~

~~2.9.3. Talent removing the measurement cells from the humidity chamber.~~

- 2.9.4. Talent disassembling the measurement cells.

- 2.9.5. Talent inspecting the membrane for visible wear and tear.

- 2.10. Dry the membrane [1] and store it in a dedicated plastic bag to verify structural and mechanical properties after the conductivity measurements [2].

- 2.10.1. Talent drying the membrane.

- 2.10.2. Talent storing the membrane in a dedicated plastic bag.

- 2.11. Analyze the EIS results by determining the membrane resistance from the Nyquist plot [1]. Read the resistance as the intersection between the high-frequency semicircle and the real axis [2].

2.11.1. SCREEN:71248\_Proton\_Conductivity\_Calculation\_Pimenta.mov: 01:47-01:55  
Videographer's Notes: 2.11.1 is a backup of the first half of all the screen shots at the end of the script. 2.11.1.1 is part two of the screen backup

- 2.11.2. SCREEN: 71248\_Proton\_Conductivity\_Calculation\_Pimenta.mov: 02:49-03:10

- 2.12. Alternatively, read the resistance from the Bode plot [1]. Identify the resistance as the ordinate at the slope rupture [2].

2.12.1. SCREEN: 71248\_Bode\_Plot\_Pimenta.mov: 00:39-00:54

2.12.2. SCREEN: 71248\_Bode\_Plot\_Pimenta.mov: 01:41-01:48

2.13. Calculate the proton conductivity from the measured membrane resistance [1].

2.13.1. SCREEN: 71248\_Proton\_Conductivity\_Calculation\_Pimenta.mov: 06:52-07:02.

*Video editor: Emphasize the formula  $\sigma = \frac{L}{RA} = \frac{L}{RWT}$*

2.14. Generate plots of proton conductivity versus temperature using the data analysis tool [1].

2.14.1. SCREEN: 71248\_Proton\_Conductivity\_Calculation\_Pimenta.mov: 07:11-07:25

2.15. Construct an Arrhenius plot [1-TXT]. Extract the activation energy from the linear fit [2-TXT].

2.15.1. SCREEN: 71248\_Arrhenius\_Plot\_Pimenta.mov: 02:54-03:08. **TXT:  $\ln(\sigma)$  versus  $1000/T$**

2.15.2. SCREEN: 71248\_Arrhenius\_Plot\_Pimenta.mov: 06:01-06:14

**TXT:  $\ln(\sigma) = \ln(A) - \frac{E_a}{k_B T}$**

2.16. Decide whether proton transport occurs by a vehicular or by a hopping mechanism [1].

2.16.1. SCREEN: 71248\_Proton\_Transport\_Mechanism\_Pimenta.mov: 00:40-00:44, 01:00-01:07

## Results

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### 3. Results

- 3.1. The Nyquist plots for the PVA mixed-matrix membranes containing MIP-207-sulfonic acid showed a reduction of the semicircle with increasing temperature [1], indicating decreased bulk resistance [2].
  - 3.1.1. LAB MEDIA: Figure 6 A. *Video editor: Highlight all the curves.*
  - 3.1.2. LAB MEDIA: Figure 6 A.
- 3.2. Proton conductivity increased monotonically with temperature for both the pristine PVA membrane [1] and the PVA mixed-matrix membranes containing MIP-207-sulfonic acid [2].
  - 3.2.1. LAB MEDIA: Figure 6 B. *Video editor: Highlight the blue line labeled "PVA" from 30 degrees Celsius to 60 degrees Celsius.*
  - 3.2.2. LAB MEDIA: Figure 6 B. *Video editor: Highlight the red line labeled "PVA/MIP-207-SO<sub>3</sub>H" from 30 degrees Celsius to 60 degrees Celsius.*
- 3.3. The PVA mixed-matrix membranes containing MIP-207-sulfonic acid exhibited a proton conductivity of 0.74 millisiemens per centimeter [1], compared with 0.31 millisiemens per centimeter for pristine PVA [2].
  - 3.3.1. LAB MEDIA: Figure 6 B. *Video editor: Highlight the red square data point at 60 degrees Celsius on the "PVA/MIP-207-SO<sub>3</sub>H" line.*
  - 3.3.2. LAB MEDIA: Figure 6 B. *Video editor: Highlight the blue circular data point at 60 degrees Celsius on the "PVA" line.*
- 3.4. Arrhenius plots showed linear fits for both the pristine PVA membrane [1] and the PVA mixed-matrix membranes containing MIP-207-sulfonic acid membrane [2].
  - 3.4.1. LAB MEDIA: Figure 7. *Video editor: Highlight the blue trendline labeled "PVA".*
  - 3.4.2. LAB MEDIA: Figure 7. *Video editor: Highlight the red trendline labeled "PVA/MIP-207-SO<sub>3</sub>H".*
- 3.5. The PVA mixed-matrix membranes containing MIP-207-sulfonic acid exhibited an activation energy of 0.65 electronvolts [1], while pristine PVA exhibited an activation energy of 0.46 electronvolts [2].
  - 3.5.1. LAB MEDIA: Figure 7. *Video editor: Highlight the value "E<sub>a</sub> = 0.65 eV" in the legend for "PVA/MIP-207-SO<sub>3</sub>H".*

- 3.5.2. LAB MEDIA: Figure 7. *Video editor: Highlight the value “ $E_a = 0.46 \text{ eV}$ ” in the legend for “PVA”.*
- 3.6. These activation energy values indicated a predominantly vehicular proton transport mechanism for the composite membrane [1], while both vehicular and hopping mechanisms occurred in pristine PVA [2].
- 3.6.1. LAB MEDIA: Figure 7. *Video editor: Highlight the red data series and trendline labeled “PVA/MIP-207-SO<sub>3</sub>H”.*
- 3.6.2. LAB MEDIA: Figure 7. *Video editor: Highlight the blue data series and trendline labeled “PVA”.*

### **Pronunciation guides:**

1. Polyvinyl alcohol  
Pronunciation link: <https://www.merriam-webster.com/dictionary/polyvinyl%20alcohol>  
IPA: /ˌpɑːliˈvaɪnəl ˈælkəˌhɔːl/, /ˌpɑːliˈvaɪnəl ˈælkəˌhɑːl/  
Phonetic Spelling: pah·lee·VY·nuhl AL·kuh·haw
2. Membrane  
Pronunciation link: <https://www.merriam-webster.com/dictionary/membrane>  
IPA: /ˈmɛmˌbreɪn/  
Phonetic Spelling: MEM·brayn
3. Mixed-matrix  
Pronunciation link: No confirmed link found  
IPA: /ˌmɪkst ˈmɛtrɪks/  
Phonetic Spelling: mikst MAY·triks
4. Sulfonic  
Pronunciation link: <https://www.merriam-webster.com/dictionary/sulfonic>  
IPA: /sʌlˈfɑːnɪk/  
Phonetic Spelling: sul·FAH·nik
5. Micrometer  
Pronunciation link: <https://www.merriam-webster.com/dictionary/micrometer>  
IPA: /maɪˈkrɑːmɪtər/  
Phonetic Spelling: my·KRAH·muh·ter
6. Geometric  
Pronunciation link: <https://www.merriam-webster.com/dictionary/geometric>  
IPA: /ˌdʒiːəˈmɛtrɪk/  
Phonetic Spelling: jee·uh·MET·rik
7. Electrodes

Pronunciation link: <https://www.merriam-webster.com/dictionary/electrode>

IPA: /ɪˈlɛkˌtroʊd/

Phonetic Spelling: ih-LEK-troh

8. Teflon

Pronunciation link: <https://www.merriam-webster.com/dictionary/Teflon>

IPA: /ˈtɛfˌlɔːn/

Phonetic Spelling: TEF-lawn

9. Interfacial

Pronunciation link: <https://www.merriam-webster.com/dictionary/interfacial>

IPA: /ˌɪntərˈfeɪʃəl/

Phonetic Spelling: in-ter-FAY-shuhl

10. Climatic

Pronunciation link: <https://www.merriam-webster.com/dictionary/climatic>

IPA: /klaɪˈmætɪk/

Phonetic Spelling: kly-MAT-ik

11. Humidity

Pronunciation link: <https://www.merriam-webster.com/dictionary/humidity>

IPA: /hjuːˈmɪdətɪ/

Phonetic Spelling: hyoo-MID-uh-tee

12. Potentiostat

Pronunciation link: <https://www.merriam-webster.com/dictionary/potentiostat>

IPA: /pəˈtɛnʃioʊˈstæt/

Phonetic Spelling: puh-TEN-shee-oh-stat

13. Frequency

Pronunciation link: <https://www.merriam-webster.com/dictionary/frequency>

IPA: /ˈfriːkwənsi/

Phonetic Spelling: FREE-kwuhn-see

14. Electrochemical

Pronunciation link: <https://www.merriam-webster.com/dictionary/electrochemical>

IPA: /ɪˌlɛktroʊˈkɛmɪkəl/

Phonetic Spelling: ih-LEK-troh-KEM-uh-kuhl

15. Impedance

Pronunciation link: <https://www.merriam-webster.com/dictionary/impedance>

IPA: /ɪmˈpiːdəns/

Phonetic Spelling: im-PEE-duhns

16. Spectroscopy

Pronunciation link: <https://www.merriam-webster.com/dictionary/spectroscopy>

IPA: /spɛkˈtrɔːskəpi/

Phonetic Spelling: spek-TRAH-skuh-pee

17. Perturbation

Pronunciation link: <https://www.merriam-webster.com/dictionary/perturbation>

IPA: /ˌpɜːtəˈbeɪʃən/

Phonetic Spelling: per-ter-BAY-shuhn

18. Alternating

Pronunciation link: <https://www.merriam-webster.com/dictionary/alternating>

IPA: /ˈɔːltərˌnɛɪtɪŋ/

Phonetic Spelling: AWL-ter-nay-ting

19. Equilibrate

Pronunciation link: <https://www.merriam-webster.com/dictionary/equilibrate>

IPA: /ˌiːkwəˈlaɪˌbreɪt/

Phonetic Spelling: ee-kwuh-LY-brayt

20. Nyquist

Pronunciation link: <https://www.merriam-webster.com/dictionary/Nyquist>

IPA: /ˈnaɪkwɪst/

Phonetic Spelling: NYE-kwist

21. Semicircle

Pronunciation link: <https://www.merriam-webster.com/dictionary/semicircle>

IPA: /ˈsemiˌsɜːkəl/, /ˈsemaɪˌsɜːkəl/

Phonetic Spelling: SEM-ee-sur-kuhl; SEM-eye-sur-kuhl

22. Ordinate

Pronunciation link: <https://www.merriam-webster.com/dictionary/ordinate>

IPA: /ˈɔːrdənət/

Phonetic Spelling: OR-duh-nuht

23. Arrhenius

Pronunciation link: <https://www.merriam-webster.com/dictionary/Arrhenius>

IPA: /əˈriːniəs/

Phonetic Spelling: uh-REE-nee-uhs

24. Activation

Pronunciation link: <https://www.merriam-webster.com/dictionary/activation>

IPA: /ˌæktəˈveɪʃən/

Phonetic Spelling: ak-tuh-VAY-shuhn

25. Vehicular

Pronunciation link: <https://www.merriam-webster.com/dictionary/vehicular>

IPA: /viˈhɪkjələr/

Phonetic Spelling: vee-HIK-yuh-ler

26. Proton

Pronunciation link: <https://www.merriam-webster.com/dictionary/proton>

IPA: /ˈproʊˌtɑːn/

Phonetic Spelling: PROH·tahn

27. Monotonically

Pronunciation link: <https://www.merriam-webster.com/dictionary/monotonically>

IPA: /məˌnɑːtəˈnɪkli/

Phonetic Spelling: muh·nah·tuh·NIK·lee

28. Millisiemens

Pronunciation link: No confirmed link found

IPA: /ˌmɪliˈsiːmənz/

Phonetic Spelling: MIL·ee·SEE·muhnz

29. Electronvolts

Pronunciation link: <https://www.merriam-webster.com/dictionary/electronvolt>

IPA: /ɪˈlekˌtrɑːnˌvoʊlt/

Phonetic Spelling: ih·LEK·tron·vohlt

30. Composite

Pronunciation link: <https://www.merriam-webster.com/dictionary/composite>

IPA: /kəmˈpɑːzət/, /ˈkɑːmpəzɪt/

Phonetic Spelling: kuhm·PAHZ·uht; KOM·puh·zit