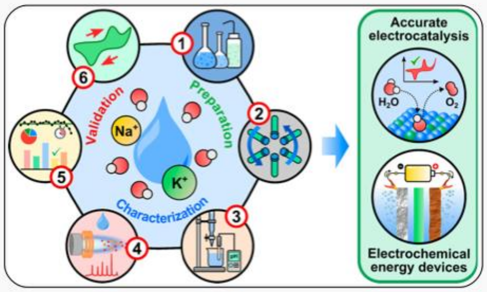
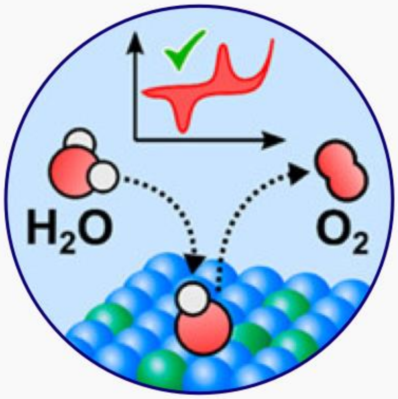
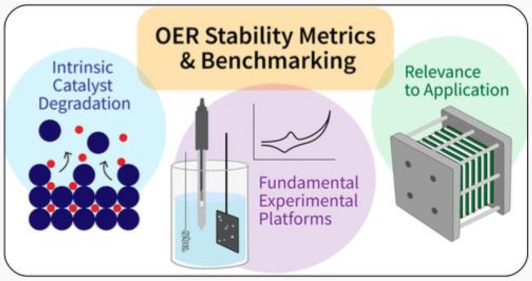
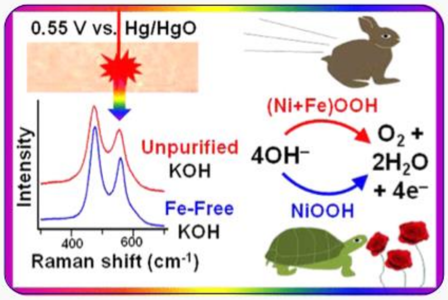
Electro Catalysis
Rigor & Reproducibility Workshop
@ UNIVERSITY *of* WASHINGTON

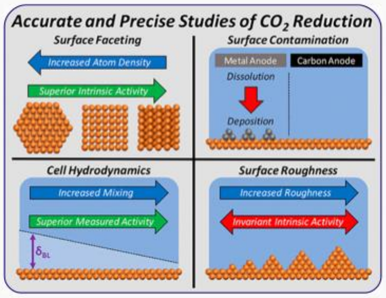
July 8 – 10, 2025











Workshop on Rigor and Reproducibility in Electrocatalysis

University of Washington
Seattle, WA USA

July 8–10, 2025

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Eric M. Stuve	University of Washington

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Ezra L. Clark	The Pennsylvania State University

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Code of Conduct

The Workshop on Rigor and Reproducibility in Electrocatalysis is a harassment-free event. We are dedicated to providing a safe and inclusive experience for all attendees. This means that the all meeting and dining spaces will be free from harassment based on, but not limited to, gender, gender identity and expression, age, sexual orientation, disability, physical appearance, body size, race, ethnicity, religion (or lack thereof), or technology choices.

Attendees will:

- Be respectful of all presenters, panelists, and of other attendees. Only ask questions during the allotted times for Q&A, breakout discussions, or other sessions where appropriate unless the speaker specifically permits.
- Present themselves professionally when interacting with others during the workshop.
- If asked, stop any harassing behavior immediately.

Attendees will not:

- Exhibit threatening or intimidating behaviors, e.g., abusive language, threats of violence, or harassment.
- Engage in verbal or written harassment during the event to disparage attendees based on race, gender, sexual orientation, physical or mental ability, or religion.
- Engage in behavior that is potentially unsafe or harmful to others.
- Engage in indecent exposure, sexual harassment, or making unwanted or inappropriate advances.
- Create disturbances with disruptive noise, e.g., loud talking or audible electronic devices.
- Vend, peddle, solicit, or petition during the workshop, including posting or distributing materials without permission.
- Inappropriately demanding the attention of other attendees who are participating in the sessions.

If you are the subject of harassment of any kind during the workshop, please let one of the workshop staff or hosts know immediately, and they will address the situation.

Observations or concerns about behavior that is potentially unsafe or harmful to self will be immediately addressed privately.

Failure to abide by any of the above or failure to comply with the instructions of a workshop organizer will result in your immediate removal from the workshop and possible further action based on the severity of the infraction.

Electro Catalysis
Rigor & Reproducibility Workshop

Workshop Welcome

July 8, 2025

Welcome to the Workshop on Rigor and Reproducibility in Electrocatalysis! This workshop builds on two earlier workshops: the Workshop on Rigor and Reproducibility in Thermal Catalysis (July 2022) and the AI for Multidisciplinary Exploration and Discovery (AIMED) workshop (October 2024). These three workshops were commissioned by Dr. Robert McCabe, Program Manager (now retired) for the Catalysis program of NSF-CBET. We are most grateful to Dr. McCabe for his foresight in bringing rigor and reproducibility to a broad range of catalysis research.

The workshop has six sessions on electrodes, electrolytes, electrochemical kinetics, and spectroscopies and operando analysis, followed by a distinguished set of keynote speakers to assimilate and provide context among the many areas affecting rigor and reproducibility. The pre-workshop survey illustrates a wide range of technical concerns, nuances, and opinions. Many thanks to those of you who responded to the survey.

We gratefully acknowledge primary support from the U.S. National Science Foundation, CBET program on Catalysis. The Clean Energy Institute of the University of Washington provided communications and logistics support for the workshop. We also thank our corporate sponsors, Hiden Analytica and easyXAFS, for supporting the dinners and poster session.

We look forward to a productive and inspiring workshop. Thank you all for attending and providing your insights!

Sincerely,

Eric, Líney, and Ezra

Workshop Overview

DAY	TIME	EVENT	LOCATION
TUESDAY JULY 8	8:00	Breakfast	Amazon Gallery
	8:40	Well-Defined Electrodes and Electrolytes	Habib Classroom
	10:45	Break-out Discussions	Habib Classroom
	12:00	Lunch & Walk	Amazon Gallery Lower Rainier Vista
	13:15	Electrochemical Kinetics	Habib Classroom
	15:15	Break-out Discussions	Habib Classroom
	16:35	Adjourn Session	
	18:00	Dinner & Poster Session	HUB 332/334
	20:30	Escorted Walk to Hotels	
WEDNESDAY JULY 9	8:00	Breakfast	Amazon Gallery
	8:45	Spectroscopies and Operando Analysis	Habib Classroom
	10:45	Break-out Discussions	Habib Classroom
	12:00	Lunch & Walk	Amazon Gallery Upper Rainier Vista
	13:20	General Session	Habib Classroom
	16:30	Adjourn Session	
	17:30	Bus to Ivar’s	Board at Gates Center
	18:00	Dinner	Ivar’s Salmon House
	20:30	Bus back to dorm/hotels	
THURSDAY JULY 10	8:00	Breakfast	Amazon Gallery
	8:45	Keynote Speakers	Habib Classroom
	11:30	Summary Discussions	Habib Classroom
	12:30	Adjourn Workshop	

Technical Program

Tuesday Morning, July 8

Well-Defined Electrodes and Electrolytes

Session Chairs

Elizabeth Corson	The University of Kansas
Brian Tackett	Purdue University

TIME	SPEAKER/EVENT	TITLE	LOCATION
8:00	Breakfast		Amazon Gallery
8:40	Eric Stuve University of Washington	Welcoming Remarks	Habib Classroom
	Anne Co The Ohio State University	Electrolyte Purity and Experimental Parameters Affecting Measurements	
9:05	Q&A		
9:15	Alexander Bagger Technical University of Denmark	Comparing Experiments & Simulations - Variations, Limitations & Challenges	
9:35	Q&A		
9:45	Kelsey Stoerzinger University of Minnesota	Defining or Understanding the Electrode: Common Approaches and Room for Improvement	
10:05	Q&A		
10:15	Coffee Break		Amazon Gallery
10:45	Break-out		Habib Classroom
11:15	Report-out		
11:35	Summary Discussion		
12:00	Lunch & Walk		Amazon Gallery Lower Rainier Vista

Tuesday Afternoon, July 8

Electrochemical Kinetics

Session Chairs	
Jason Adams	Rice University
Robert Warburton	Case Western Reserve University

TIME	SPEAKER/EVENT	TITLE	LOCATION
13:15	Marcel Schreier University of Wisconsin-Madison	Electrocatalysis and monolayer studies - What are we really measuring?	Habib Classroom
13:35	Q&A		
13:45	Ezra Clark Pennsylvania State University	Monitoring Electrolyte Impurities for CO_2 Reduction and Measurement of Reaction Products with DEMS	
14:05	Q&A		
14:15	Elizabeth J. Biddinger City College of New York, CUNY	Closing the Mass Balance in Electro-Organic Reactions	
14:35	Q&A		
14:45	Break		Amazon Gallery
15:15	Break-out		Habib Classroom
15:45	Report-out		
16:05	Summary Discussion		
16:35	Adjourn Session		
18:00	Dinner & Poster Session		HUB 332/334

Tuesday Evening, July 8

Poster Session

TIME	SPEAKER/EVENT	TITLE	LOCATION
18:00	Elizabeth Corson The University of Kansas	Measuring Interfacial pH during Electrocatalysis using ATR‚ÄìSEIRAS	HUB 332/334
18:00	Guoxiang (Emma) Hu Georgia Institute of Technology	Computational Design of Electrocatalysts: The Critical Role of Operando Structures	
18:00	Qiaowan Chang Washington State University	Mechanistic Understanding of MOF Electrocatalytic Stability via In-situ Characterization	
18:00	Elizabeth J. Biddinger City College of New York, CUNY	Electric Field & Current Distribution Effects in Reactor Design: A CO_2 Electroreduction Case Study	
18:00	Dohyung Kim University of Pennsylvania	Non-uniform Enhancement in CO_2 Electroreduction by Hydrophobic Additives	
18:00	Ezra L. Clark The Pennsylvania State University	Electrochemical H_2 Pumping Enhances the Activity of Selective Semi-Hydrogenation Catalysts	
18:00	Adam Hoffman SLAC National Accelerator Laboratory	Potpourri of Interesting Electrochemical Results Observed with In-situ XAS	
18:00	Jason S. Bates University of Virginia	Influence of Organocations on the Kinetics and Mechanism of Electrocatalytic Hydrogen Evolution in Alkaline Media	

Wednesday Morning, July 9

Spectroscopies and Operando Analysis

Session Chairs

Jason Bates	University of Virginia
Haotian Wang	Rice University

TIME	SPEAKER/EVENT	TITLE	LOCATION
8:00	Breakfast		Amazon Gallery
8:45	Jingguang Chen Columbia University	Dynamic Changes of Oxygen Evolution and CO_2 Reduction Catalysts Studied with Synchrotron XRD and X-ray Absorption Spectroscopy (XANES and EXAFS)	Habib Classroom
8:35	Q&A		
9:15	Matthias Waegele Boston College	Pitfalls in the Practice of Surface-Enhanced IR Absorption Spectroscopy	
9:35	Q&A		
9:45	Linsey Seitz Northwestern University	Considerations for Spectroscopy and Electrochemistry Techniques to Characterize Dynamic Electrocatalyst Materials	
10:05	Q&A		
10:15	Break		Amazon Gallery
10:45	Break-out		Habib Classroom
11:15	Report-out		
11:35	Summary Discussion		
12:00	Lunch & Walk		Amazon Gallery Upper Rainier Vista

Wednesday Afternoon, July 9

General Session

Session Chairs

Kasun Gunasooriya	The University of Oklahoma
Guoxiang (Emma) Hu	Georgia Institute of Technology

TIME	SPEAKER/EVENT	TITLE	LOCATION
13:20	Adam Holewinski University of Colorado Boulder	Understanding the Interplay of Bifunctional and Electronic Effects: Kinetic Fingerprints of C_1 Electro-Oxidation on Bimetallics	Habib Classroom
13:35	Q&A		
13:40	Nirala Singh University of Michigan	Electrocatalyst and Reactor Control to Understand Selectivity for Series and Parallel Reactions	
13:55	Q&A		
14:00	Joaquin Resasco University of Texas at Austin	Smoke and Mirrors in Electrocatalysis	
14:15	Q&A		
14:20	Raul Marquez University of Texas at Austin	Getting the Basics Right: Electrolyte Essentials for Electrocatalysis	
14:35	Q&A		
14:40	Break		Amazon Gallery
15:10	William Tarpeh Stanford University	Dynamic Electrolytes and Electrodes during Wastewater Nitrate Reduction	Habib Classroom
15:25	Q&A		
15:30	Juliana S. A. Carneiro Columbia University	Electrocatalysis in Solid Oxide Fuel Cells and Electrolyzers	
15:45	Q&A		

TIME	SPEAKER/EVENT	TITLE	LOCATION
15:50	Carlos Morales-Guio University of California Los Angeles	Decoupling Mass, Heat, and Charge Transport in Electrocatalysis: A Gastight RCE Platform for Determining Intrinsic Kinetics and Constructing Multi-scale Transport-Reaction Models	
16:05	Q&A		
16:10	A. Shoji Hall University of Pennsylvania	Promoting CO_2 Reduction by Tuning the Interfacial Properties of H_2O	
16:25	Q&A		
16:30	Adjourn Session		
18:00	Dinner		Ivar's Salmon House

Thursday Morning, July 10

Keynote Speakers

Session Chairs

Líney Árnadóttir	Oregon State University
Ezra L. Clark	The Pennsylvania State University
Eric M. Stuve	University of Washington

TIME	SPEAKER/EVENT	TITLE	LOCATION
8:00	Breakfast		Amazon Gallery
8:45	Peter Strasser Technical University Berlin	Mistakes that Matter: Toward Accuracy and Reproducibility in Electrocatalyst Research	Habib Classroom
9:15	Q&A		
9:30	Michael Janik Pennsylvania State University	Best Practices in Electrocatalysis DFT	
10:00	Q&A		
10:15	Break		Amazon Gallery
10:45	Alexis T. Bell University of California Berkeley	Rigor and Reproducibility in Studies of the Electrochemical Reduction of CO_2	Habib Classroom
11:15	Q&A		
11:30	Summary & Discussion		
12:30	Adjourn Workshop		

Abstracts

Well-Defined Electrodes and Electrolytes

Electrolyte Purity and Experimental Parameters Affecting Measurements

Anne Co
The Ohio State University

Abstract:

While electrochemical measurements are relatively straightforward to perform, obtaining reliable and reproducible data requires meticulous attention to experimental detail. The inherent sensitivity of electrochemical techniques, though advantageous for detecting subtle changes, also makes them highly susceptible to variability arising from factors such as electrode geometry and positioning, cell configuration, surface conditioning of the electrodes, electrolyte composition and purity, and environmental conditions. This presentation will focus on methods for determining the minimum electrolyte purity, as well as the importance of controlling and reporting electrolysis conditions to ensure reproducible activity measurements, enabling reliable mechanistic interpretation in CO_2 reduction (CO_2R) studies.

R&R

Comparing Experiments & Simulations - Variations, Limitations & Challenges

Alexander Bagger
Technical University of Denmark
alexbag@dtu.dk

Abstract:

I will discuss how experiments and computational simulations can support each other to develop fundamental insights for electrochemical reactions. I will focus on the special requirements to make a successful and fair comparison, while highlighting limitations and challenges. I will use the following systems in my discussion:

i) Predicting experimental product distribution with simulations in the electrochemical reactions[1-7].

ii) Predicting experimental cyclic voltammetry with simulations across materials and electrolytes reactions[8-10].

With the main goal to discuss minimalistic models that do not overfit or over interpretating experimental data.

[1] Hori et. al., J. Chem. Soc., Faraday Trans., **1989**.
[2] Bagger et. al., *ChemPhysChem*, **2017**.
[3] Albrechtsen, Bagger, *Current Opinion in Electrochemistry*, **2025.**
[4] Tsuneto et al., *JEAC*. **1994**.
[5] Bagger et. al., *ACS Energy Letters*. **2024**.
[6] Shibata et. al., *J. Electrochem. Soc.* **1998**.
[7] Wuttke, Bagger, *Commun. Chem.* **2025**.
[8] Bagger et. al, *ChemPhysChem*, **2019**.
[9] Rossmeisl, Jensen, Petersen, Arnarson, Bagger et. al., The Journal of Physical Chemistry C, **2020.**
[10] Sebastián-Pascual, Petersen, Bagger et al., *ACS Catalysis.* **2021**.

——————— R&R ———————

Defining or understanding the electrode: common approaches and room for improvement

Kelsey Stoerzinger
University of Minnesota

Abstract:

Some approaches to electrocatalyst fabrication (often referred to as a surface science or bottom-up approach) simplify the catalyst surface to a uniform environment, enabling new approaches for quantitative characterization and comparison. However, many reactions--and indeed applied systems--are far from this simplicity, necessitating characterization of both electrode complexity and, perhaps, average state. I will discuss some approaches typically used and highlight the fundamental assumptions they make (and therefore their inherent limitations).

——————— R&R ———————

Electrochemical Kinetics

Electrocatalysis and monolayer studies - What are we really measuring?

Marcel Schreier
University of Wisconsin-Madison

Abstract:

In this presentation, I will discuss our methods to control electrocatalytic processes in real-time by coupling sensitive electrochemical mass spectrometric analysis with the application of time-dependent potentials. I will discuss challenges encountered in these measurements and I will also discuss the question as to what the data resulting from any electrocatalytic measurement really means in practice.

——— R&R ———

Avoiding Common Pitfalls in the Pursuit of Meaningful Electrocatalytic Activity Data

Ezra L. Clark
The Pennsylvania State University

Abstract:

Several common experimental pitfalls that negatively impact the acquisition of intrinsic electrocatalytic activity data will be discussed through a series of short vignettes. These short vignettes will utilize studies of CO_2 and CO reduction over a variety of different electrocatalysts in both H-cells and zero-gap reactors to demonstrate the impact of these often-overlooked variables. First, the importance of quantifying electrochemical cell hydrodynamics and distinguishing between intrinsic activity measurements and those significantly influenced by mass transfer will be discussed.[1,2] Second, the importance of rigorously validating electrocatalyst purity and avoiding common sources of contamination will be discussed.[1] Third, a common error in the quantification of CO_2 reduction product generation rates from high current zero-gap devices will be discussed.[3–5] Fourth, the importance of immersing nanoparticle-based electrocatalysts under potential control will be discussed.[6] Fifth, the importance of rigorously quantifying the uncompensated resistance using a variety of complementary methods will be discussed.

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(1) Clark, E. L.; Resasco, J.; Landers, A.; Lin, J.; Chung, L.-T.; Walton, A.; Hahn, C.; Jaramillo, T. F.; Bell, A. T. Standards and Protocols for Data Acquisition and Reporting for Studies of the Electrochemical Reduction of Carbon Dioxide. *ACS Catal.* **2018**, *8* (7), 6560–6570.

(2) Lobaccaro, P.; Singh, M. R.; Clark, E. L.; Kwon, Y.; Bell, A. T.; Joel W. Ager, I. Effects of Temperature and Gas–Liquid Mass Transfer on the Operation of Small Electrochemical Cells for the Quantitative Evaluation of CO_2 Reduction Electrocatalysts. *Phys. Chem. Chem. Phys.* **2016**, *18*, 26777–26785.

(3) Larrazábal, G. O.; Strøm-Hansen, P.; Heli, J. P.; Zeiter, K.; Therkildsen, K. T.; Chorkendorff, I.; Seger, B. Analysis of Mass Flows and Membrane Cross-over in CO_2 Reduction at High Current Densities in an MEA-Type Electrolyzer. *ACS Appl. Mater. Interfaces* **2019**, *11* (44), 41281–41288.

(4) Ma, M.; Clark, E. L.; Therkildsen, K. T.; Dalsgaard, S.; Chorkendorff, I.; Seger, B. Insights into the Carbon Balance for CO_2 Electroreduction on Cu Using Gas Diffusion Electrode Reactor Designs. *Energy Environ. Sci.* **2020**, *13* (3), 977–985.

(5) Larrazábal, G. O.; Ma, M.; Seger, B. A Comprehensive Approach to Investigate CO_2 Reduction Electrocatalysts at High Current Densities. *Accounts Mater. Res.* **2021**, *2* (4), 220–229.

(6) Hochfilzer, D.; Sørensen, J. E.; Clark, E. L.; Scott, S. B.; Chorkendorff, I.; Kibsgaard, J. The Importance of Potential Control for Accurate Studies of Electrochemical CO Reduction. *ACS Energy Lett.* **2021**, *6* (5), 1879–1885.

——————— R&R ———————

Closing the Mass Balance in Electro-Organic Reactions

Elizabeth J. Biddinger
City College of New York, CUNY

Abstract:

In order to gain a full perspective on reaction performance, it is imperative that the reactants and products are fully accounted for in electro-organic reactions. This is particularly challenging due to many possible losses of the reaction species beyond the electrochemical reaction – homogeneous side reactions, evaporation, precipitation, polymerization, and membrane crossover are all contributions to the losses. In this work, best practices for capturing reaction species, accounting for cross over and incorporating into mass balances will be presented. Examples used will be for: 1) furfural electroreduction in acid – a particularly challenging reaction system that has fouling, polymerization and evaporation of products; and 2) benzyl alcohol dehydrogenation – a reaction that has significant membrane crossover challenges.

——— R&R ———

Poster Session

Measuring Interfacial pH during Electrocatalysis using ATR–SEIRAS

Elizabeth Corson
The University of Kansas

Abstract:

The interfacial pH during electrocatalysis can be significantly different from the bulk pH due to the consumption or generation of protons. For example, protons are consumed during the carbon dioxide reduction reaction (CO2RR), nitrate reduction reaction (NO3RR), and hydrogen evolution reaction (HER) and protons are generated in the oxygen evolution reaction (OER). While a pH probe can measure the bulk pH, it cannot detect the pH gradient that forms at the electrode–electrolyte interface. Directly measuring the interfacial pH will lead to a better understanding of the reaction conditions and how to control them through electrolyte, reactor, or electrochemical engineering strategies.

We used in situ attenuated total reflectance–surface-enhanced infrared adsorption spectroscopy (ATR–SEIRAS) to quantitatively measure the interfacial pH within 5–10 nm of the electrode surface during electrocatalysis. A 0.5 M phosphate buffer was used to measure the local pH through the ratio of peak areas from the two dominant phosphate anions in each pH range. Three calibration ranges were established: pH 1–5, pH 5–9, and pH 9–13. We studied the repeatability of the technique and performed electrochemical nitrate reduction as a model reaction to observe the time evolution of the interfacial pH and the impact of pulsed electrolysis on reducing the pH gradient. By correlating the ATR–SEIRAS results with electrochemical experiments measuring NO3RR selectivity, activity, and efficiency, we can relate bulk electrolyte properties with interfacial properties, and interfacial properties with electrocatalytic performance. We demonstrate how this correlation can lead to engineering strategies to optimize ammonia production in electrochemical NO3RR and discuss how the technique could be applied to other electrocatalytic reactions.

———— R&R ————

Computational Design of Electrocatalysts: The Critical Role of Operando Structures

Guoxiang (Emma) Hu
Georgia Institute of Technology

Abstract:

Understanding electrocatalyst performance requires detailed insight into active structures under operating conditions. We use advanced computational methods, validated against experimental observations, to investigate the dynamic evolution of catalyst surfaces during electrochemical reactions. Case studies on metal nanoclusters, dual-atom catalysts, and amorphous transition metal dichalcogenides reveal that operando structures deviate significantly from static models, profoundly impacting catalytic activity and selectivity. These results highlight the essential role of operando perspectives in the computational design of next-generation electrocatalysts.

——————— R&R ———————

Mechanistic Understanding of MOF Electrocatalytic Stability via In-situ Characterization

Qiaowan Chang
Washington State University

Abstract:

Metal–organic frameworks (MOFs) have emerged as promising candidates in electrocatalysis, particularly for the electrochemical reduction of carbon dioxide (CO2RR). Despite advances in enhancing catalytic activity and selectivity, the long-term electrocatalytic stability of MOFs remains insufficiently understood. In this study, we examine the stability and structural evolution of Zn-ZIF-8, a prototypical MOF catalyst, under CO2RR conditions. We observed that stability is strongly dependent on the applied electrochemical potentials. To elucidate degradation mechanisms, we conducted *in-situ* X-ray absorption near-edge structure (XANES) and *in-situ* X-ray diffraction (XRD) analyses. *In-situ* XANES revealed evolutions in the Zn–N4 coordination environment of the active sites, while *in-situ* XRD indicated bulk transformations in the long-range crystalline structure of Zn-ZIF-8 during electrolysis.

——————— R&R ———————

Electric Field & Current Distribution Effects in Reactor Design: A CO_2 Electroreduction Case Study

Elizabeth J. Biddinger
City College of New York, CUNY

Abstract:

Electric fields influence reaction performance across all scales – from the nanoscale to the macroscale. The reactor design has a significant influence on the macroscale electric fields and current distributions. This in turn, impacts the current distribution, speciation and local pH in the reaction, thereby influencing the overall reaction performance. In this presentation, a case study of using model electrode systems made of the same electrode materials and geometric areas was used in CO_2 electroreduction reactions. Depending upon the electrode shape, the partial current densities and reaction selectivities changed significantly. Experimental results and COMSOL simulations were used to demonstrate the impact of electric fields and current density distributions on the reaction performance.

——— R&R ———

Non-uniform Enhancement in CO_2 Electroreduction by Hydrophobic Additives

Dohyung Kim
University of Pennsylvania

Abstract:

We demonstrate that the enhancement effects of hydrophobic additives are highly confined to undercoordinated metal sites. Advanced surface characterization, combining XPS, ToF-SIMS, and LEIS, coupled with electrochemical analysis, reveal that cetyltrimethylammonium enhances the CO_2-to-CO turnover frequency of undercoordinated sites by nearly two orders of magnitude, while the rest of the other sites exhibit only a 2-fold increase. Since the undercoordinated sites constitute only a small fraction of the total surface, nanomolar concentrations of impurity metals in solution can completely nullify the overall enhancement effects. These findings highlight the significant site-dependent nature of environmental adjustments near catalytic surfaces and the need for a deeper understanding of the interplay between surfaces and near-surface environments to advance electrocatalytic systems.

——— R&R ———

Electrochemical H_2 Pumping Enhances the Activity of Selective Semi-Hydrogenation Catalysts

Michael Lemelin and Ezra L. Clark
The Pennsylvania State University

Abstract:

Semi-hydrogenation catalysts typically suffer from either poor activity or poor selectivity. Noble metals exhibit high selectivity for the semi-hydrogenated product but also exhibit low rates of reaction due to their sluggish H_2 activation kinetics [1]. To remedy this issue, noble metals are typically promoted with small amounts of a more reactive metal, which is dispersed over the noble metal surface as single isolated atoms. These isolated atoms catalyze H_2 dissociation, which spills over to the neighboring noble metal sites [2]. Unfortunately, these catalysts often exhibit poor stability due to the tendency of the isolated reactive metal atoms to coalesce over time. Thus, there is a need to develop alternative methods for promoting the activity of selective semi-hydrogenation catalysts. Electrochemical H_2 pumps (EHPs) are an emerging technology for on demand H_2 purification [3]. They operate by selectively oxidizing the H_2 contained in the feed gas to protons and electrons over a Pt-based anode electrocatalyst. The protons are transported through a proton exchange membrane to the cathode, where they are reduced to form a pure H_2 stream. Semi-hydrogenation reactions can be performed in an EHP by simply feeding the alkyne of interest into the cathode chamber. The potential advantage of this approach is that the surface H* required for semi-hydrogenation is supplied directly to the catalyst surface via electrochemical pumping, circumventing H_2 activation over the semi-hydrogenation catalyst [4]. This presentation demonstrates that electrochemical H_2 pumping can enhance the rate of selective acetylene semi-hydrogenation over catalysts that are kinetically limited by H_2 activation.

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4. Otsuka, K.; and Yagi, T. *J. Catal.*, 145, 2 (1994).

——— R&R ———

Potpourri of Interesting Electrochemical Results Observed with In-situ XAS

Adam S. Hoffman
SLAC National Accelerator Laboratory

Abstract:

This poster looks at three different in-situ XAS experiments centered around the question of metal site accessibility and its dynamics under electrocatalytic reactions.

——— R&R ———

Influence of Organocations on the Kinetics and Mechanism of Electrocatalytic Hydrogen Evolution in Alkaline Media

Jason S. Bates
University of Virginia

Abstract:

Organocations strongly influence alkaline HER rates, yet the mechanistic basis for this effect remains unclear. This study combines turnover rate measurements, mechanism-derived rate expressions, and the coverage-dependence of Tafel slopes to discriminate among candidate HER mechanisms. Our analysis indicates that the addition of tetrapropylammonium cations to a supporting sodium perchlorate electrolyte (at constant pH = 12) systematically increases the rate constant for the kinetically relevant Heyrovsky step on Pt without altering surface hydrogen coverages. Work is ongoing to rigorously differentiate among candidate hypotheses to explain the chemical origins of these measurements.

——— R&R ———

Spectroscopies and Operando Analysis

Dynamic Changes of Oxygen Evolution and CO_2 Reduction Catalysts Studied with Synchrotron XRD and X-ray Absorption Spectroscopy (XANES and EXAFS)

Jingguang Chen
Columbia University

Abstract:

——— R&R ———

Pitfalls in the Practice of Surface-Enhanced IR Absorption Spectroscopy

Matthais Waegele
Boston College

Abstract:

Surface-enhanced infrared absorption spectroscopy (SEIRAS) is a highly versatile tool for investigating electrocatalytic interfaces during electrocatalysis. SEIRAS is relatively straightforward to implement, and the required instrumentation is widely available. As such, SEIRAS has become the method of choice for probing a broad range of interfacial species, processes, and properties, including reaction intermediates, double layer fields, local pH, and solvent structure. Although it is relatively straightforward to collect SEIRA spectra, it is also easy to misinterpret them or inadvertently record artifactual data. In this talk, I will discuss challenges in the practice of SEIRAS, with a focus on interfacial solvent structure, electric double layer fields, and reaction intermediates. I will propose strategies for avoiding common pitfalls in experimental design and interpretation of SEIRA spectra.

——— R&R ———

Considerations for Spectroscopy and Electrochemistry Techniques to Characterize Dynamic Electrocatalyst Materials

Linsey Seitz
Northwestern University

Abstract:

I will discuss some of our in situ and ex situ synchrotron x-ray based techniques for catalyst characterization and types of degradation; where things can and have gone wrong; and recent developments in surface site quantification for iridium-based catalysts using Hg UPD.

——— R&R ———

Understanding the Interplay of Bifunctional and Electronic Effects: Kinetic Fingerprints of C_1 Electro-Oxidation on Bimetallics

Adam Holewinski
University of Colorado Boulder

Abstract:

Electro-oxidations of small organic molecules are some of the most widely studied processes in electrocatalysis for both fundamental and technological reasons. Many reactions relevant to wastewater treatment, sensing, distributed-scale chemical synthesis, and a variety of direct-organic fuel cells all converge in a network of interconnected C_1 oxidation steps. Bottlenecks are often identified from pathways that culminate in formation of CO* strongly bound to electrode surfaces. Thus, there is particular interest in developing CO-tolerant materials to improve the efficiency of organic-converting fuel cells, electrolyzers, and other related devices.

A number of bimetallics, (e.g. Pt_xRu_{1-x}, Ag_xPd_{1-x}) show enhanced oxidation activity relative to their constituent metals, and the effects are often interpreted in terms of "bifunctional" mechanisms, in which one element activates carbon-containing species and the other serves to introduce OH* (from water) to the surface to facilitate C-O coupling. This talk will discuss kinetic signatures of bifunctional mechanisms and show a series of experimental investigations on bimetallic alloy electro-oxidation catalysts across a range of conditions to assess the extent to which enhancements may be attributed to bifunctional vs. electronic effects. Consequences for catalyst design will further be discussed.

R&R

Electrocatalyst and Reactor Control to Understand Selectivity for Series and Parallel Reactions

Nirala Singh
University of Michigan

Abstract:

Particular topics in my group have focused on the electrocatalytic reduction of nitrate, carbon dioxide, and oxygenated organic molecules. Under these reducing conditions there is a need to understand the active state of the electrocatalyst (e.g., oxidation state, whether restructuring occurs). One route to address this is post-mortem characterization or spectroscopy during operating conditions. An additional challenge for reactions with selectivity questions, particularly where products are in series, is controlling the reactor to enable fair comparison from test to test. We give a few examples where selectivity on identical catalysts differs depending on the reactor design, potentially leading to incorrect conclusions.

——— R&R ———

Smoke and Mirrors in Electrocatalysis

Joaquin Resasco
University of Texas at Austin

Abstract:

Measuring intrinsic activity is essential for the fundamental understanding and design of electrocatalyst materials. Catalytic rates must be determined under conditions where surface kinetics—rather than mass transport—govern performance and must be normalized by active-site count to yield meaningful turnover frequencies. Neglecting variations in active-site density has led to widespread misunderstanding of the intrinsic activity of common electrocatalytic materials in key reactions. Mass-transport limitations have further distorted observed performance by altering local reactant and product concentrations in many systems. In this talk, we will discuss strategies to avoid these pitfalls, using recent results from our group as illustrative examples.

——— R&R ———

Getting the Basics Right: Electrolyte Essentials for Electrocatalysis

Raul A. Marquez
University of Texas at Austin

Abstract:

The electrolyte is a critical component of any electrochemical system, shaping the properties of the electrode interface and influencing electrocatalytic performance. In this talk, I will review key experimental guidelines and protocols for electrolyte use, with a focus on reproducible electrolyte preparation, impurity control, purification methods, and standardization practices. Experimental considerations for the correct handling of ion-conducting polymer electrolytes will also be discussed. Collectively, the guidelines presented herein aim to make electrolyte use in electrocatalysis simple and reliable.

——— R&R ———

Dynamic Electrolytes and Electrodes during Wastewater Nitrate Reduction

William Tarpeh
Stanford University

Abstract:

During electrochemical nitrate reduction, both sides of the electrocatalyst-electrolyte interface experience dynamic behavior. This talk will describe ex situ and in situ synchrotron characterization techniques for titanium electrocatalysts, including hydride formation and transport. It will also describe how the electrolyte changes with time, including bulk and interfacial pH and ion composition. Together, these efforts help characterize the dynamic reaction microenvironment for nitrate reduction and inspire rigorous comparisons across electrocatalysts and electrolyte environments to appropriately attribute performance changes and rational design to each side of the electrocatalyst-electrolyte interface.

——— R&R ———

Electrocatalysis in Solid Oxide Fuel Cells and Electrolyzers

Juliana S. A. Carneiro
Columbia University

Abstract:

The growing interest in energy-to-X and X-to-energy technologies (where X includes green hydrogen, carbon-based fuels, and ammonia) has accelerated research in electrochemical conversion and storage. Solid oxide electrochemical cells (SOCs) are among the most promising platforms for these applications. However, current electrocatalytic materials at SOC electrodes are not yet optimized for reversible operation and fuel flexibility. This talk will explore SOC systems, assess electrocatalyst performance across different reactions, and outline key guidelines for evaluating electrode materials.

——— R&R ———

Decoupling mass, heat, and charge transport in electrocatalysis: A gastight RCE platform for determining intrinsic kinetics and constructing multi-scale transport-reaction models

Carlos Morales-Guio
University of California Los Angeles

Abstract:

The rational design and scale-up of CO_2 and CO electroreduction systems require the decoupling of intrinsic electrocatalytic kinetics from transport phenomena. In this talk, I will present our lab's development of a gastight rotating cylinder electrode (RCE) platform engineered to quantitatively probe transport-limited regimes in electrochemical reactions. Our approach enables the systematic tuning of mass, heat, and charge transfer under well-defined hydrodynamics near polycrystalline copper electrodes, allowing the extraction of intrinsic kinetics through the analysis of the residence times of intermediates near the catalyst surface.

We introduce a continuously stirred tank reactor (CSTR) approximation that facilitates the extraction of reaction kinetics from reactor kinetics. This framework reveals how mesoscale transport, and not simply catalyst structure, critically dictates product selectivity in CO_2 electroreduction on transition metal catalysts. The separation of reaction kinetics from reactor kinetics addresses directly the challenges in rigor and reproducibility in electrocatalytic analysis as it explains why the same catalysts can give different product distributions when tested in different electrochemical cells or under different transport conditions. The RCE platform allows

the systematic discussion of current distributions on electrode surfaces and the quantification of how these affect the extraction of Tafel slopes typically used for the discussion of fundamental mechanisms in electrocatalysis.

Finally, this talk will discuss how the integration of CFD simulations and automated data collection supports high-throughput parameterization for kinetic modeling that enables the construction of predictive multi-scale transport-reaction models for reactor scale-up. These insights are directly applicable to the broader challenges of designing reactors for electrified chemical manufacturing with well-defined and scalable transport characteristics.

——— R&R ———

Promoting CO_2 reduction by tuning the interfacial properties of H_2O

A. Shoji Hall
University of Pennsylvania

Abstract:

The electrified metal-solution interface is a critical component in electrochemical devices, enabling the efficient and sustainable production of fuels and synthetic precursors through electrolysis and energy generation in fuel cells. Central to the interface's microenvironment is water, which plays a crucial role in modulating interfacial characteristics. Water not only solvates ions, thereby influencing their adsorption at the electrode, but also interacts with reaction intermediates through hydrogen bonding. Moreover, it influences the accessibility of active sites and acts as a source of protons or oxygen via water decomposition. The characteristics of the interfacial microenvironment are dependent on the complex interplay among active sites, electrolyte components, substrates, products, and catalytic intermediates. Despite advancements in electrolyte design, our understanding of how interfacial water affects electrochemical reactivity remains limited. This seminar will explore how the manipulation of the interfacial water properties controls electrochemical reactivity, with a particular focus on C-C coupling from the reduction of CO_2 or CO. This strategy seeks to fill the knowledge gap in designing interfacial microenvironments, potentially revolutionizing renewable energy technologies.

——— R&R ———

Keynote Speakers

Mistakes that Matter: Toward Accuracy and Reproducibility in Electrocatalyst Research

Peter Strasser
Technische Universität Berlin

Abstract:

I will discuss case studies and examples of what to look for and what to be aware of to improve reproducibility and accuracy. I will include examples from the workshop or examples from my lab, independent of reaction process or catalyst type.

——— R&R ———

Best Practices in Electrocatalysis DFT

Michael Janik
The Pennsylvania State University

Abstract:

Density functional theory (DFT) has become an essential tool to examine the bond breaking and forming elementary steps that occur at heterogeneous catalyst surfaces. Electrocatalysis brings additional challenges in representing the reaction environment at an electrode-electrolyte interface, as well as the involvement of electron and ion transfer. This talk will overview the various DFT model construction strategies used to approach this interface, and discuss best practices in performing DFT calculations, reporting and discussing the imprecision and assumptions made as the work is being done, and in partnering with experimental researchers to productively partner with experimentalists in studying electrocatalysis.

——— R&R ———

Rigor and Reproducibility in Studies of the Electrochemical Reduction of CO_2

Alexis T. Bell
University of California Berkeley
Lawrence Berkeley National Laboratory

Abstract:

This talk will address two major themes. The first is what is required to obtain reproducible experimental measurement of total and product polarization curve in studies of the electrochemical reduction of CO_2 (CO_2R) using planar electrode cells containing either stagnant or flowing electrolyte and how such results can be used to determine the intrinsic kinetics for individual products. We will show that to obtain reproducible results requires avoiding contamination of the cathode by metal from the anode and metallic impurities in the electrolytes and accounting for the electrochemical area of the catalyst. It will also be shown that extraction of intrinsic kinetics from measured product polarization curves necessitates accounting for the effects of mass transfer and buffer reactions, as well as the influence of cation composition on the strength of the electrostatic field in the electrical double layer. The second theme discussed will be interpretation of CO_2R occurring in an MEA. We will examine, in particular, whether the kinetics for specific products obtained from planar electrode cells can be used to describe the kinetics of reactions occurring in the catalyst layer of an MEA, where the catalyst is covered by a thin layer of ionomer. We will see that in this case, the composition and state of hydration of the ionomer influence the kinetics occurring at the catalyst surface.

——— R&R ———

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Lower box:

Upper left and circle:

R. A. Márquez, K. Kawashima, Y. J. Son, G. Castelino, N. Miller, L. A. Smith, C. E. Chukwuneke, and C. B. Mullins, “Getting the Basics Right: Preparing Alkaline Electrolytes for Electrochemical Applications,” ACS Energy Letters 2023, 8, 1141–1146. https://doi.org/10.1021/acsenergylett.2c02847

Middle left:

J. Edgington and L. C. Seitz, “Advancing the Rigor and Reproducibility of Electrocatalyst Stability Benchmarking and Intrinsic Material Degradation Analysis for Water Oxidation,” ACS Catalysis 2023 13, 3379–3394. https://doi.org/10.1021/acscatal.2c06282

Lower left:

S. Klaus, Y. Cai M. W. Louie, L. Trotochaud, A. T. Bell, “Effects of Fe Electrolyte Impurities on Ni(OH)$_2$/NiOOH Structure and Oxygen Evolution Activity,” Journal of Physical Chemistry C, 2015, 119, 7243–7254. http://dx.doi.org/10.1021/acs.jpcc.5b00105

Lower right:

E. L. Clark, J. Resasco, A. Landers, J. Lin, L. T. Chung, A. Walton, C. Hahn, T. F. Jaramillo, and A. T. Bell, “Standards and Protocols for Data Acquisition and Reporting for Studies of the Electrochemical Reduction of Carbon Dioxide,” ACS Catalysis 2018, 8, 6560–6570. https://doi.org/10.1021/acscatal.8b01340

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