Meeting the Need: Formal Electrochemistry Training through Workshops

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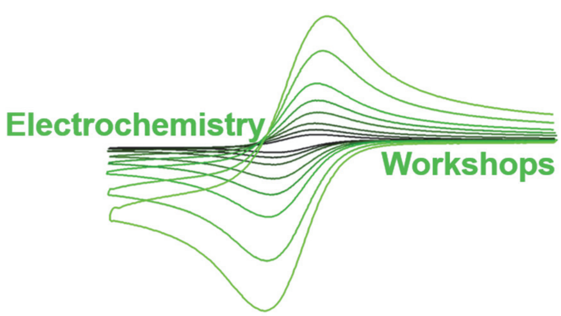
ABSTRACT

A need for enhanced access to formal electrochemistry training has led to the launch of several standalone workshops, including Cyclic Voltammetry Boot Camp. In this article, we describe the history of Cyclic Voltammetry Boot Camp, its scope and mission, the structure and content covered during the three-day workshop, and the impact the workshop has had on participants. The workshop is also contextualized through a brief summary of related workshops.

GRAPHICAL ABSTRACT



KEYWORDS

Upper-Division Undergraduate, Graduate Education, Continuing Education, Analytical Chemistry, Interdisciplinary, Hands-on learning, Electrochemistry

Across subdisciplines, chemists are turning to electrochemistry to push the frontiers of science. For instance, organic chemistry is witnessing a revival of electrosynthesis,[1–3] inorganic chemists are developing catalysts to mediate electrochemical fuel-producing reactions with unprecedented efficiencies,[4–7] and materials chemists are advancing electrochemical energy storage strategies.[8–11] However, despite underpinning innovation in energy science, catalysis, and point-of-care diagnostics, many scientists lack the formal training necessary to fully exploit the power of electrochemistry to advance their work.[12–14] Beyond stymying research and scientific advances, shortcomings in chemistry curricula have led to a major dearth of scientists with formal training in electrochemistry, causing a human resource crisis in rapidly growing industries such as electrochemical energy storage and wearable diagnostic devices.

A 2019 National Academies of Science workshop on “Advances, Challenges, and Long-Term Opportunities in Electrochemistry: Addressing Societal Needs” articulated the need to educate the next generation of electrochemists, calling for better integration of electrochemistry into existing higher education curriculums with a forward-looking focus and an emphasis on practical applications.[12] Improved rigor and consistency in the teaching of electrochemistry is widely recognized as necessary for preparing students for important and emerging applications of electrochemistry in research and industry.[12,13] While scientists call for a complete reinvention and reinvigoration of electrochemistry education across the undergraduate and graduate chemistry curriculums, a complete overhaul requires systemic change and will likely be on the years-to-decades timescale. In the interim, pedagogical tools like open-source curricula, virtual short-courses, and in-person workshops with hands-on laboratory experiences can help provide a stopgap measure to provide rigorous electrochemistry training to a diverse group of scientists.

Recognizing the need for immediate formal, hands-on training in electrochemistry, specifically cyclic voltammetry, we launched a three-day workshop titled “Cyclic Voltammetry Boot Camp” that combines lectures and laboratory experiences in 2019. In this perspective, we detail the history and format of the course, comment on its impact, and summarize related workshops.

HISTORY

In 2018, the concept of Cyclic Voltammetry Boot Camp (CVBC) was conceived in conversations between Frank Dalton, Alex Peroff, and Tim Paschkewitz at Pine Research Instrumentation and Jillian Dempsey at the University of North Carolina at Chapel Hill (UNC-CH). We were encouraged by the broad chemistry community's response to Dempsey's tutorial article, A Practical Beginner's Guide to Cyclic Voltammetry,[15] published in the *Journal of Chemical Education* in November 2017, and were eager to expand access to electrochemical training. Pine Research Instrumentation scientists proposed partnering to lead a workshop that would afford attendees a structured introduction to cyclic voltammetry. We envisioned an audience of scientists who 1) had no formal training in electrochemistry, 2) were conducting research in the fields of inorganic and organic chemistry, broadly defined, 3) were eager to employ electrochemical methods like cyclic voltammetry in their research but didn't know where to start, and 4) spanned the spectrum of training and career stages, including undergraduate students, graduate students, postdocs, and faculty. Focused planning meetings generated a list of fundamental and practical learning objectives for participants from organic and inorganic chemistry background, and from this list, a series of lecture topics and hands-on laboratories were constructed (see below). The workshop would be held at UNC-CH, which had classroom space for lectures and the Morehead Laboratories teaching labs for the hands-on parts of the workshop. Pine Research Instrumentation would provide eight electrochemical workstations and other necessary supplies, including electrochemical cells and electrodes. Professor Jeffrey Dick was invited to join the team as an instructor. Dempsey, Dick, and Peroff designed the laboratory experiences, and students in both the Dempsey and Dick labs volunteered to be teaching assistants for the laboratory sessions.

The first CVBC was piloted in June 2019 with 17 UNC-CH students and postdocs (Table 1, Figure 1). The workshop was a success, and planning for a 2020 workshop that would be open to participants from other institutions commenced. Due to renovations in Morehead Laboratories, a Fall 2020 date was selected for the workshop, but planning halted with the onset of the COVID-19 pandemic. The pandemic further prevented a workshop offering in 2021, so the second CVBC was ultimately held in July 2022. 24 participants representing 16 institutions were selected from 61 applications to attend an expanded three-day workshop. Participants were selected based on their aligned field of study and the potential for impact of CVBC participation on their research. Professor Megan Jackson joined the UNC-CH faculty in 2022 and quickly became part of the instructional team. Dr. Alex Peroff from Pine Research Instrumentation also expanded his contributions by joining as an instructor. Dick transitioned off the instructional team as he relocated to Purdue University. The third CVBC was held in June 2023, with 26 participants representing 19 institutions selected from 56 applications. Each iteration of CVBC incorporated student, instructor, and teaching assistant feedback from the previous year (see below).

Table 1. Cyclic Voltammetry Boot Camp Participants

	2019	*2022*	*2023*
Undergraduates	3	3	2
Graduate Students	13	17	19*
Postdocs	1	2	3
Faculty	0	2	2
Total Participants	17	24	26*
Institutions represented	1	16	19

*due to limited laboratory space, two participants (from UNC-CH) participated in lectures only

Figure 1. Photographs of Cyclic Voltammetry Participants in top) 2019, middle) 2022, and bottom) 2023.

STRUCTURE OF THE WORKSHOP

Topics for CVBC were chosen to provide students with a balance of fundamental and practical knowledge necessary to confidently and rigorously conduct cyclic voltammetry experiments and meaningfully analyze their data. While the exact scope of the workshop has evolved over the three years of offerings (see below), the 2023 workshop is representative and will be discussed here (Figure 2). CVBC begins with a lecture on the fundamentals of electrochemistry, led by instructor Dempsey. In this lecture, students learn the basics of interfacial electron transfer, the mechanisms and impact of mass transport, and the basic foundations of cyclic voltammetry. They are introduced to the Nernst equation and gain understanding of the distance-dependent composition of the diffusion layer at different timepoints along the voltammogram. They also learn about how scan rate influences metrics of a cyclic voltammogram. In the second lecture, led by instructor Jackson, students are introduced to the practical aspects of electrochemistry. Students engage in a comprehensive discussion of the components of an electrochemical cell; consider the key factors behind choosing a working, counter, and reference electrode; learn how to choose an electrolyte; gain familiarity with cell setups; and gain initial exposure to troubleshooting an electrochemical setup. In Lecture 3, led by instructor Peroff, students learn how a potentiostat works. Students are introduced to electrical feedback, operational amplifiers, iR compensation, and a basic potentiostat circuit. Lecture 3 walks students through potentiostat operation during the cyclic voltammetry experiments conducted in Lab 1, relating the physical electrodes and cell cables they used in lab to the potentiostat circuit diagram. In Lecture 4, instructor Dempsey introduces the concepts of chemical and electrochemical reversibility. Students learn how to distinguish between these two forms of reversibility and gain a qualitative understanding of how chemical reactions impact reversibility. This lecture then bridges into the topic of coupled chemical reactions and the difference between EC, CE, ECE, EE, and EC′ pathways.[16] In Lecture 5, instructor Peroff introduces cyclic voltammetry simulations using Pine Research Instrumentation's AfterMath Electrochemical Studio software. Lecture 5 primarily consists of a live demonstration of cyclic voltammogram simulation that incorporates simulations of the mechanisms covered in Lectures 1 and 4, simultaneously serving as a review for students. This lecture demonstrates that, used appropriately, simulation software can act as both a teaching tool and a tool for data analysis. Finally, on Day 3 participants are given the opportunity to meet one-on-one with CVBC instructors to discuss implementing newly learned electroanalytical experiments in their research.

Wednesday June 28	
8:45	Welcome and Introductions
9:00	Lecture 1: Fundamentals of Electrochemistry
10:45	*Break*
11:00	Lecture 2: Practical Aspects of Electrochemistry
12:15	*Lunch*
1:00	Aftermath Tutorial Safety Orientation
1:30	Laboratory 1: Cyclic Voltammetry of Ferrocene
Thursday June 29	
9:00	In-Class Data Workup: Analyze Data from Laboratory 1

11:00	Lecture 3: Potentiostat Basics & iR Compensation
11:45	*Lunch*
12:45	Laboratory 2: Electrochemistry Troubleshooting Stations
3:00	*Break*
3:30	Lecture 4: Electrochemical and Chemical Reversibility/Coupled Reactions
4:30	Lecture 5: Introduction to Cyclic Voltammetry Simulations
Friday June 30	
9:00	Laboratory 3: Coupled Chemical Reactions
12:00	Laboratory Cleanup
12:15	*Lunch*
1:15	In-class Data Workup & One-on-One Sessions with UNC-CH and Pine Instructors about applying electroanalytical measurements in research
3:30	Flash Presentations

Figure 2. 2023 Cyclic Voltammetry Boot Camp Schedule.

During CVBC, students participate in three laboratories. The laboratories are each supported by 3–5 teaching assistants from UNC-CH and Pine Research who, along with the instructors, supervise the labs and provide hands-on assistant to participants. The teaching assistants prepare for their role in a preparatory test run of the labs a few weeks ahead of the workshop. In advance of the laboratory sessions, students get a short tutorial in Pine Research's Aftermath software and a safety orientation. Time is built into the three-day workshop to allow students to analyze their data from Laboratories 1 and 3 with the support of the instructors and teaching assistants.

In Laboratory 1, students get their first hands-on experience with cyclic voltammetry, working in groups of three with members of each group representing a range of training and career stages. In this lab, they collect voltammograms of ferrocene in acetonitrile solutions with tetrabutylammonium hexafluorophosphate supporting electrolyte at two different concentrations, 50 and 250 mM. Voltammograms are collected at six different scan rates between, 0.05 – 10 V s^{-1}. After collecting data for the 50 mM data without iR compensation (ohmic drop correction), students measure the uncompensated resistance of their cells using positive feedback and then recollect voltammograms with iR compensation. In the data analysis session, students are asked to compare their voltammograms collected without iR compensation in 50 and 250 mM electrolyte, discovering the enhanced peak-to-peak splitting for the lower electrolyte concentration. Subsequently, they carry out a more quantitative analysis, determining the anodic ($E_{p,a}$) and cathodic ($E_{p,c}$) peak potentials and creating a trumpet plot by plotting $E_{p,a} - E_{1/2}$ and $E_{p,c} - E_{1/2}$ vs. the logarithm of the scan rate ($\log(\upsilon)$ in V s^{-1}). Students discover that minimal peak-to-peak separation can be achieved through either using a high electrolyte concentration or iR compensation and connect the concepts of solution resistance and the iR compensation process. Next, students apply the Randles-Sevcik equation to their data to calculate the diffusion coefficient for ferrocene. In the final step of the laboratory, the teaching assistants add an unknown analyte to the ferrocene solutions. Students collect a voltammogram, adjusting their potential window as necessary to observe the redox features of the unknown analyte. In the data analysis process, students learn how to use an internal standard (ferrocene) to reference their voltammograms. Laboratory 1 also serves as a hands-on introduction to the practical aspects of running cyclic voltammetry experiments and using the software that controls the WaveNow Wireless potentiostats. To complete this lab, students need to assemble a non-aqueous reference electrode, assemble an electrochemical cell, degas the cell, polish and pre-treat working electrodes, collect background voltammograms, adjust potential windows, and reference to an internal standard. This laboratory is based off of training modules presented in Dempsey's publication, A Practical Beginner's Guide to Cyclic Voltammetry,[15] published in the *Journal of Chemical Education*

In Laboratory 2, students rotate between six troubleshooting stations. At each station, a teaching assistant presents them with an obstacle that they are likely to encounter during a cyclic voltammetry experiment related to the working electrode, counter electrode, reference electrode, electrode range and autorange, iR compensation, or potential window. Troubleshooting stations are designed within the framework of the Potentiostat Basics lecture. Teaching assistants challenge students to think about why the problem is occurring in the context of how a potentiostat works. While each station has a protocol to follow, students are encouraged to experiment outside the constraints of the protocol and explore the limits of the electrochemical system. Most stations use inexpensive screen-printed electrodes in aqueous solvents, allowing students to experiment without worrying about damaging precious and expensive electrode materials. After rotating through all stations, students have practical knowledge of how to identify, diagnose, and solve common problems encountered during electrochemical experiments.

In Laboratory 3, students learn about coupled electrochemical reactions by exploring the chemical reactivity coupled to the reduction of a cobalt complex $[Co(Cp)(dppe)(CH_3CN)][PF_6]_2$ (Cp = cyclopentadienyl, dppe 1,2-bis(diphenylphosphino)ethane).[17,18] Upon reduction of the cobalt(III) species, the coordinated CH_3CN ligand dissociates in an EC mechanism. Students apply cyclic voltammetry to identify from which oxidation state ligand dissociation occurs, quantify the kinetics of dissociation, and determine the associated equilibrium constant. To do so, students first record voltammograms in dichloromethane electrolyte. They observe an irreversible cobalt(III/II) reduction due to dissociation of CH_3CN upon reduction. By varying the scan rate, students observe a shift in $E_{p,c}$ as a function of scan rate and use these data to extract a rate constant for CH_3CN dissociation. Students then titrate aliquots of CH_3CN into the electrolyte and observe that reversibility is regained. From these data, students plot the $E_{1/2}$ of their wave as a function of CH_3CN concentration to determine the equilibrium constant K for dissociation. Through Laboratory 3, students learn how different reaction schemes (EC, CE, ECE) impact cyclic voltammograms, diagnose the chemical step through scan rate- and concentration-dependent voltammograms, and learn how thermodynamic and kinetic information can be extracted for homogeneous reactions. Finally, students us digital simulation software to examine EC reactivity as a function of scan rate and coordinating ligand concentration. At the conclusion of the workshop, student laboratory groups are assigned to present a subset of their analyzed data, including responses to discussion questions posed in the laboratory manual. Between all eight groups, fully-analyzed data from each experiment was presented and students provide context and interpretation of the data through their presentation of discussion question responses. Students answered questions about their data from instructors and other participants. Overall, students demonstrated comprehension of the material through these presentations and instructor questions and comments were used to address any misconceptions.

IMPACT AND FEEDBACK-DRIVEN COURSE CHANGES

Since the inaugural year in 2019, 64 students from around the country have completed CVBC (Table 1, Figure 1). Each year at the completion of CVBC, students were asked to fill out an anonymous survey to provide feedback on the course. In 2019, 16 of 17 participants responded to the survey; in 2022 15 of the 24 participants responded to the survey, and in 2023, 13 out of 26 participants responded. Averaging across all years, on a scale of 1–5, the average rating of the course was a 1.17 (1 = "extremely valuable" and 5 = "a waste of my time"). When asked about how confident students felt about carrying out cyclic voltammetry experiments in their independent research, students uniformly expressed confidence in what they had learned (Average = 1.83, with 1 = "very confident" and 5 = "not prepared at all"). The survey also included open-ended questions about which topics they found most valuable, what they enjoyed about the course, and what they would change. Several changes were made to the workshop since the inaugural year in 2019 based on feedback from the instructional team, TAs, students, and post-course surveys. Incorporating these changes had a marked improvement on student's confidence in incorporating CV experiments into their own research. While the average overall course rating stayed relatively constant across the three years of CV boot camp (1.13 in 2019, 1.15 in 2022, and 1.23 in 2023), student confidence jumped noticeably from 2.00 in 2019 and 2.07 in 2022 to 1.61 in 2023. The significant changes we made each year are detailed below.

One major change was the expansion from a two-day workshop in 2019 to a three-day workshop in 2022. The extra day primarily allowed students time for in-class data analysis with the support of instructors and TAs, and also allowed for additional lectures on potentiostat basics and rotating disk electrodes, flash presentations, and time for students to speak with instructors one-on-one about their independent research. Dempsey and Jackson also offered lab tours where attendees could see what electrochemistry setups look like in a research setting.

Small changes to content have also been made over the years. In 2019, students were offered a choice between three laboratories for the third lab: Coupled Chemical Reactions, Rotating Disc Electrode Voltammetry, and Ultramicroelectrodes. In 2022, only Coupled Chemical Reactions and Rotating Disc Electrode Voltammetry were offered. In 2023, the instructional team decided to streamline the workshop by offering only the Coupled Chemical Reactions experiment, noting that it was of broadest appeal. The lecture on rotating disk electrochemistry was replaced with a more detailed discussion of electrochemical simulation software and coupled chemical reactions. Finally, the workshop was offered at no cost (except for housing) in 2019 and 2022; in 2023 a nominal registration fee of $150 was instated to help cover the cost of consumables, chemicals, printed course materials, and meals.

OTHER RELATED WORKSHOPS

CVBC is not the only independent workshop that seeks to augment academic course-based electrochemistry training. The Workshop on Electrochemical Measurements, Electrochemical Bootcamp, Cyclic Voltammetry International school, and Organic Electrochemistry Short Course all provide combined lecture and laboratory-based curriculums (Table 2). The scope of the workshops ranges from workshop to workshop. Some, like Organic Electrochemistry Short Course, have a tailored application scope, while others like the Electrochemical Bootcamp touch on a range of applications. Similarly, the scope of electroanalytical methods introduced in a single workshop ranges from exclusively cyclic voltammetry to a diverse array of electroanalytical and preparative electrolysis methodologies. The workshops are all united in their mission to empower participants with the theoretical and practical knowledge necessary to carry out meaningful electrochemistry experiments in their research. In the context of these other workshops, unique aspects of CVBC is the combined focus on providing electrochemistry training for novices and on targeting participants with backgrounds in inorganic and organic chemistry that represent a range of training and career stages. In addition, CV Boot Camp has a unique focus on instrumentation and troubleshooting, facilitated by the partnership between Pine Research and UNC-CH.

Table 2. Selected workshops for electrochemistry training.

Workshop Name	*Workshop on Electrochemical Measurements*	*The Electrochemical Bootcamp*	*Cyclic Voltammetry International School*	*Organic Electrochemistry Short Course*
Year Started	Early 1980s	2013	2015	2019*
Length of Workshop	5 days	3 days	5 days	
Current Organizers	Daniel Scherson (Ernest B. Yeager Center for Electrochemical Science, Case Western Reserve University)**	Joaquin Rodríguez-López (University of Illinois, Urbana Champagne)	Cyrille Costentin (Université Grenoble Alpes), Marc Robert, François Mavré & Niklas von Wolff (Laboratoire d'Electrochimie Moléculaire, Université Paris Cité), Cédric Tard (Ecole Polytechnique)***	Shannon Stahl (University of Wisconsin-Madison), Mohammad Rafiee (University of Missouri-Kansas City)

Target Audience	Academic, industrial and national laboratory communities	Undergraduate students, graduate students, postdocs	Graduate students, postdocs, early career faculty	Upper-level undergraduate students, graduate students, postdocs, industrial researchers, and faculty members with a background in organic chemistry
Scope of the workshop	interfacial physical chemistry, corrosion, electrocatalysis, energy conversion and energy storage, electrochemical engineering, electrochemical kinetics, and electroanalytical chemistry	Theoretical and practical aspects of electrochemistry, spanning basic electrochemical setup, electrocatalysis, simulations, ultramicroelectrodes, batteries, preparative electrochemistry, and scanning electrochemical microscopy	Application of cyclic voltammetry to contemporary challenges in energy-related catalysis, small molecule activation, and redox enzymes	Introduction to theory and practical aspects of organic electrochemistry, including electrosynthesis (bulk electrolysis) and electroanalytical studies
Workshop format	Lectures and hands-on laboratories	Lectures and hands-on laboratories and demonstrations	Lectures and hands-on laboratories	Lectures and recorded laboratory demonstrations
Goal	Provide theoretical and hands-on experimental laboratory experience	Expand knowledge of electrochemical concepts and prepare students for practical aspects of designing and conducting experiments	Spread theoretical knowledge and practical experience in cyclic voltammetry	Demonstrate how electrochemistry can be used for organic synthesis and to help its attendees understand the theoretical and practical aspects of this tool. Promote the broad use of electrochemistry by organic chemists
Notes *Initially in-person, migrated to online version, now an asynchronous virtual short course **founded by Ernest B. Yeager **Other faculty involved in the initial workshops include J.-M. Savéant and B. Limoges				

CONCLUSIONS

245 While the complete reinvention of formal electrochemical education might take decades to accomplish, CVBC, together with other workshops and short courses, has begun to fill a need for

enhanced formalized training in electrochemistry. Workshops and short courses can be developed relatively quickly and focus on electrochemical topics most relevant to current research topics. For those who are interested in starting an electrochemistry workshop, we recommend the following steps based on our own development of CVBC.

1) **Identify pedagogical goals and target audience.** CVBC is specifically designed for students who have little or no exposure to electrochemistry to feel confident carrying out simple electrochemistry experiments in their research.
2) **Find partners, both academic and industrial to aid in development.** Industrial partners such as Pine Research Instrumentation can provide physical equipment that might be a challenge for starting such a workshop.
3) **Find the appropriate lab space.** Teaching labs such as Moorehead are designed for undergraduate chemistry labs, which can accommodate the space requirements for a large workshop. Consider the experiments you plan to have students do. Will they need hoods and gas cylinders?
4) **Include workshop time for data analysis and discussion.** Students cannot incorporate cyclic voltammetry experiments into their research unless they feel confident with both experimental setup and data analysis.

Each year ca. 24 students are accepted to CVBC, based on lab space and equipment. However, over 60 people apply each year to take part in CVBC, which is primarily marketed through social media and word-of-mouth, highlighting the high demand for electrochemistry workshops. As such, we encourage electrochemical researchers and scientists to develop workshops to help address the dearth of training opportunities available for scientists who wish to add electrochemistry into their research.

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