Standard Operating Procedure: Electrochemistry Experiments

Last Updated 05/15/17 by Selene Li

Reagents:

- Check the availability of solvent in the small glovebox. If solvents need to be pumped in the box, make sure they are dry and airfree. If not, please dry and degas it.
- Common supporting electrolytes used in the McCusker lab are NBu_4PF_6, NBu_4BF_4, and NBu_4ClO_4. Please check the availability for the chosen supporting electrolyte prior use.

Preparation Before Experiments:

- Most of the electrochemical techniques employ the use of a standard three-electrode system, including working (either platinum[1], or glassy carbon), reference (Ag/AgCl or Ag wire pseudo reference), and auxiliary/counter (Pt wire or graphite) electrodes. Please choose appropriate materials for your experiments[2].
- A smooth, clean, and uniform electrode surface is necessary for reproducible problem free electrochemistry experiments. Make sure all working electrodes are polished prior use. See SOP: Electrode Polishing and Care for details. Please handle with care to protect their surface.
- The Ag/AgCl reference will need to be rinsed with the solvent used in your measurement, and pumped into the box by 20 quick cycles. Please see appointed person for operation. The Ag/AgCl reference electrode should only be out of the 1M KCl solution for no longer than 3 hours.
- For the use of Ag wire pseudo reference, wet-sand the electrode thoroughly to get rid of the oxidized outer layer of the wire, and rinsed with DI water and the solvent used for your measurement and dried before use. Rinse the customized glass tube with DI water, MeOH and the solvent used for measurement. After that, cover the Ag wire with the glass tube.
- Rinse the Pt wire counter electrode with water, MeOH, and the solvent used in measurement. Let the electrodes air dry or by blowing N_2. It is not recommended to use the graphite electrodes unless you need to perform corrosion tests. Please see the electrochemistry point-person for more questions if graphite counter electrodes are preferred.

Beginning Experiments:

- Pump your samples, all electrodes, the customized vials with special hole-drilled caps into the small glovebox. See SOP: Glovebox Operations for details.
- Turn on the CH Instrument Electrochemical Workstation. The switch is a button on the front, facing the small box, and the button will be green when it's on. MAKE SURE TO TURN IT OFF AT END OF USE.
- Open the CHI650d software locating on the desktop of the computer next to the small box. 

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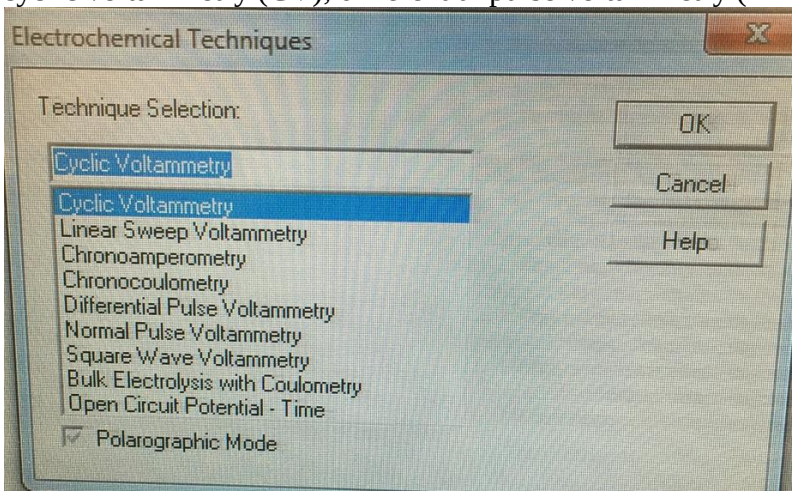
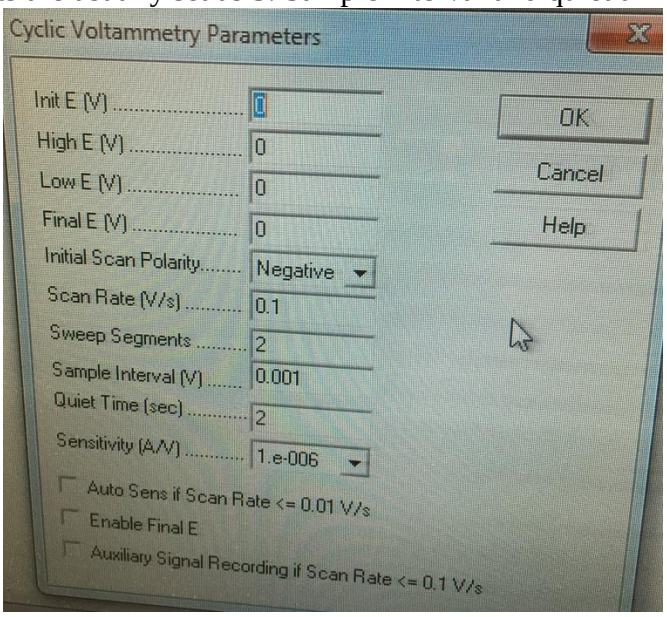
- Click Setup and Techniques to choose your method of electrochemistry, e.g. cyclic voltammetry (CV), differential pulse voltammetry (DPV) etc.

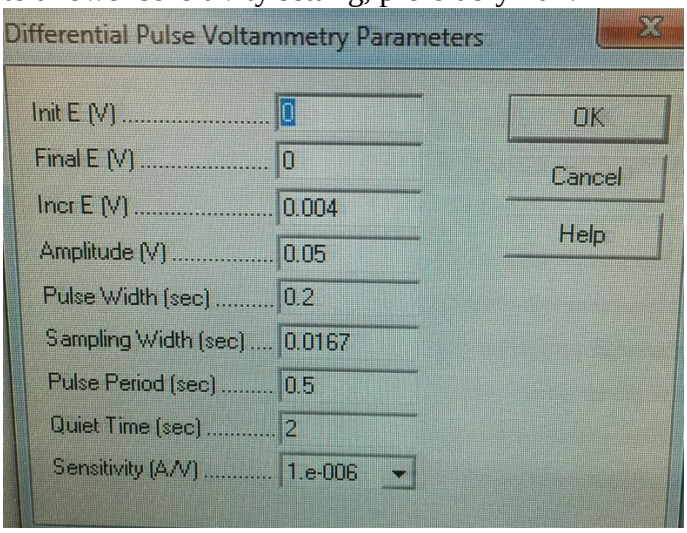


- Click Parameters to set up your method, and save the change.
  - For CV, set initial and final E (usually 0.00V), and high and low E will be determined by the desired potential window for your experiment. Scan polarization will depend on which direction you want to scan first, negative or positive. Scan rate is usually set as 0.1V/s, and you can change this to the desired scan rate based on your needs. Set the sensitivity to be the value bigger than what you think the current will be. It can stay as default, 10^{-6}, or you can change it to a lower sensitivity setting, preferably 10^{-5}. See the Echem point-person for questions. Sweep segments are usually set as 5. Sample interval and quiet time will stay as default.

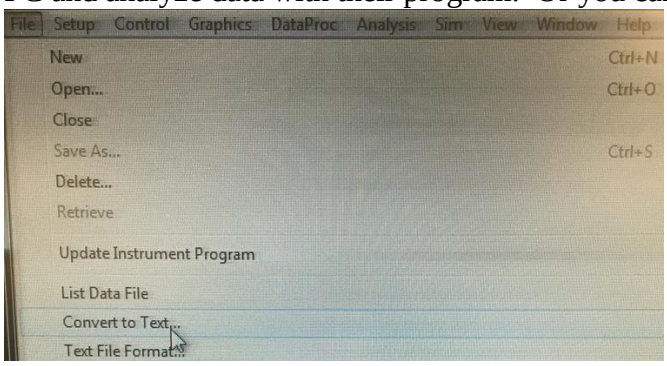


  - For DPV, set initial and final voltage as the desired potential window for your experiment. Increment E, amplitude E, pulse width, sampling width, pulse period, and quiet time will stay as default, which are 0.004V, 0.05V, 0.2 s, 0.0167 s, 0.5 s, and 2 s respectively. These values can be changed according to your needs. The sensitivity is set as 10^{-6}, or you can change it

to a lower sensitivity setting, preferably 10^{-5}.



- Once the parameters are set, click the Play button to start the measurement. You can Pause and Stop a run.
- When the measurement is done, make sure to save the data. It will say Run Unsaved at right if you haven't saved.
- Data saves as a CH Instruments file, you can download the software if you have a PC and analyze data with their program. Or you can convert the data to text.



- If the data is very noisy, smoothing can be performed by the software by clicking DataProc and smooth.
    - The noise of the data can also due to the fluctuation of the glovebox atmosphere. If this is the case, repeated measurement is recommended. See pointed person for questions.

<u>Troubleshoot</u>

**Only see a flat line without signals. —Check whether your electrodes are connected properly.

**Only see noise or weird curves—check whether electrodes are connected properly and are not touching the side or each other, and ARE submerged down into the solution.

**When you open the program you get an error message—shut down program and turn on Echem workstation first, then open program.

**If you see signal at first and then it flats out—stop the run and set your sensitivity to a higher value, or less sensitive; you are just out of the range of the current you chose.

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After Experiments:

- Used Pt wire counter electrodes need to be soaked in 1M HNO_3 for at least 1 h.
- Used Ag wire reference electrodes with the glass tubes need to be rinsed by the solvent used for Echem data collection at least 3 times, and soaked in 1M HCl for about 1 h. Then they need to be air dried or blow-dried by N_2 before putting back in the Echem drawer.
- Used working electrode (Pt or glassy carbon) need to be rinsed by the solvent used for echem measurement at least 3 times. Then, they need to be air dried or blow-dried by N_2 before putting back in the Echem drawer.
- Used Ag/AgCl reference electrode will be soaked in solvent used for experiments, DMF, and MeOH for 5 mins respectively. After that, rinse the electrode in DI water, and put in back in a vial with fresh 1M KCl solution.
- Turn off the CH Instrument Electrochemical Workstation

Personal Protective Equipment:

Safety glasses, nitrile gloves, and long sleeves are required for the use of the small glovebox are required.

Waste Disposal:

Any hazardous waste generated during the experiment should be pumped out of the small box and disposed properly according to the standard operating procedure.

Hazards:

When chemicals and glassware pumped out from the gloveboxes, they can be dangerous to you. Please handle them with care.

Material Safety Data Sheets:

Call the ORCBS (517-355-0153) or see lab copy for samples in questions if available.

Green = Working electrode
Red = Reference electrode
White = Counter electrode
Black = Ground (connect to nothing)

Appendix:

[1]CHI102 Pt Disk Working Electrode: The supporting material is Kel-F with chemical property similar to Teflon. Kel-F will become soft and change its shape when the temperature is above 60˚C. Please refer to CHI Instruments, Inc. for more information.
[2]Kelly, R. S., Analytical Electrochemistry: The Basic Concepts. http://www.asdlib.org/onlineArticles/ecourseware/Kelly_Potentiometry/EC_CONCEPTS1.HTM for general reference.