General Electrochemistry Guidelines

- Either purge the purgebox in the IR room or begin collecting your solvent of choice before getting ready to run electrochemistry. You need dry solvent to run electrochemistry, and we have some in the purgebox and the stills can also be used (although only if no electrochemically pure solvent is available).
    - Put a 50mL Erlenmeyer flask in the purgebox antechamber, then purge the box as directed on the signs on the box. The purging process takes about 30 minutes, so plan ahead. The same applies for solvent distillation.
- Obtain a vial and the [n-Bu_4N][PF_6] from the dessicator.
- Weigh out about 750mg (0.75g) and place in the vial (this creates an ~0.2M solution in 10 mL solvent. This must be adjusted for electrolytes other than [n-Bu_4N][PF_6]). It also must be adjusted if you use only 5 mL or so.
- Replace [n-Bu_4N][PF_6] in dessicator when done with it.
- Carefully rinse off the working electrode (black), the Pt wire (counterelectrode, metal wire), the reference electrode (Ag/AgCl, stored in 3M NaCl solution) and the Teflon® cell cap with water and then the solvent you will be using in your experiment. Acetonitrile and dichloromethane are commonly used.
- Dry all surfaces of electrodes with a Kimwipe. Be careful with the electrodes as to not damage them. For the reference and working electrodes, gently dab the bottoms to avoid scratching or damaging the sensitive parts of the electrode.
- Put working and counter-electrodes in the Teflon® cap and gently place into vial containing the [n-Bu_4N][PF_6].
- Put completed cell assembly in the clamp on the stirplate.
- Get a new pipette from the second drawer from the top and place in the Tygon® tubing that supplies N_2 (above the stirplate) and carefully insert the thin end into one of the smaller holes in the Teflon® cell cap. Clamp down the pipette so that it stays in place, but that it can move with minimal force.
- Adjust the electrode height so that the Pt wire is ¾ the way down (not really adjustable) and that the working (black) electrode is ½-way down the vial.
- Make sure that the O-ring on each electrode is touching the Teflon® cap.
- Add your dry solvent/or solven/electrolyte mixture if you make a stock solution to fill the vial to ~2/3 full (or less if you push the electrodes down further you can get away with much less solvent which is important if you do not have a lot of compounds) . (assuming that you have it out).
- Open the N_2 valve on the wall by the phone to ~30^o. Carefully open the Swagelok needle valve until there is a steady, but not violent, stream of bubbles in the solution. This is to deoxygenate the cell/solution.
- Insert the reference electrode but do not submerge it while bubbling N_2 through the solution. Submerging the reference electrode while bubbling N_2 could cause unwanted contamination in the electrode itself.
- Connect the alligator clamps as follows for CV and DPV:
    - Red clamp on counter-electrode (Pt wire)
    - Green clamp on working (black) electrode
    - White clamp on reference electrode.
- Bubble N_2 through your solution for 5 minutes at a minimum.

- Turn on the instrument (the toggle switch is located on the top-right corner of the back panel). A red light should appear on the front above 'Power'.
- Log into the account on the CV computer (Dunbar Group, acetone) and open CHI620A (on the desktop).
- Go to Setup→Technique and select appropriate method (Cyclic Voltammetry or Differential Pulse Voltammetry)
- Go to Setup→Parameters and fill out accordingly. Starting parameters for CV are below:
    - Init E(V): 0V initially, but if a redox event occurs there it will need to be shifted such that the starting point is at a voltage more negative than the most negative oxidation event or more positive than the most positive reduction event—discuss this with Kim
    - High E(V)/LowE(V): This depends on the electrochemical range for each solvent-check on the provided chart. For acetonitrile, this is 2.0V and -2.0V respectively, though we typically use 1.8V and -1.8V.
    - Initial scan polarity: Negative (this indicates whether the scan will go towards negative or positive potentials first. Negative is the usual choice).
    - Scan Rate (V/s): 0.2V/s is recommended by Kim. Faster is not recommended. Slower is fine but will naturally increase the time required.
    - Sweep segments: 3 or more. This is the number of sweeps. If you only use 2, part of the cathodic current (for a negative scan polarity) will be missing, namely the portion prior to the starting voltage.
    - Sample Interval: Leave default.
    - Quiet time: Leave default.
    - Sensitivity (A/V): $1X10^{-6}$ A is the default value and usually fine, though if you have a highly concentrated sample, this may need to be lowered. Avoid going less sensitive than $2X10^{-5}$.
    - Parameters for DPV are similar, but have a start and end V and a scan rate. Set to the solvent window that was used for the CV.
- Once the run is set up, pull the N_2 pipette out of the solution and adjust the flow rate such that the solution is not disturbed at all (no ripples or bubbles) but a blanket of N_2remains in the cell. Lower the reference electrode (with the white wire) into the solution halfway.
- Press the 'Play' button (►). The blank run will start.
- Check to ensure that the blank is flat and narrow with no oxygen or water redox activity (refer to standard CVs that are near the computer).
- Once the blank is satisfactory, add your sample (a spatula-tip or so, consider the MW of your compound and how fluffy your material is…weigh it if necessary).
- Run the CV/DPV at the initial parameters and then adjust the parameters if necessary and repeat until you have an acceptable run. Make sure that the CV does not start on a redox event and that any irreversible reductions/oxidations are cut off (save one CV with them in it for posterity) so that only the reversible couples are shown.
- Once done, clean everything! This involves rinsing off ALL electrodes with copious amounts of your solvent, then acetone and then water and then drying them, emptying and cleaning the vial with solvent, soap and water and leaving it upside down on a towel to dry, putting the electrodes back in their respective places (the working/counter electrodes in the holder into the vial and the reference

electrode back into the salt solution from whence it came) and removing/disposing of all unused solvent, syringes, glassware, compound etc.

- SIGN THE LOGBOOK!