Standard Operating Procedure

Task: Troubleshooting Electrochemistry
Date: 5/15/2014
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Background:

- Electrochemical experiments often present a variety of challenges in obtaining and interpreting data. A short primer on some common problems is presented.

Training Requirements:

- Laboratory safety training
- Electrochemistry Training

Potential Hazards:

- Shock – ensure current is not running when manipulating leads

Materials Needed:

- Assembled experiment (potentiostat, electrodes, electrode leads, cell, solution)

Procedure:

- If an electrochemical experiment produces irreproducible, noisy, or distorted results, there are a number of components that may be the cause. A troubleshooting workflow, and problems we have encountered are listed below.
- Typical troubleshooting procedure:
    - Ensure that all electronics are connected on the same circuit. Different outlets can be at different potentials leading to anomalous currents in the system. This has particularly been noticed when the pH probe is used in electrochemical cells.
    - Ensure that the electrodes are connected to the correct lead:
        - Red/orange – working (1)
        - Purple/blue – working (2, WaveDriver only)
        - Green – counter
        - White – reference
        - Black – ground
    - Ensure that none of the electrodes or leads are touching each other or other conducting metals.
    - Ensure that all of the electrodes are submerged in the electrolyte solution.
    - Try grounding the cell or changing where the ground is clipped
    - Ensure that no motors are running in the vicinity (fans, stirplates…)
    - Test each electrode separately.
        - Disconnect the alligator clip from the white banana clip and plug into the back of the green clip. Run the experiment. If the problem has disappeared, the problem is with the reference electrode.
            - Common reference electrode problems are bubbles in tube, dry vycor (particularly with new reference electrodes), and low solution level in the glass tube.

        - Polish glassy carbon (if using) and check to make sure that the electrode surface is shiny and reflective.
            - If the surface is distorted, the configuration of the cell might be damaging the electrodes, seek help before proceeding.
            - The brass pins at the top of the electrode can rust (especially if stored near solvent), which can lead to high resistance. The rust can be gently sanded off. Seek assistance before sanding any electrodes.
        - Pt has not been observed as a problem. Check the wire for cracks or films deposited on the metal.
    - If the problem is not resolved, completely disassemble and reassemble the circuit.
        - Disconnect USB from computer and potentiostat and reconnect.
        - Disconnect electrode leads from cell. Disassemble the cell. Take a break.
        - Approach the experiment again as though starting for the first time with fresh solutions, clean electrodes and new electrical connections.
    - If the problem is not resolved, seek help.

- Common problems and their solutions:
    - Erratic spiderweb formations
        - Typically caused by a disconnected working electrode, a short circuit or by a bubble in the reference electrode.
    - Regular, periodic noise
        - Grounding problem
        - 60 Hz noise caused by electronics in the vicinity

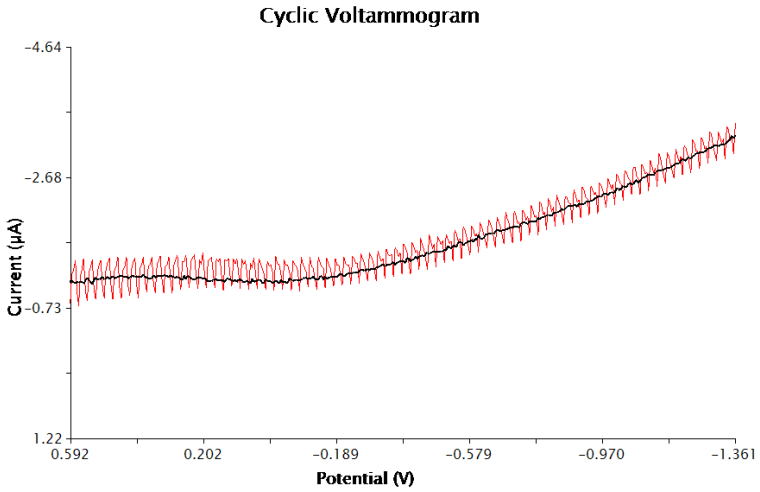

Figure 1: CVs of identical solution with (red) and without (black) the box fan on

    - Smeared out peaks
        - High solution resistance: add electrolyte or change cell configuration to move reference electrode closer to the working electrode.

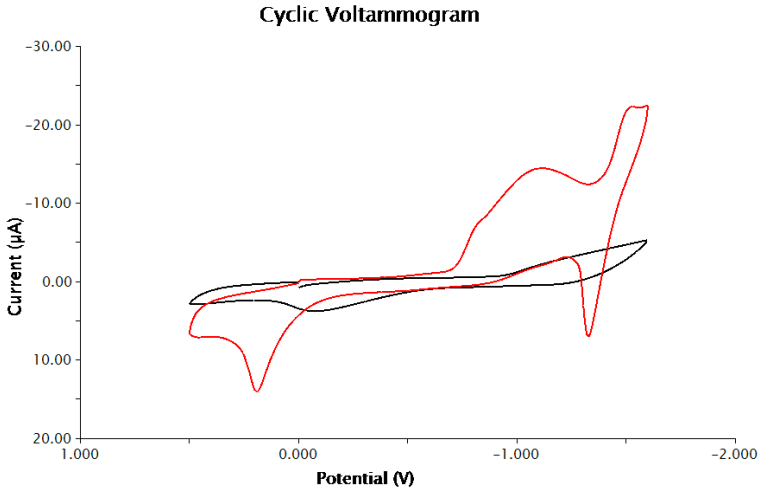

*Figure 2: CVs (0.5V/s) of the same solution of [Cp*Ir(bpy)Cl]Cl in tris buffer with a frit separating the working and reference electrodes (black) and in an undivided cell (red)*

- Very noisy bulk electrolysis
  - Poor stirring

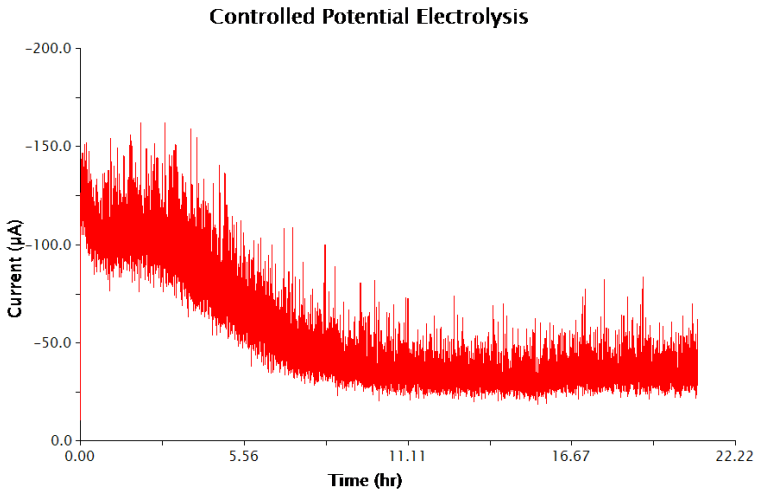

Figure 3: Noise caused by using a stirbar too large for the electrolysis cell

- Linear regions and missing data points
  - Autoranging causes data points around the range change to be disregarded. To improve data, turn autoranging off.