Standard Operating Procedure for Cyclic Voltammetry using the BAS 100 Electrochemical Analysis System Last Modified March 4, 2011

Considerations Prior to Beginning Experiment

- Cyclic voltammetry requires the use of a working (often gold, platinum, or glassy carbon), reference (often Ag/AgCl) and auxiliary (often a platinum coil) electrodes. Be sure to use the appropriate electrode materials for your experiment.
- To avoid complications of migration, experiments are generally run in solutions of high ionic strength that is maintained using a supporting electrolyte. Common supporting electrolytes are 0.1 M KCl or KNO₃.
- The condition of the working electrode surface has a large impact on the quality of results. Be sure to use appropriate polishing techniques.
- Connect the leads of the BAS 100 as follows: BLACK to Working Electrode, RED to Auxiliary Electrode and WHITE to Reference Electrode.

Instrument Start-up Procedure

• The main instrument power switch is on the back of the BAS100. Turn the switch on and allow the instrument to initialize prior to opening the software. The instrument will produce a short series of beeps when it is initialized.

Preparing the Instrument for a Measurement

1. On the computer desktop, find the program icon "BAS 100W" and click on the icon to start the program.

General Steps for Making a Measurement

- In order to run a cyclic voltammetry experiment, click on the Method menu and choose Select Mode. A dialog box should appear, in this box, choose 1. Sweep Techniques and Cyclic Voltammetry and then click OK.
- To prepare the instrument for a specific experiment it will be necessary to adjust the potential window, scan rate, etc. by clicking on the **Method** menu and choosing **General Parameters**. Doing this brings up the dialog box shown at the right. Once all values have been set, click on **OK** to continue.

CV General Parameters	×
Initial E: 🗕 🛛 mV	
<u>H</u> igh E: 0 mV	
Low E: 0 mV	
Scan <u>R</u> ate : 100 mv/s	·
Initial Direction	_
⊙ <u>N</u> egative C <u>P</u> ositive	
N <u>u</u> mber of Segments : 2	
Sensitivity 1 uA/V	
Cancel OK Specif	ic > <u>></u>

- The **Sensitivity** setting controls the current-tovoltage converter in the potentiostat. It is best to set the sensitivity to the smallest number of amps per volt that allows the voltammogram response to stay within the range of the potentiostat. If the tops of voltammetric peaks are cut off, increase the number of amps per volt.
- The **Number of Segments** setting, corresponds to the number of times that the potential changes direction. For a typical experiment where the potential starts at one end of a range, scans to the other end and returns to the original potential, the number of segments will be two (2).
- 3. Assuming all electrodes are connected, you can now initialize a run by clicking **Control Start Run** or by pressing **F2**.

Data Handling

Saving Data

- 1. Save the CV using the File Save command
 - Each CV is stored as an individual file.
 - There is an eight character limit for filenames. It is suggested that a dated format be used such as xxyyzz## where xx is the year, yy is the month, zz is the day and ## is a file number.

Peak Potential and Peak Current

1. Determine the peak potential and peak current for each peak on each CV by clicking on **Analysis -Results Graph** or by pressing **F6**.

Instrument Shut-down Procedure

- Be sure that all electrodes have been disconnected and rinsed with distilled-deionized water.
- Power the instrument off.