

**Standard Operating Procedure for
Cyclic Voltammetry using the
eDAQ Potentiostat and Electrochemical Analysis System
Last Modified March 4, 2011**

Considerations Prior to Beginning Experiment

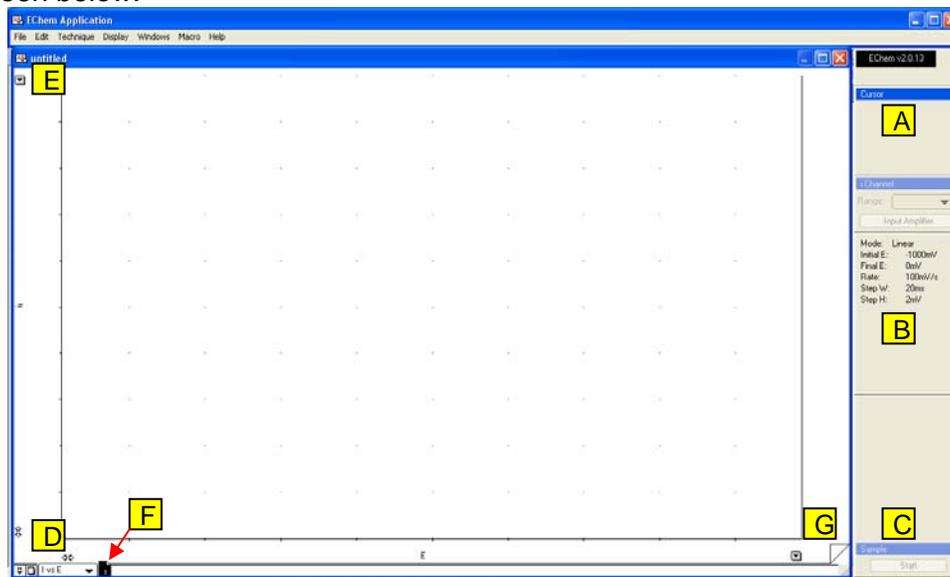
- Cyclic voltammetry requires the use of a working (often gold, platinum, or glassy carbon), reference (often Ag/AgCl) and auxiliary (often a platinum coil) electrodes. Be sure to use the appropriate electrode materials for your experiment.
- To avoid complications of migration, experiments are generally 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 EDAQ potentiostat as follows: GREEN to Working Electrode, RED to Auxiliary Electrode and YELLOW to Reference Electrode.

Instrument Start-up Procedure

- Ensure that the eCorder is plugged in to a USB port on the computer.
- The main instrument power switch is on the back of the eCorder unit (the larger of the two boxes). Turn the switch on and allow the instrument to initialize prior to opening the software.

Preparing the Instrument for a Measurement

1. On the computer desktop, find the program icon “eDAQ Echem” and click on the icon to start the program. If the eCorder is connected and powered on, you should see the screen below:



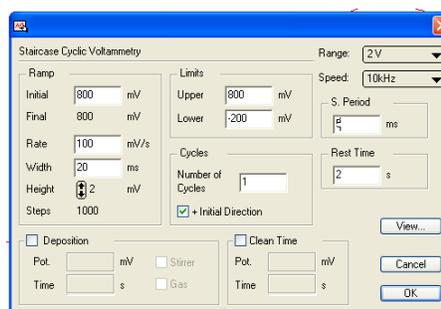
Aside from the menu bar, the following characteristics of the window are:

- A. If data is present in the main window, a cursor is pinned to the data. The **Cursor** portion of the window provides the x and y coordinates for the cursor.

- B. The current settings for the selected technique are displayed here. The sensitivity of the current to voltage converter is set using the **Range** dropdown list.
- C. Clicking **Start** initiates a run.
- D. Clicking on the appropriate arrow expands or contracts the x or y axis range. These limits can be set manually through the menu bar.
- E. Typically cyclic voltammetry data is plotted with negative potential to the right and negative (cathodic) current going up. Clicking on the dropdown arrow will allow the user to select **Invert Axis** to change the direction with which data are displayed on the y-axis. There is a corresponding dropdown arrow for the x-axis as well.
- F. Individual scans are stored in a single file as pages. Once a new scan is created, a new page will appear corresponding to that scan. Clicking on an existing page displays that page's data. The active page is highlighted in black.
- G. Clicking on the appropriate "dog ear" graphic advances or goes back one page at a time.

General Steps for Making a Measurement

1. In order to run a cyclic voltammetry experiment, click on the **Technique** menu and choose **Cyclic Voltammetry**. The dialog box at the right should appear.
2. To prepare the instrument for a specific experiment it will be necessary to adjust the potential window, scan rate, etc. to appropriate values. Once all values have been set, click on **OK** to continue. The most common values that are changed are:



- The **Limits** setting controls the upper (positive) and lower (negative) potentials for the potential waveform.
 - The **Initial** setting determines the starting potential for the waveform.
 - The **Rate** setting determines the scan rate for the voltammogram.
 - The **Number of Cycles** setting, corresponds to the number of times that the potential returns to its original value. 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 cycles will be one (1). If more than one cycle is collected, each cycle will be recorded on its own page.
 - The **Range** setting determines the maximum positive and negative potentials that the potentiostat can produce. A setting of 1V or 2V is typical. The smaller the range setting, the better the voltage resolution, but the narrower the voltage range.
3. In the main window, the **Range** setting under the **I Channel** portion of the window controls the sensitivity of the current-to-voltage 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.
 4. Assuming all electrodes are connected, you can now initialize a run by clicking **Start** under the **Sample** portion of the window.

Data Handling

Saving Data

1. Save the CV using the **File - Save of File – Save As** command
 - Several CVs can be stored as individual pages in one file.

Peak Potential and Peak Current

1. Determine the peak potential and peak currents can be determined using the **Cursor** portion of the display and moving the cursor to the top of a peak and recording the appropriate values.
2. A second approach to making these determinations is to use the **Data Pad**, which is available through the **Windows** menu. The Data Pad allows automatic recording of many parameters related to the data. Instructions for use of the Data Pad begin on page 55 of the eDAQ EChem Software Manual. This manual is available in pdf form through the **Help** menu of the EChem software.

Instrument Shut-down Procedure

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