CYCLIC VOLTAMMETRY

1. **Computer**

Log in to computer

2. **How to start-**

The link between the PC and the epsilon EC will automatically be established. The status of the link will be displayed in the link dialog box, which will disappear once connection has been established.

2.1 Turn on the Epsilon EC

2.2 Open the Epsilon PC software. If the connection between the Epsilon EC and the PC software is successful a link dialog box will appear describing the successful connection.

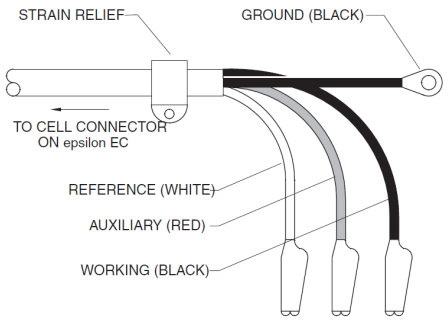
If the connection has failed, you will receive a dialog box requesting that you retry connection.

3. Cell

3.1 Auxiliary electrode: Platinum wire(Red lead)

3.2 Working electrode: Platinum (Black lead)

3.3 Reference electrode: Ag/AgCl (White lead)



4. How to run a CV

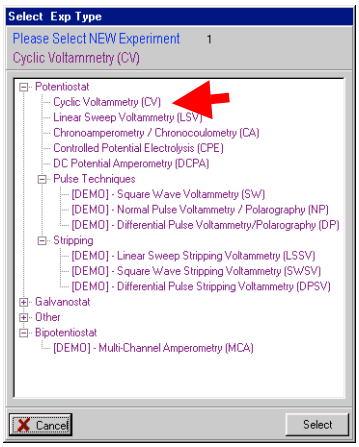
4.1 Prepare a 0.1 M solution of electrolyte ($Bu_4N^+BF_4^-$ in the drawer labeled CV supplies) with a fresh distilled solvent (CH_2Cl_2, THF, CH_3CN, TFT…). Recommended volume is 10-15 mL. Add about 10 mg of sample. The volume of the cell is 20 mL so ~15 mL is the maximum volume you may use.

4.2 Degass (N_2 pressure not higher than 5 psi) and stir the solution using switches on the CV.

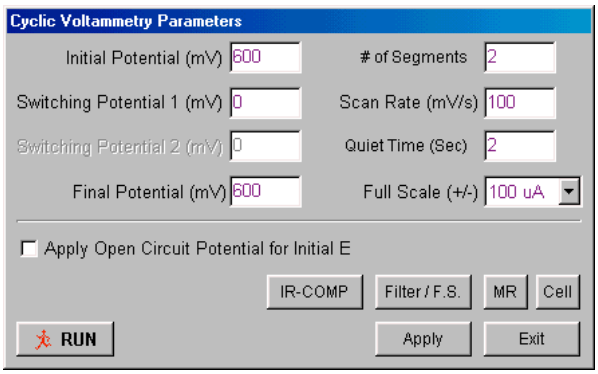
4.3 Rinse the reference electrode with whatever solvent you will use.

4.4 Connect the electrodes according to the color of the leads.

4.5 Click **New** in the **File** menu (or use the **F2** key) to set up a new experiment. The list of available techniques is displayed. This label indicates that this technique is NOT active on this particular epsilon.

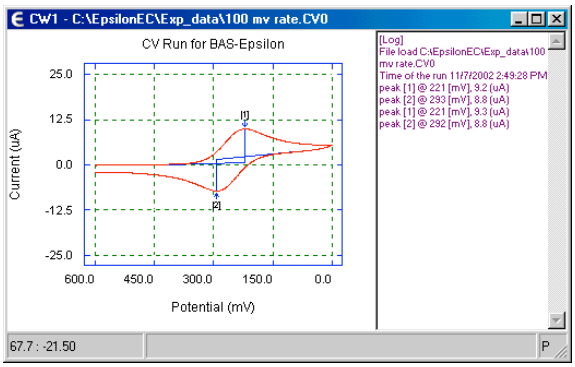


4.6 The **Change Parameters** dialog box will now be displayed. Enter the desired parameters. Once these changes have been entered, an experiment using these parameters can be run by clicking the **RUN** button in this dialog box. If **Exit** is clicked before **Apply**, any changes in the parameters will be lost. After exiting the dialog box an experiment can be run by clicking the **RUN** icon on the Tool Bar. This icon will change to **STOP** during the experiment, and can be used to abort the experiment.

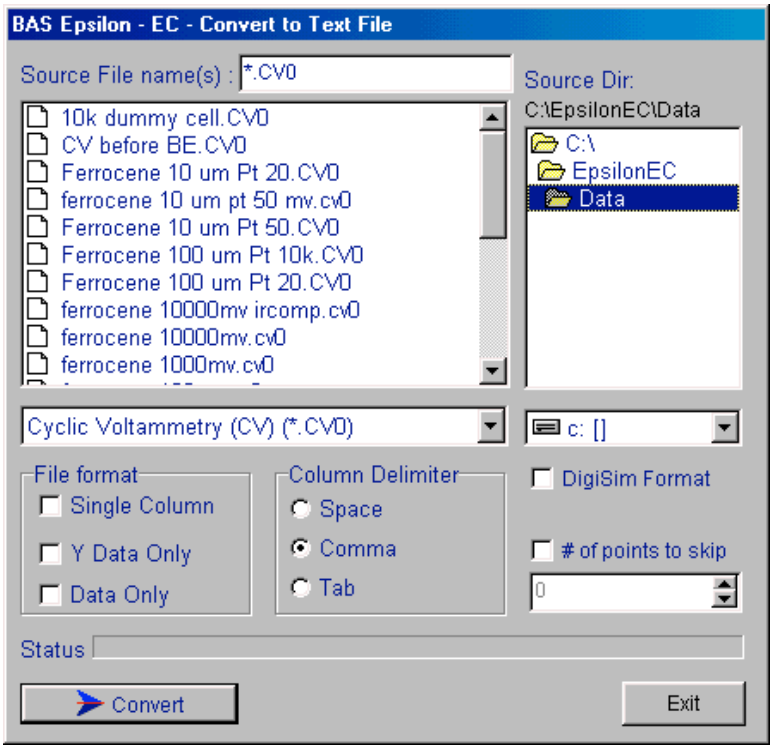


4.7 Stop degassing and stirring, but maintain a flow of N_2 in the cell. The solution should be stirred between experiments in order to restore initial conditions but it should *not* be stirred during the experiment.

4.8 After the experiment has been run, the voltammogram will be displayed. Note that the information about the experiment and the peak parameters are on the right side of the graph (this can be removed by clicking **Text Info (Right Column)** in the **Graph-Display** menu or the pop-up menu).



4.11 Use **Save** in the **File** menu (or the **F4** key) to save the data in the active experiment window (it should be noted that the each technique has its own extension; for example, .cv0 for cyclic voltammetry). The data is saved in a binary format, but, once saved, it can be converted to a number of different text formats using **Convert to Text File** in the **File** menu. Select the file(s) to be converted, the format, and the delimiter, and then click **Convert** to start the conversion.



4.14 **Calibration.** Add a small amount of ferrocene in the cell and repeat the degassing and stirring then repeat the scan. Standard values of ferrocene E°´ value can be found in the following publication : Connellly, N.G.; Geiger, W.E. *Chem. Rev.* **1996**, *96*, 877.

5. **How to clean the** electrodes

5.1 Working electrode : 1. CH_2Cl_2 2.MeOH

5.2 Auxiliary electrode: 1. CH_2Cl_2 2.MeOH and polish it with alumina following the polishing instructions provided in the polishing kit. (the polishing isn't necessary after each use)

5.3 Reference electrode: 1. CH_2Cl_2 2.distilled water. Store it in 3M NaCl solution.

6. **How to present the results**

See JAG paper # 257: Meyer, W; Amoroso, A.J.; Jaeger, M.; Le Bras, J.; Wong, W.-T., Gladysz, J.A. *J. Organomet. Chem.* **2000**, *616*, 44.

Complex	$E_{p,a}$ [V]	$E_{p,c}$ [V]	E° [V]	ΔE [mV]	$i_{c/a}$
2a	0.22	0.15	0.19	70	1
2b	0.17	0.06	0.12	110	1
2c	0.18	0.08	0.12	100	1
2d	0.24	0.12	0.18	120	1
2e	0.14	0.06	0.10	80	1
2f	0.01	-0.07	-0.03	80	1

(a) 1-3 × 10^{-3} M in 0.1 M Bu_4N^+ BF_4^-/CH_2Cl_2 at 22.5 ± 1 °C; Pt working and counter electrodes, potential vs Ag wire pseudoreference; scan rate 100 mV/s; ferrocene = 0.46 V.