

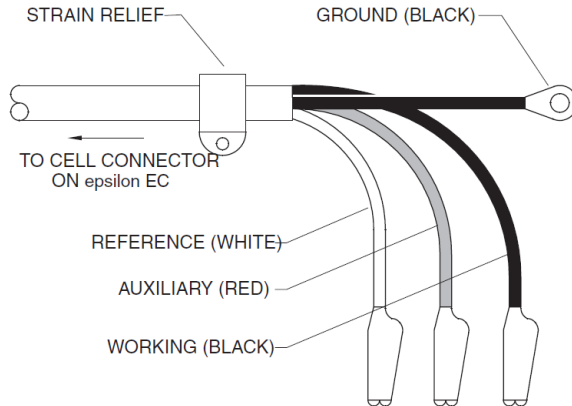
# Cyclovoltammetry

- **Log in:** the computer that controls the GC also controls the CV; use the "Group" account.
- **Turn on the EPSILON EC:** Switch on the analyzer first, then the cell. The link between the PC and the instrument will automatically be established. The status of the link will be displayed in the link dialog box, which will disappear once the connection has been established.



- **Open the valve of the argon cylinder:** The pressure should not exceed 5 psi!
- **Prepare solution(s) of the analyte(s):** Prepare a 0.1 M solution of electrolyte ( $\text{Bu}_4\text{N}^+$   $\text{BF}_4^-$  in the drawer labeled "CV supplies") with a freshly distilled solvent ( $\text{CH}_2\text{Cl}_2$ , THF,  $\text{CH}_3\text{CN}$ , TFT...). Recommended volume is 10-15 mL. Add sample (usually about 10 mg). The volume of the cell is 20 mL, so ~15 mL is the maximum volume you may use. Prepare air-sensitive sample on your line or in the glove box and take them to the CV in a syringe. Flush the cell with argon before adding the solution.
- **Prepare the electrodes:** Clean the electrodes before use. For good results polish the working electrode following the instructions provided in the cleaning manual (inside the CV drawer). Rinse the reference electrode with water, then the solvent you will be using.
- **Set up the cell:** Add a stirring bar and the sample solution to a clean "shot glass". Connect the three electrodes according to the colors and make sure they are well separated inside the cell.
  - Auxiliary electrode: Platinum wire (Red wire)
  - Working electrode: Platinum or Carbon (Black wire)

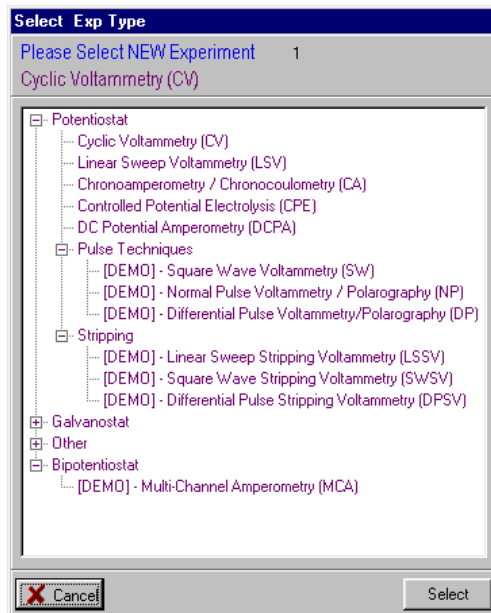
- Reference electrode: Ag/AgCl (White wire)



- One of the two Teflon hoses should be above the solution (providing an argon blanket), the other one submerged (degassing the solution).

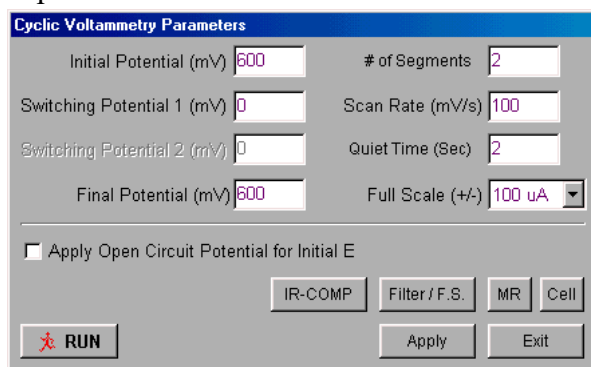
- **Run the CV:**

- Degas and stir the solution
- Click **New** in the **File** menu (or use the **F2** key) to set up a new experiment. The list of available techniques will be displayed

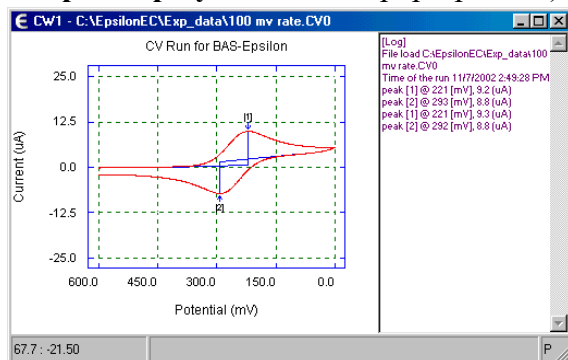


- Choose **Cyclic Voltammetry**

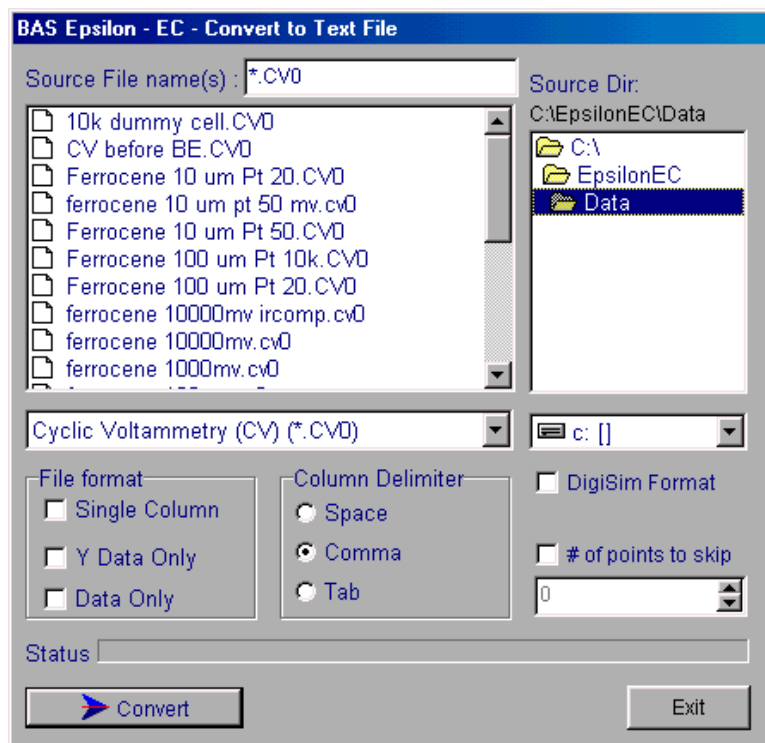
- The **Change Parameters** dialog box will now be displayed. Enter the desired parameters. Once these changes have been entered, an experiment using these parameters can be run by clicking the **RUN** button in this dialog box. If **Exit** is clicked before **Apply**, any changes in the parameters will 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.



- Stop degassing and stirring but maintain a flow of argon above the solution. The solution should be stirred between experiments in order to restore initial conditions, but it should not be stirred during the experiment.
- 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).



- Use **Save** in the **File** menu (or the **F4** key) to save the data in the active experiment window (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.



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- **Calibration with ferrocene:** After your measurement, add a small amount of ferrocene in the cell and repeat the degassing and stirring then repeat the scan. Standard values of ferrocene  $E^{\circ}$  value can be found in the following publication: Connolly, N.G.; Geiger, W.E. *Chem. Rev.* **1996**, *96*, 877.
- **Clean everything thoroughly:** Use the solvent you used for the measurement for the first rinse. Then  $\text{CH}_2\text{Cl}_2$  and finally MeOH. Rinse the reference electrode with water before returning it to the storage solution (3 M NaCl solution).

## **SPECIAL CONSIDERATIONS**

- Cleanliness is very important for a meaningful cyclic voltammogram:
  - Clean cells and electrodes thoroughly; for best results: polish the working electrode.
  - Avoid contaminating the electrolyte: Never return any extra electrolyte to the bottle. This salt is extremely clean and quite expensive.
  - All solvents should be of the best quality possible and degassed.
- The reference electrode is sensitive:
  - Make sure the stirring bar does not touch it. It can break (glass!)
  - Don't let it dry out. Immediately return it to the storage container if not in use.
- Don't bend the wires of the electrodes too much.
- Close the valve of the argon cylinder after use.
- Good resources: <https://sop4cv.com/index.html> and the *Chem. Ed.* article in the directory