Cyclic Voltammetry

How to start:
1. Log in to the computer (also used for the GC; use "Group" account).
2. Turn on analyzer (first) and cell (second). Power switches on the back.

The cell

- Main wires:
  - Red lead- Auxiliary Electrode (Platinum wire)
  - Black lead- Working Electrode (Platinum or Carbon)
  - White lead- Reference Electrode (Ag/AgCl)
- Stirring plate
  - Controlled via dial.
  - Can swing to the right.
- Plastic Tubing
  - Purge tubes.
  - Also controlled via dial on side.
 - Argon pressure must not be higher than 5 psi!

POWER

STIR
ON
Remote
OFF

RPM
400 450
350 500
300 550
250 600
200 650
150 700
100 750
50 800

PURGE
ON
Remote
Blanket

Running a CV

1. Prepare a 0.1 M solution of electrolyte (e.g. $Bu_4N^+F^-$) with dry solvent (i.e. CH_2Cl_2, THF, MeCN). Add ~ 10 mg of sample.

    - Use 10-15 mL. Cell volume is only 20 mL (smaller cell available).

2. Degas and stir solution (plastic tubing).

3. Rinse reference electrode with a) water and b) solvent of choice.

4. Connect electrodes by leads.

    - Red lead- Auxiliary Electrode (Platinum wire)

    - Black lead- Working Electrode (Platinum)

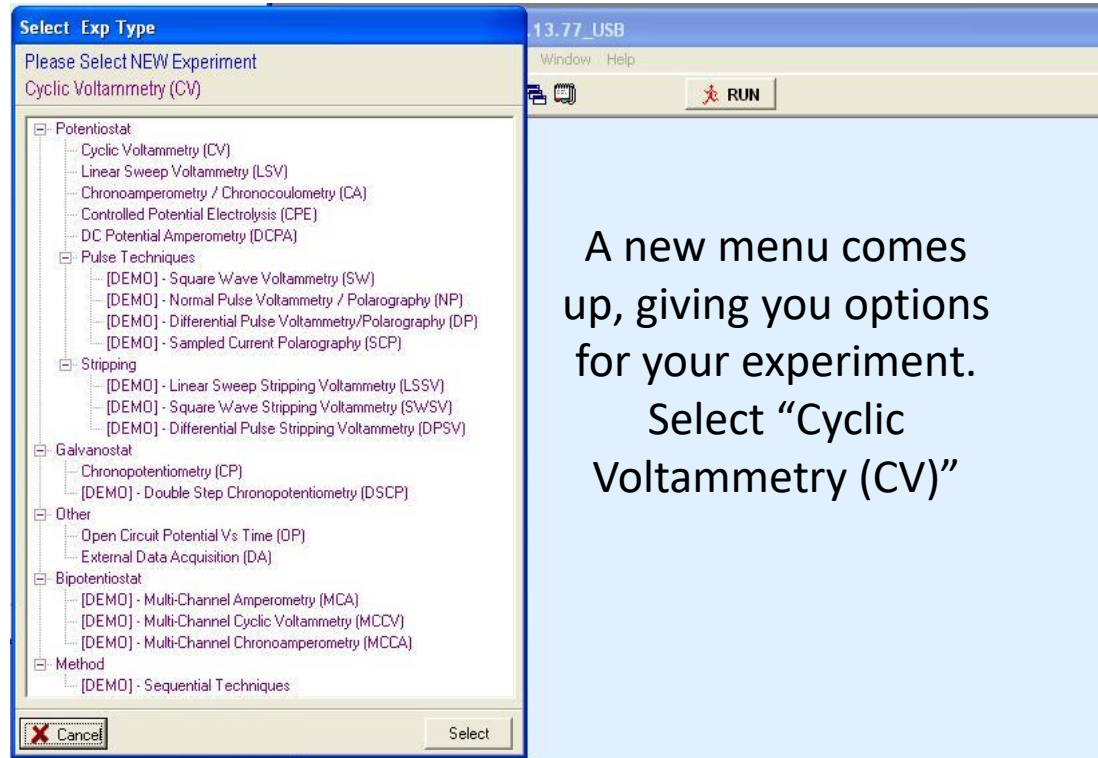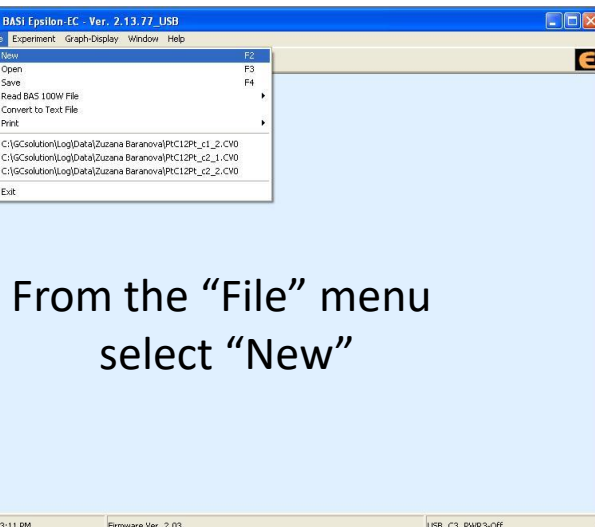   - White lead- Reference Electrode (Ag/AgCl)

CV Software

Use this icon to open the CV software ➡️

EpslionEC-USB
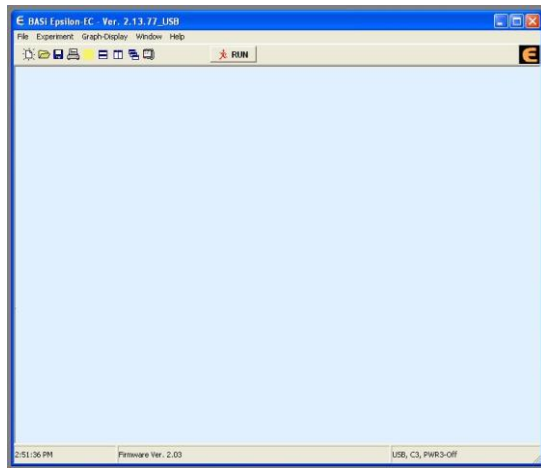
A new window like this pops up

From the "File" menu select "New"

A new menu comes up, giving you options for your experiment. Select "Cyclic Voltammetry (CV)"

Select Exp Type

Please Select NEW Experiment
Cyclic Voltammetry (CV)

- Potentiostat
  - Cyclic Voltammetry (CV)
  - Linear Sweep Voltammetry (LSV)
  - Chronoamperometry / Chronocoulometry (CA)
  - Controlled Potential Electrolysis (CPE)
  - DC Potential Amperometry (DCPA)
  - Pulse Techniques
    - [DEMO] - Square Wave Voltammetry (SW)
    - [DEMO] - Normal Pulse Voltammetry / Polarography (NP)
    - [DEMO] - Differential Pulse Voltammetry/Polarography (DP)
    - [DEMO] - Sampled Current Polarography (SCP)
  - Stripping
    - [DEMO] - Linear Sweep Stripping Voltammetry (LSSV)
    - [DEMO] - Square Wave Stripping Voltammetry (SWSV)
    - [DEMO] - Differential Pulse Stripping Voltammetry (DPSV)
- Galvanostat
  - Chronopotentiometry (CP)
  - [DEMO] - Double Step Chronopotentiometry (DSCP)
- Other
  - Open Circuit Potential Vs Time (OP)
  - External Data Acquisition (DA)
- Bipotentiostat
  - [DEMO] - Multi-Channel Amperometry (MCA)
  - [DEMO] - Multi-Channel Cyclic Voltammetry (MCCV)
  - [DEMO] - Multi-Channel Chronoamperometry (MCCA)
- Method
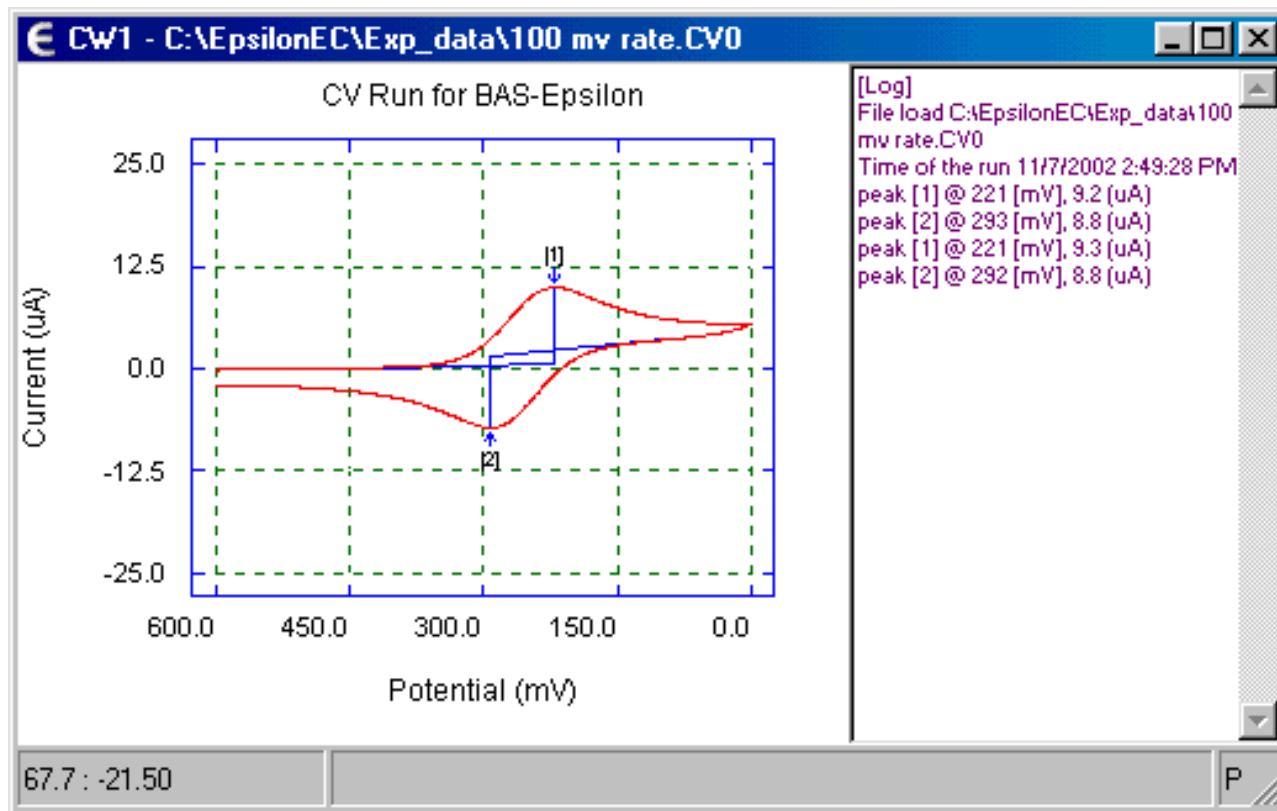 - [DEMO] - Sequential Techniques

Cancel   Select

CV Parameters

Enter parameters here! **Note: When done, hit "Apply", *then* "Exit", or your parameters will be lost!
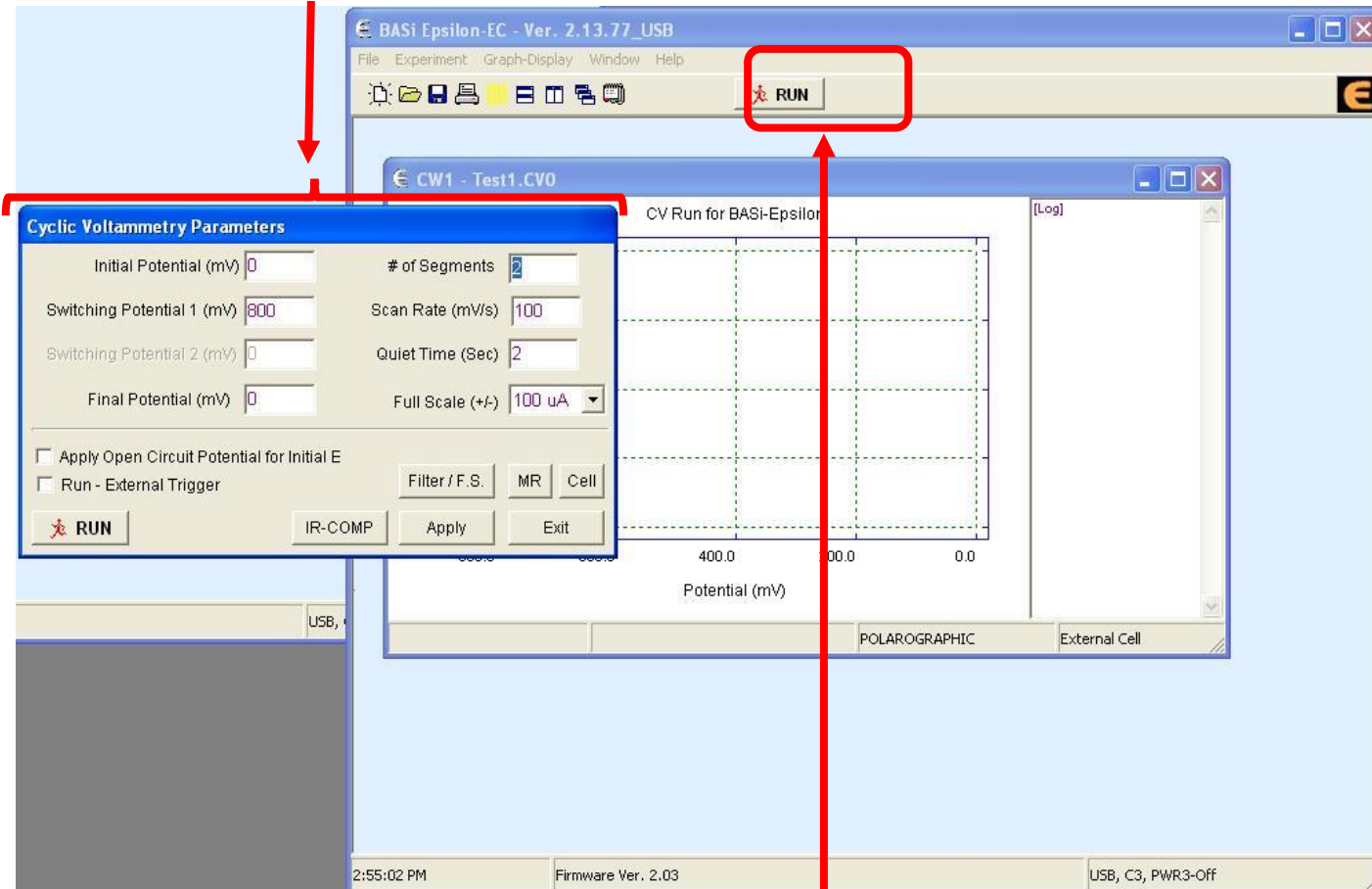
With parameters entered, click "Run" to gather data. At this point, continue the flow of Argon (blanket only, don't bubble through the solution), and stop the stirring.

Saving

From the "File" menu, you can "Save" the data. It's initially saved in binary format, but it can be converted to text using "Convert to Text File" under the "File" Menu.

Calibration and Clean-Up

- Calibrate using ferrocene as reference.
    - Add a small amount of ferrocene.
    - Degas, stir, and repeat the scan.
    - Standard values for ferrocene can be found here:
        - Connelly, N.G.; Geiger, W. E. *Chem. Rev.* **1996**, *96*, 877.

- CLEAN THE ELECTRODES!
    - Working electrode:
        1. Rinse with solvent used, then CH_2Cl_2
        2. Rinse with MeOH
    - Auxiliary Electrode
        1. Rinse with used, then CH_2Cl_2
        2. Rinse with MeOH
        3. Polish with Alumina (follow instructions in kit; not always necessary)
    - Reference Electrode
        1. Rinse with solvent used, then CH_2Cl_2
        2. Rinse with distilled water
        3. Store in 3M NaCl solution

What can go wrong?

- Cleanliness is extremely important for a meaningful cyclovoltammogram:
  - 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.
  - Run a CV on only solvent and electrolyte to check for contaminants.
- Bubbles on the electrodes can ruin your results. Always check.
- 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.
- Read the *Chem. Ed.* article (in the directory) and check out:
  https://sop4c.com/index.html