

Supplementary Materials: Instructions to set up the MicroLab / Pine interface:

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1. Connect the PINE / MicroLab interface:

Plugs coming from PINE electrode chip (male) to model 290 MicroLab (female)

Male red → female red (Voltage In)

Male white → female yellow (Current In)

Male blue → female blue (DAC Voltage Out)

The male yellow remains unplugged; the female black receives no plug.

2. Plug the Model 290 sensor adaptor into the MicroLab FS522 or 507 General-purpose Input A. Open MicroLab.exe.

3. Set up the MicroLab FS522 to acquire data:

In the “Data Sources / Variables” box, click the “Add Sensor” button. In the “Choose Sensor” dialog box that appears, choose “DAC Output” from the drop down menu, click on the icon that represents the plug into which the Model 290 is connected, then click “Finish”.

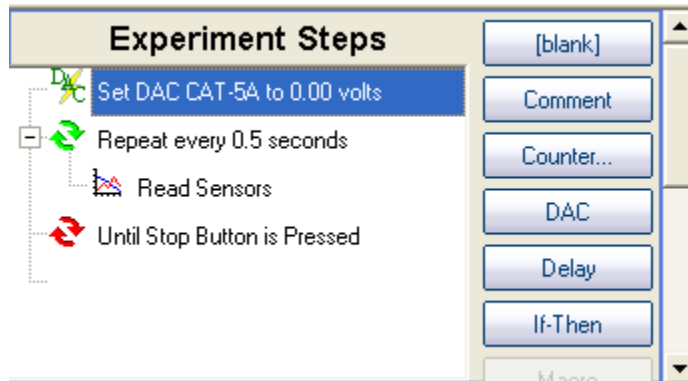
Again, in the “Data Sources / Variables” box, click the “Add Sensor” button. In the “Choose Sensor” dialog box that appears, choose “Current”, click on the button next to +/- 2.5 mA sensitivity, and finally click on the icon that represents the plug into which the Model 290 is connected, then click “Finish”.

Again, in the “Data Sources / Variables” box, click the “Add Sensor” button. In the “Choose Sensor” dialog box that appears, choose “Voltage”, and click on the icon that represents the plug into which the Model 290 is connected, then click “Finish”.

4. Program the experiment: The following sequence of programming steps will carry out a CV scan that begins at 1.0 Volt vs. the reference electrode, decrements the potential each 0.2 second in steps of 0.02 V to -1 V while reading the current at each potential, and then switches, incrementing the potential by 0.02 V each 0.2 second and reading each current until the potential returns to 1.0 V. The current - potential plot can be generated in real time in the MicroLab program by dragging the V and I sensors to the graphing window.

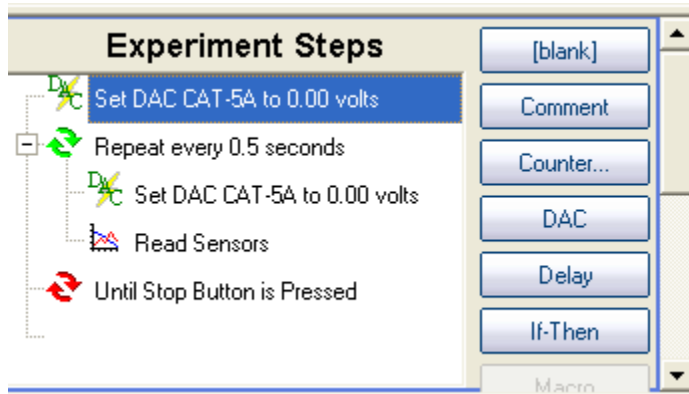
Right click on experiment steps → show programming buttons.

Click on the “DAC” button. “Set DAC CAT-5A to 0.00 volts appears” in the experiment steps. Click and drag this experiment step to the beginning of the experiment steps:



Double click on the “Set DAC CAT-5A to 0.00 volts” experiment step. A “Set DAC” dialog box appears. Set the voltage at which you wish to start your sweep (First set it at 1V).

Click on the “DAC” button again. “Set DAC CAT-5A to 0.00 volts” appears in the experiment steps. Click and drag this experiment step to right after the “Repeat every 0.5 seconds” step:

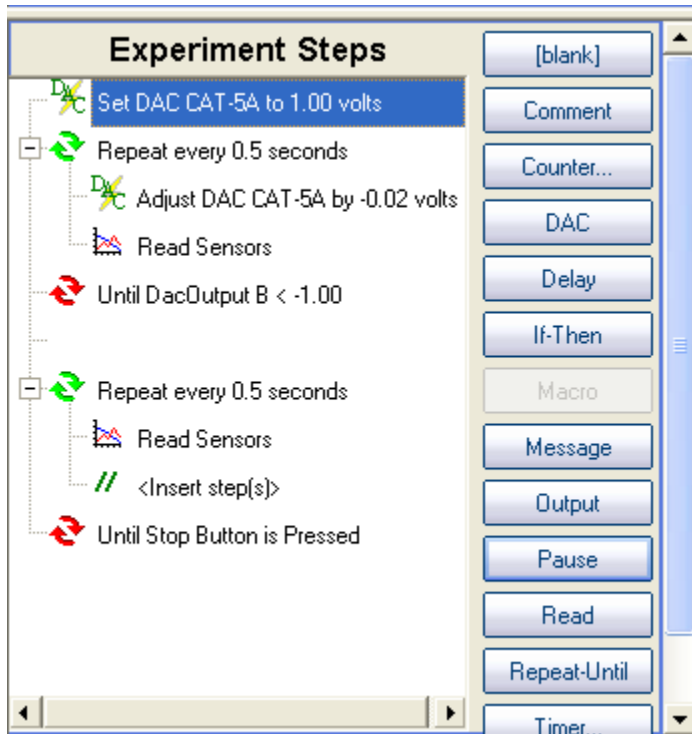


Double click on the “Set DAC CAT-5A to 0.00 volts” experiment step you just added. The “Set DAC” dialog box appears. Click the button next to “adjust by +/-“, and then enter the voltage step. (I set it at -0.02 V).

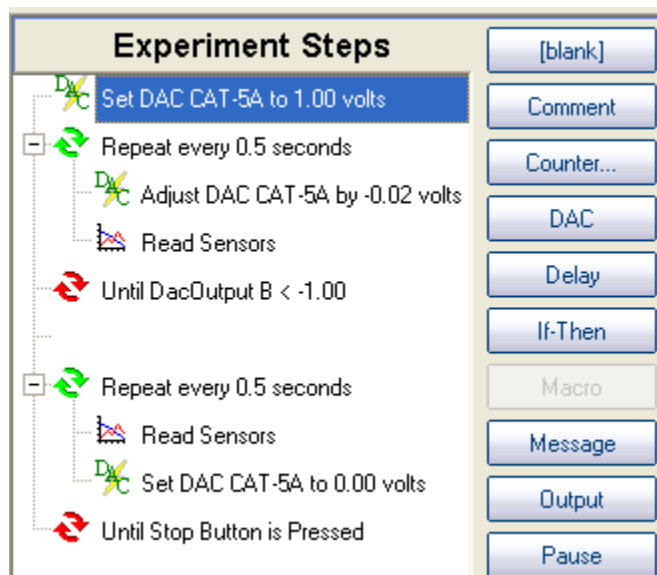
Double click the “Until Stop Button is Pressed” experiment step. An “Until” dialog box appears. Click the button next to “Condition True...”, then click on the “Edit Condition” button.

A “Condition” dialog box appears. Enter information in the condition dialog box so that the condition is **DAC is less than -1 V**: Select DAC from the first drop down menu, “<” from the second drop down menu, and “constant” from the third drop down menu. Enter -1 in the space for constant. Click OK, and then OK again. The experiment is now set to start at 1 V and sweep down to -1 V by 20 mV increments.

Click on the “Repeat Until” button. Two steps appear in the “Experiment Steps” box. One step reads “Repeat every 0.5 s”. The other reads “Until Stop Button is Pressed”. Click on the “Read” button. Drag the “Read Sensors” step that appears in between the steps that read “Repeat every 0.5 s” and “Until Stop Button is Pressed”:



Click on the “DAC” button again. “Set DAC CAT-5A to 0.00 volts” appears in the experiment steps. Click and drag this experiment step in between the steps that read “Repeat every 0.5 s” and “Until Stop Button is Pressed”. Also, click on the step that reads “Insert step(s)” and press delete (see next page):



Double click on the “Set DAC CAT-5A to 0.00 volts” experiment step you just added. The “Set DAC” dialog box appears. Click the button next to “adjust by +/-“, and then enter the reverse voltage step. (I set it at 0.02 V).

Double click the “Until Stop Button is Pressed” experiment step. An “Until” dialog box appears. Click the button next to “Condition True...”, then click on the “Edit Condition” button.

A “Condition” dialog box appears. Enter information in the condition dialog box so that the condition is **DAC is greater than 1 V**: Select DAC from the first drop down menu, “>” from the second drop down menu, and “constant” from the third drop down menu. Enter 1 in the space for constant.

The experiment is now set to start at 1 V, sweep to -1 V by 20 mV increments, and then back up to 1 V by 20 mV increments.

Before you begin the experiment, we need to set some options so as to minimize noise. In the toolbar, select Setup Options → Low-Level Options. A dialog box appears. Click the button next to 1/5 second. Click OK.

Assuming your sample is prepared, you’re ready to run, so click Start! We have found that no electrode pre-treatment is necessary with compounds we have run (aqueous solution), but sometimes it is necessary to bubble solutions in N₂ to expunge O₂ before running samples.

For data analysis, we usually export into a spreadsheet and do some signal averaging to get smoother data.

Potassium ferricyanide is often used as the prototype reversible electrochemical system for cyclic voltammetry. ("Cyclic Voltammetry Experiment", Van Benschoten, James J.; Lewis, Jane Y.; Heineman, William R.; Roston, Daryl A.; Kissinger, Peter T., JCE, 1983, 60, 772.) The figure below (see next page) shows the CV of a 1 mM ferricyanide ($K_3Fe(CN)_6$) solution with 1 M KNO_3 as supporting electrolyte, without bubbling in N_2 . It was obtained using the MicroLab program illustrated, though the axis scales were modified with the MicroLab software after the experiment.

