
Epsilon ECLipse™

Potentiostat / Galvanostat / Bipotentiostat

Chemical Test Procedure



August 2022





2701 KENT AVENUE

WEST LAFAYETTE, INDIANA 47906 USA WWW.BASINC.COM

Thank you for purchasing the Epsilon EClipse™. Your new BASi instrument will provide years of service for your electrochemical requirements. The Epsilon EClipse™ was fully tested electronically and with a chemical test before shipping. The chemical test procedure (shown in the manual) and results are included in the folder. Please follow the procedure below when setting up your instrument.

A full electronic manual is available at www.basinc.com/manuals.

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This instrument, either wholly or in part, is manufactured for research purposes only. Use for medical diagnosis is not intended, implied, or recommended by the manufacturer. Use for this purpose and accountability for same rest entirely with the user.



PURPOSE

The purpose of this test is to perform an examination of the epsilon performance on a “live” electrochemical cell and a three-electrode assembly. From the outputs of the experiments listed below, the customer can verify that the instrument is working properly on arrival and can gain some experience in its operation.

INSTRUMENT INSTALLATION

The Epsilon should be plugged into the mains supply (N.B. first check that the fuse panel has been set to the correct voltage). The Epsilon EClipse™ system requires a computer running Windows 7 or later. Connect a standard USB cable between any USB port on the computer and the USB port (Figure A) on the back of the Epsilon EClipse™. Please refer to the Epsilon Eclipse Quick Start Guide for Software Version 2.13.77 or 3.0.84. The Epsilon EClipse software version 2.13.77 should be used to conduct this procedure and is included on a USB stick with each purchase. For the most up-to-date version of the software, please email ec@basinc.com. If available, a BASi cell stand should be connected to the CELL STAND (Figure A) port of the epsilon. If you are going to use a computer not supplied BASi, then you will have to install the software on your computer.

TEST SOLUTION

2 mM potassium ferricyanide with 1 M potassium nitrate in water.

PREPARATION OF TEST SOLUTION

1. Weigh 16.5 mg potassium ferricyanide and place in a 25 mL volumetric flask.
2. Weigh 2.53 g potassium nitrate and add to the same volumetric flask.
3. Add about 20 mL deionized water to dissolve the potassium ferricyanide and potassium nitrate.
4. Dilute to 25 mL with deionized water.

CELL (C3 CELL STAND)

Platinum (PTE) Working Electrode (Black lead)
Platinum Wire Auxiliary Electrode (Red lead)
Silver/Silver Chloride RE-5 Reference Electrode (White lead)

Add 10-15 mL of the ferricyanide solution to the cell vial and place in the cell holder (see Cell Stand instructions). Polish the PTE with 0.05 mm alumina following the polishing instructions provided in the polishing kit.

If the RE-5 electrode is new, carefully remove the yellow plastic sheath before use. In addition, there may be air bubbles inside the electrode next to the CoralPor™ frit; these must be displaced (by flicking the electrode). RE-5 electrodes must be stored in 3M sodium chloride when not in use.

Various operations of BASi Cell Stands (i.e., purge and stir for the C3 Cell Stand; purge, stir, and knock/ dispense for the CGME Mercury Electrode) can be controlled from the epsilon (Figure B).

1. Connect the Cell Stand (C3 or CGME) to the epsilon using i) the 25-pin ribbon cable provided with the Cell Stand between the CELL STAND port on the rear panel of the epsilon and the REMOTE port of the Cell Stand and ii) the LEMO-LEMO cable provided with the Cell Stand between the CELL port of the Cell Stand and the W1 port of the epsilon.
2. Select the appropriate Cell Stand (C3 - Cell Stand or CGME SMDE Mode) in the Setup / Manual Settings (I/O) dialog box (F1) in the Experiment menu as shown in Figure B on the next page.

FIGURE A BASi CELL STAND AND BASi EPSILON ECLIPSE CONNECTIONS

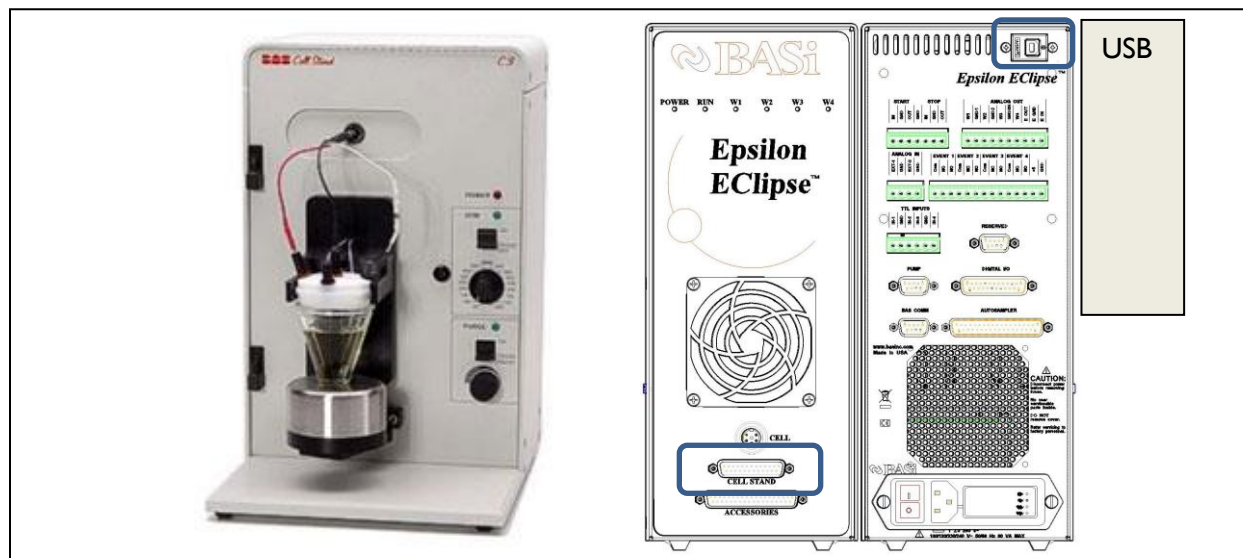
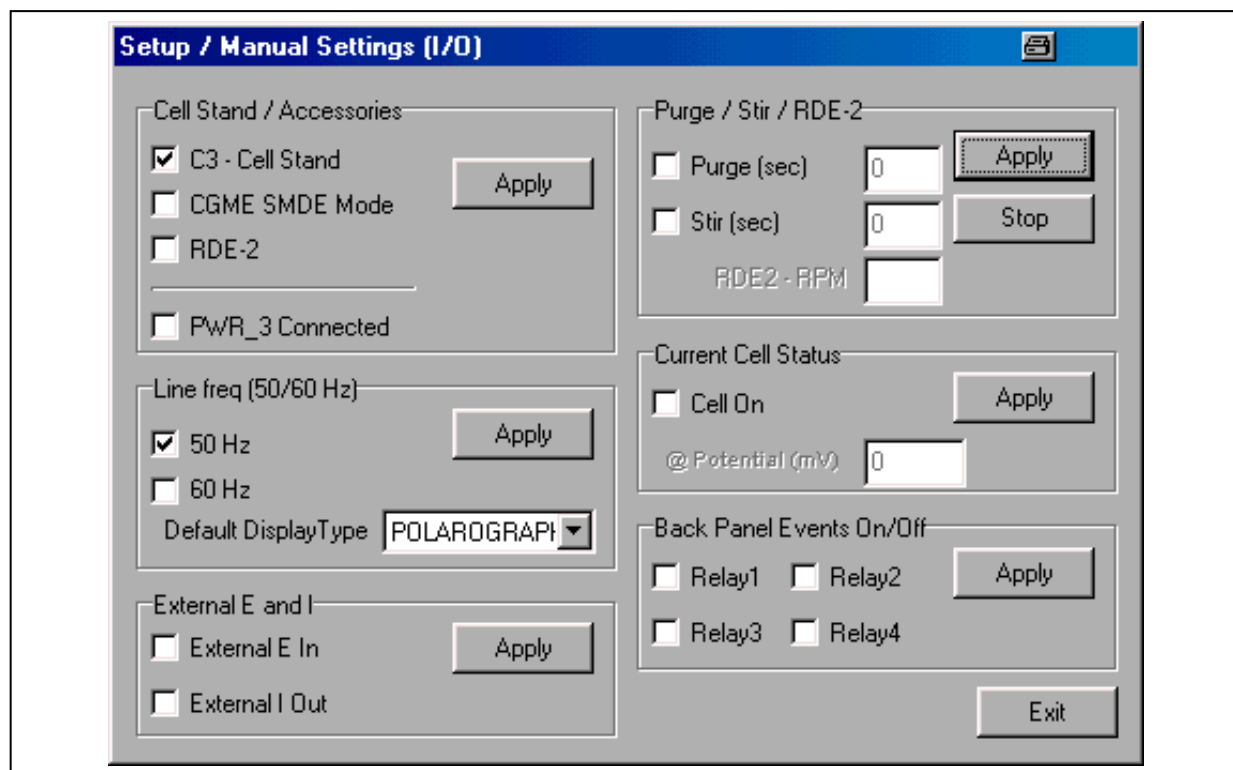


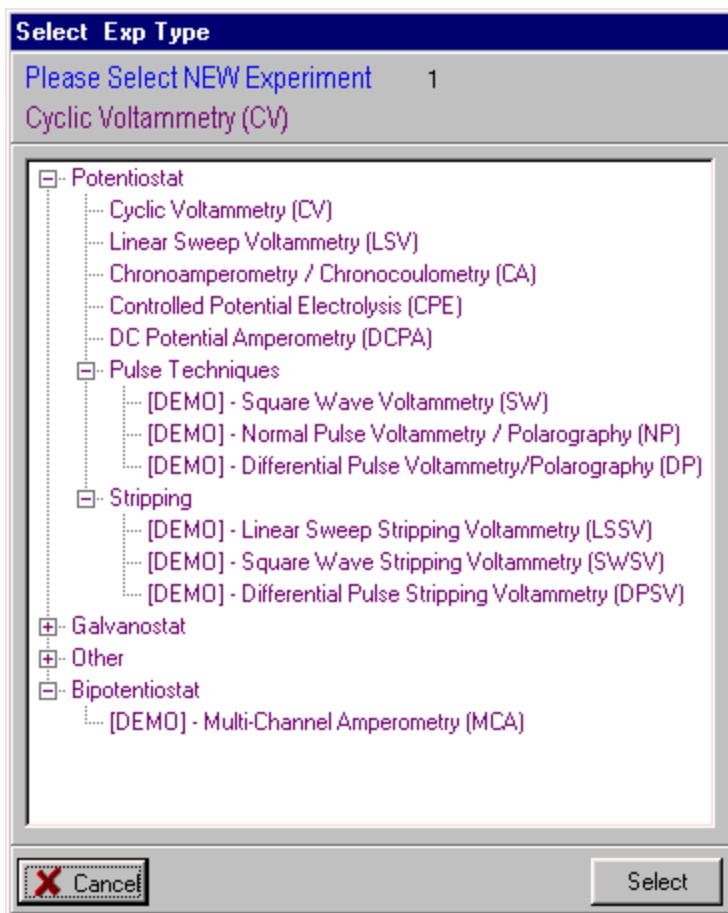
FIGURE B SETTING UP C3 CELL STAND FROM THE MANUAL SETTINGS



PROCEDURE

1. Turn the Power switch on the epsilon to on.
2. Open the epsilon software by clicking the epsilon icon. The CS Dialog box will appear and will show the progress of establishment of the communication between the epsilon and the PC. If the link is established, the CS Dialog box will automatically disappear. If the link is not established, click the Retry button. If the link is lost after being established, use Reconnect Epsilon in the Experiment menu to reestablish the link.
3. Click New in the File menu (or use the F2 key) to set up a new experiment. The list of available techniques is displayed (Fig 1) (this list can also be accessed using Select New Experiment in the Experiment menu). It should be noted that there are some techniques that are labeled as DEMO. This label indicates that this technique is NOT active on this Epsilon. However, it is possible to load a data file for that technique to examine the parameters and the typical output. If the RUN button is clicked when a DEMO data file is displayed in the active window, an error message will be shown. The technique list shown in Figure 1 is the list for the basic epsilon (i.e., pulse techniques, stripping techniques, and multi-channel amperometry are NOT available).

FIGURE 1 LIST OF TECHNIQUES



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4. Select Cyclic Voltammetry. The Change Parameters dialog box will now be displayed. Enter the values shown in Figure 2. Note that Switching Potential 2 is not required since there are only 2 segments (Initial Potential to Switching Potential 1 to Final Potential). Once these changes have been entered, an experiment using these parameters can be run by clicking the RUN button in this dialog box. If an experiment is not to be run immediately, but these parameters are to be saved, then the Apply button must be clicked before exiting the 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 using either Run in the Experiment menu, in the pop-up menu obtained by clicking the RIGHT mouse button (Figure 3), the RUN icon on the Tool Bar, or the F5 key. This icon will change to STOP during the experiment and can be used to abort the experiment.

FIGURE 2. CYCLIC VOLTAMMETRY PARAMETERS

Cyclic Voltammetry Parameters

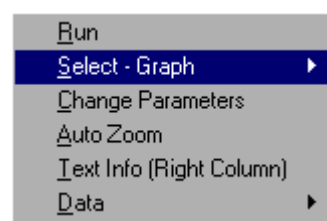
Initial Potential (mV)	600	# of Segments	2
Switching Potential 1 (mV)	0	Scan Rate (mV/s)	100
Switching Potential 2 (mV)	0	Quiet Time (Sec)	2
Final Potential (mV)	600	Full Scale (+/-)	100 uA

Apply Open Circuit Potential for Initial E

IR-COMP Filter / F.S. MR Cell

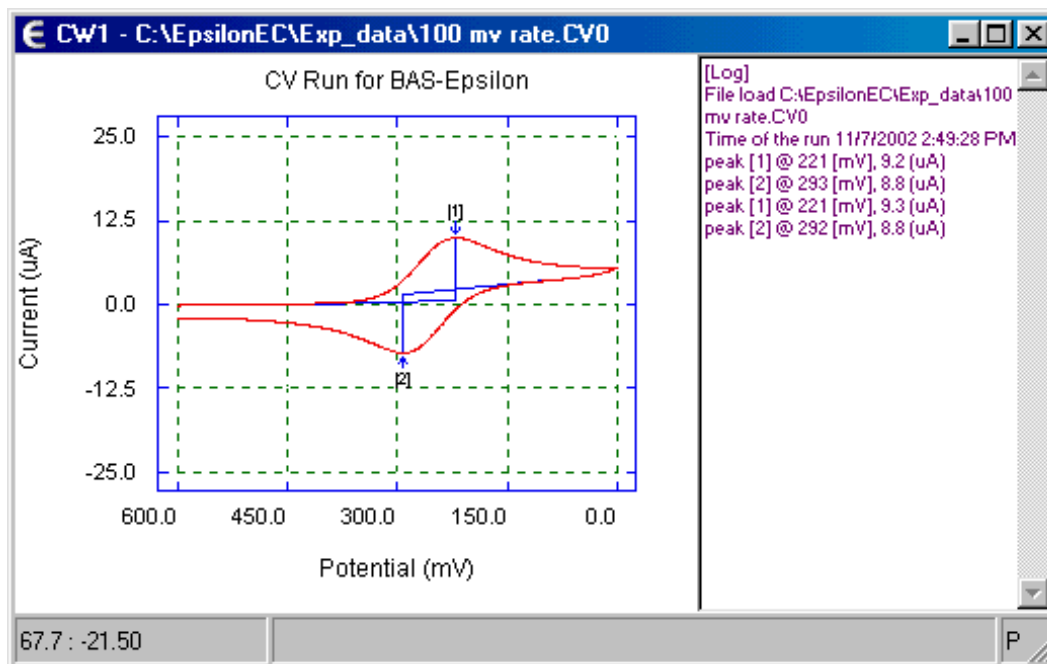
RUN Apply Exit

FIGURE 3 . RUN OPTION



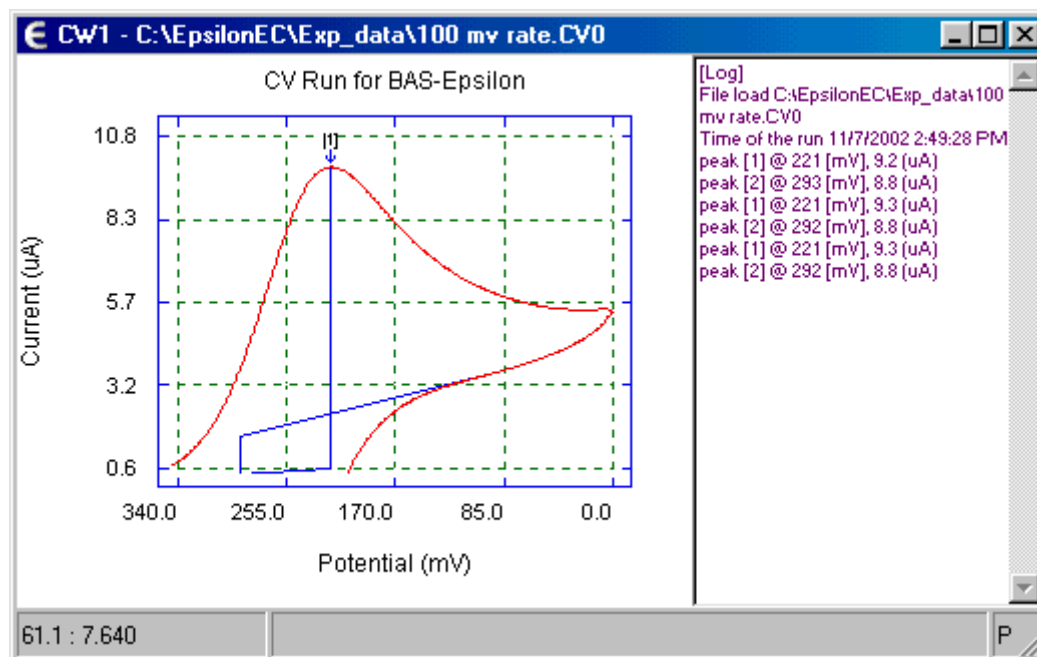
5. After the experiment has been run, the voltammogram will be displayed (Figure 4). Note the information about the experiment and the peak parameters on the right side of the graph (this can be removed by clicking Text Info (Right Column) in the Graph-Display menu or the pop-up menu).

FIGURE 4. CYCLIC VOLTAMMETRY



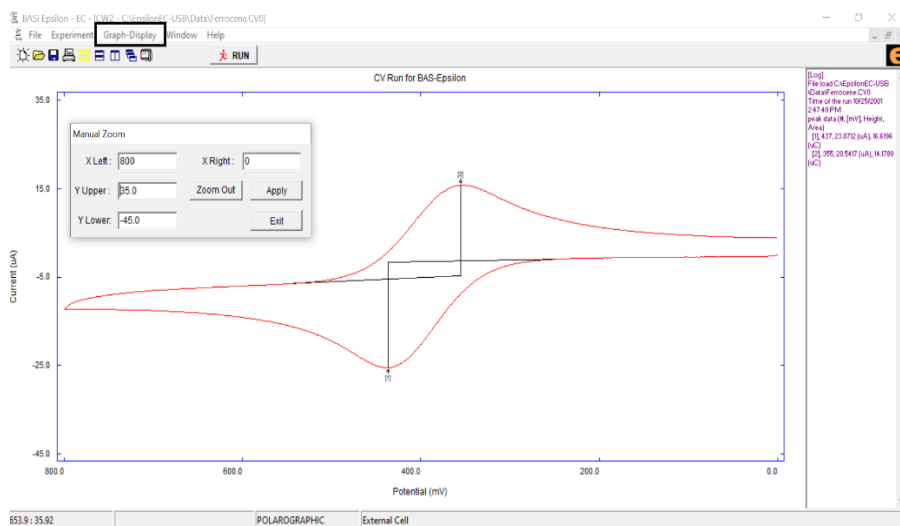
- A specific area of the graphic can be enlarged by either using the mouse cursor (and the left mouse button) to define the area (Figure 5) or entering the x and y values in the Manual Zoom dialog box (Figure 6). The original graph can be restored using Auto Zoom in the Graph-Display menu.

FIGURE 5. ZOOMING IN ON A SPECIFIC AREA



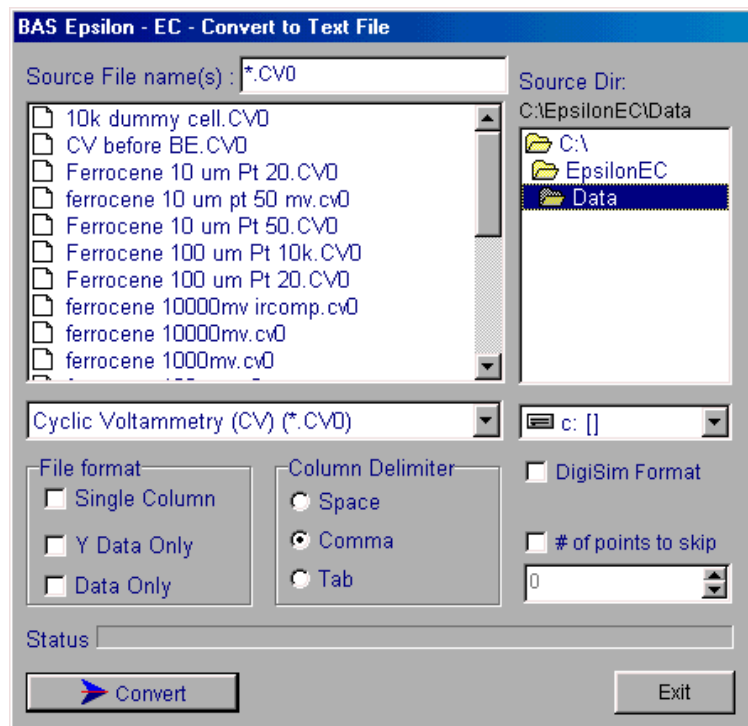
PROCEDURE (CONTD.)

FIGURE 6. MANUAL ZOOMING FROM GRAPH DISPLAY



7. Use Save in the File menu (or the F4 key) to save the data in the active experiment window (it should be noted that 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 several different text formats using Convert to Text File in the File menu (Figure 7). Select the file(s) to be converted, the format, and the delimiter, and then click Convert to start the conversion.

FIGURE 7. CONVERT TO TEXT DIALOG BOX



PROCEDURE (CONTD.)

- Various experimental data can be entered into the Notes in the Experiment menu (Figure 8). These notes will also be saved when the experimental data is saved.

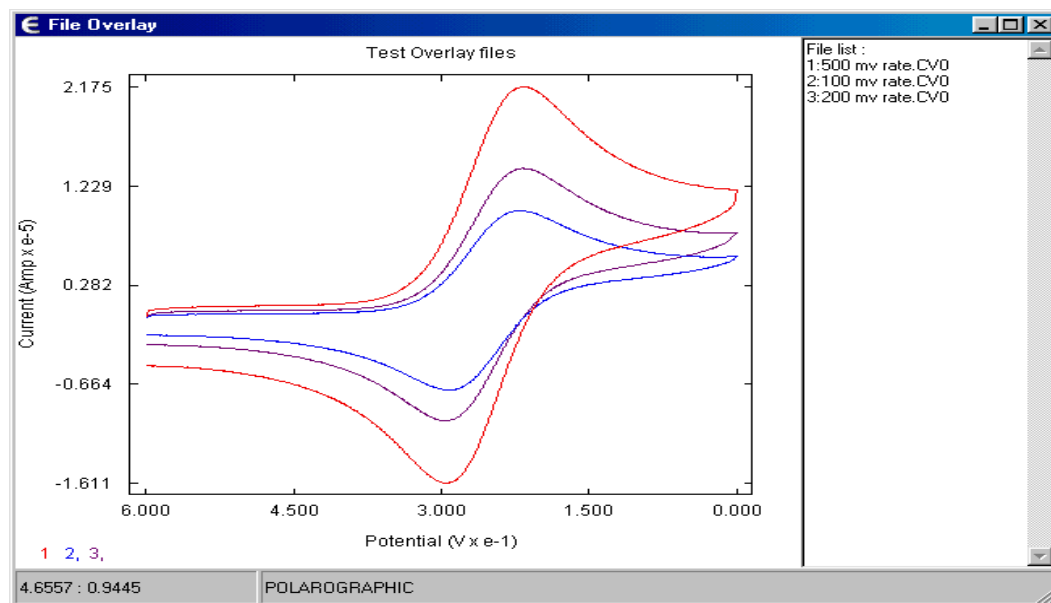
FIGURE 8. NOTES IN EXPERIMENTAL MENU

- Open the Change Parameters dialog box (using Change Parameters in either the Experiment menu, the pop-up menu, or the F6 key), and change the Scan Rate to 200 mV/s. Run the experiment again. Note that the new data is displayed in the experiment window; that is, the new experimental data has replaced the old experimental data. Save this data. Change the scan rate to 500 mV/s, run the experiment, and save the data.
- The three data sets run at different scan rates can be displayed on the same sets of axes using the File Overlay function in the Graph-Display menu. The first time this function is selected after the software is started, an empty set of axes is displayed. Subsequent activations will display the previously selected data sets. Click the right mouse and select Setup from the pop-up menu. The File Overlay Setup dialog box will be displayed (Figure 9).

FIGURE 9. FILE OVERLAY SETUP DISPLAY BOX

11. The scaling of the axes is determined by the Base File. Therefore, when comparing voltammograms with the same potential range, the voltammogram with the highest current should be selected as the Base File. In this example, the cyclic voltammogram run at a scan rate of 500 mV/s should be the Base File. Select the name of this data file and click New. This data file is now displayed on the axis set (which remains in the background). The other two data files (cyclic voltammograms run at 100 and 200 mV/s) should now be selected (using standard Windows functions). Once selected, click Add to display these data sets on the axis set. Click Exit to remove the Setup dialog box. The overlaid data sets can now be viewed (Figure 10).

FIGURE 10. OVERLAYED CYCLIC VOLTAMMOGRAMS



12. Click New in the File menu and select Cyclic Voltammetry again. A new experiment window will appear, together with an associated Change Parameters dialog box. Enter the parameters shown in Figure 11 and run this experiment. Since there are 3 segments in this experiment, all 4 potential parameters must be defined (Initial Potential to Switching Potential 1 to Switching Potential 2 to Final Potential).

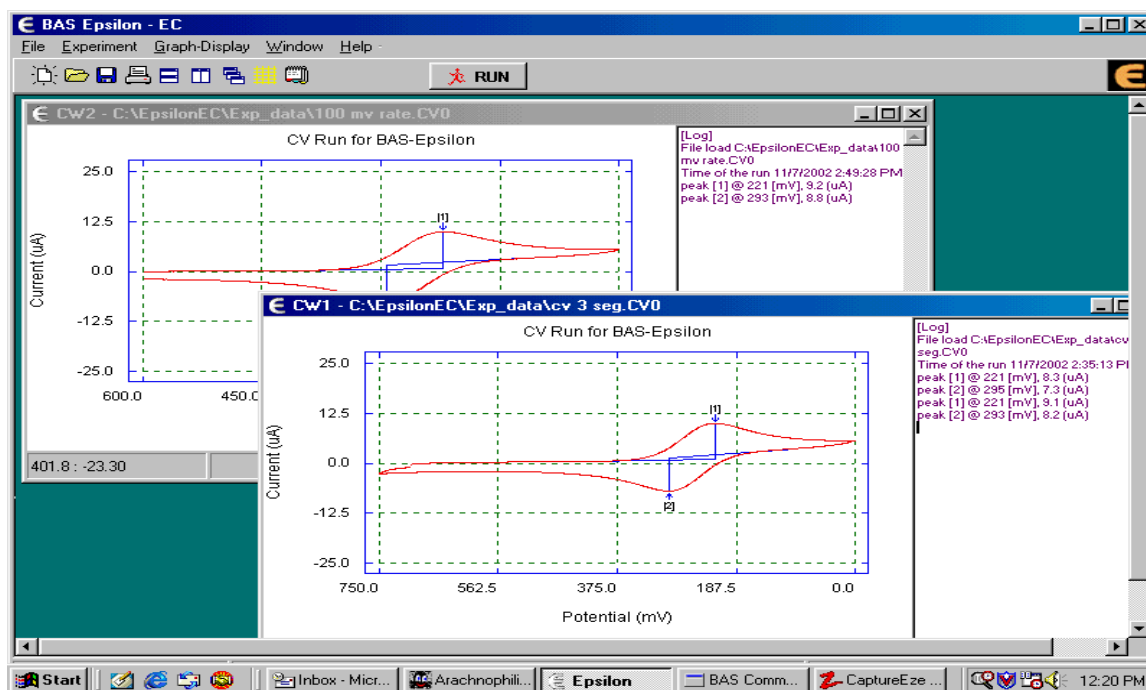
FIGURE 11. CYCLIC VOLTAMMETRY PARAMETERS

Cyclic Voltammetry Parameters			
Initial Potential (mV)	700	# of Segments	3
Switching Potential 1 (mV)	0	Scan Rate (mV/s)	100
Switching Potential 2 (mV)	750	Quiet Time (Sec)	2
Final Potential (mV)	700	Full Scale (+/-)	100 uA
<input type="checkbox"/> Apply Open Circuit Potential for Initial E			
IR-COMP		Filter / F.S.	MR Cell
RUN		Apply	Exit

PROCEDURE (CONTD.)

13. When this experiment has finished, both experiment windows are displayed in the main epsilon window (Figure 12). Up to 6 experiment windows can be displayed. However, only one experiment window can be active at any one time, and any epsilon functions and operations are applied only to the data in the active experiment window.

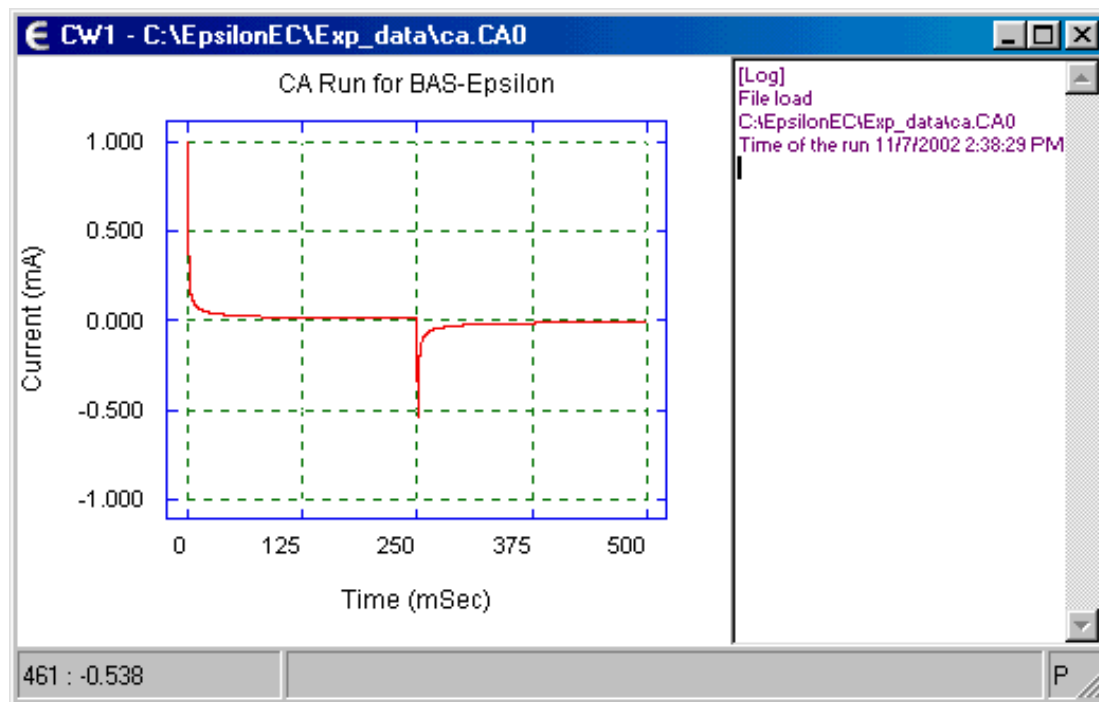
FIGURE 12. MULTIPLE VIEW OF EXPERIMENTAL WINDOWS



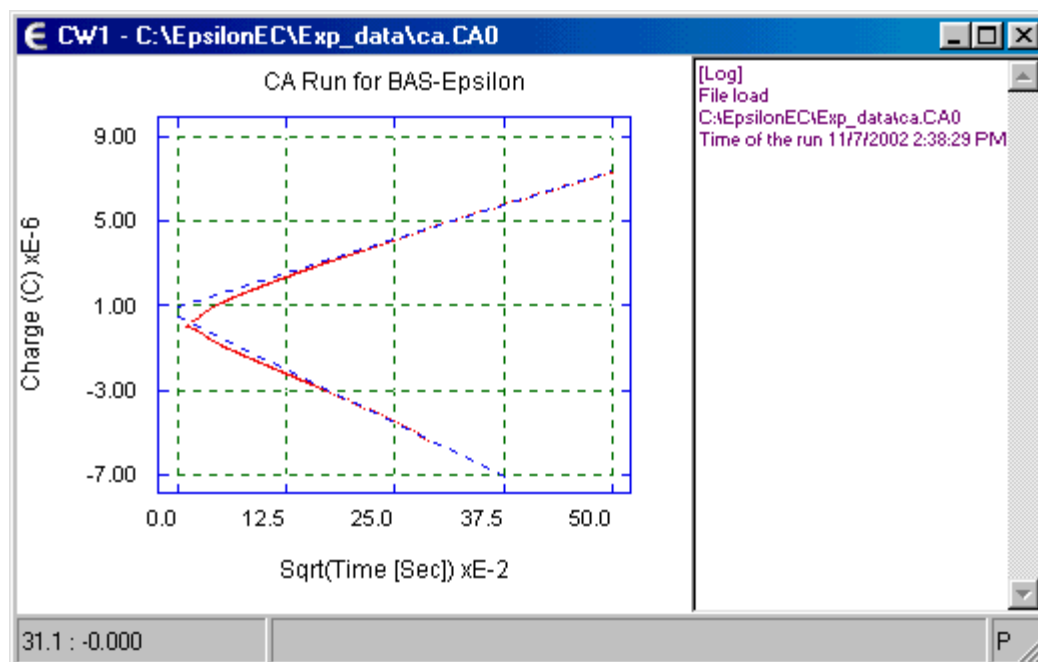
14. Click New and select Chronoamperometry / Chronocoulometry from the list of techniques. A third window will appear, together with an associated Change Parameters dialog box. Enter the parameters shown in Figure 13. Running the experiment generates the plot shown in Figure 14.

FIGURE 13. CHRONOAMPEROMETRY / CHRONOCOULOMETRY SET UP PARAMETERS

Chronoamperometry / Chronocoulometry Parameters	
Initial Potential (mV)	700
Quiet Time (Sec)	0
First Step E (mV)	0
First Step Time	250
Second Step E (mV)	700
Second Step Time	250
Full Scale (+/-)	1 mA
Time Units	mSec
Sample Interval = 100 us	Max # of Points in a Step
	4000
<input type="checkbox"/> Apply Open Circuit Potential for Initia Filter / F.S. MR Cell	
RUN Apply Exit	

FIGURE 14. CHRONOAMPEROMETRY / CHRONOCOULOMETRY GRAPH

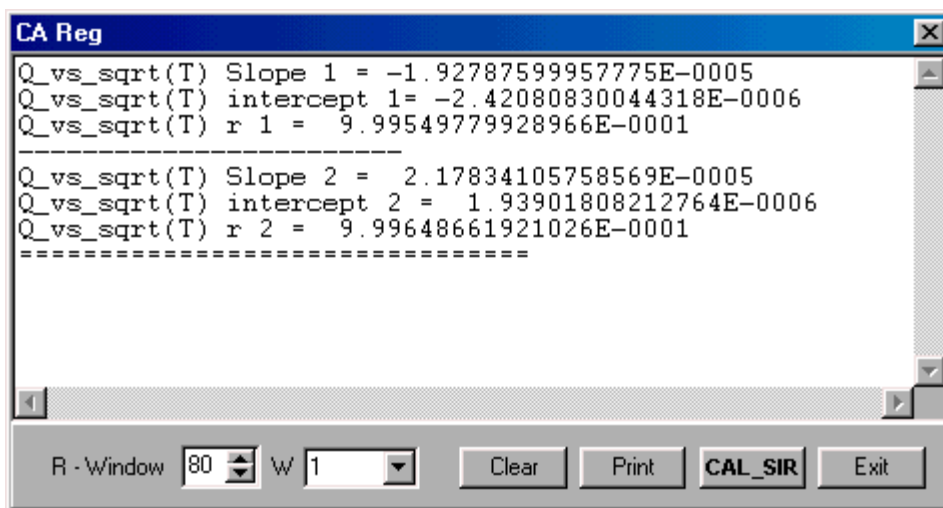
15. The data from a Chronoamperometry/Chronocoulometry experiment can be plotted in several different formats, which can be selected using Select Graph in the pop-up menu in the chronoamperometry / Chronocoulometry experiment window. The Q vs. sqrt(T) plot is shown in Figure 15.

FIGURE 15. CHRONOAMPEROMETRY / CHRONOCOULOMETRY GRAPH Q VS SQRT (T)

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16. The epsilon software can calculate the slope and intercept of the linear Q vs \sqrt{t} plot when it is displayed. Selecting Data - Calculate CA-SIR from the pop-up menu generates the information box shown in Figure 16. The lines used for the linear fitting are the dashed blue lines in Figure 15 (note that the first 20% of the data points are not used in the calculation, due to interference from the charging current and other experimental artifacts).

FIGURE 16. CHRONOAMPEROMETRY / CHRONOCOULOMETRY - SLOPE & INTERCEPT CALUCLATION



17. Click New and select Chronopotentiometry. A fourth experiment window will be opened, together with the associated Change Parameters dialog box. Enter the parameters shown in Figure 17, then run the experiment (note the sign convention for the current - cathodic (reduction) currents are positive for polarographic). Typical data for a Chronopotentiometry experiment is shown in Figure 18.

FIGURE 17. CHRONOPOTENTIOMETRY SETUP PARAMETERS

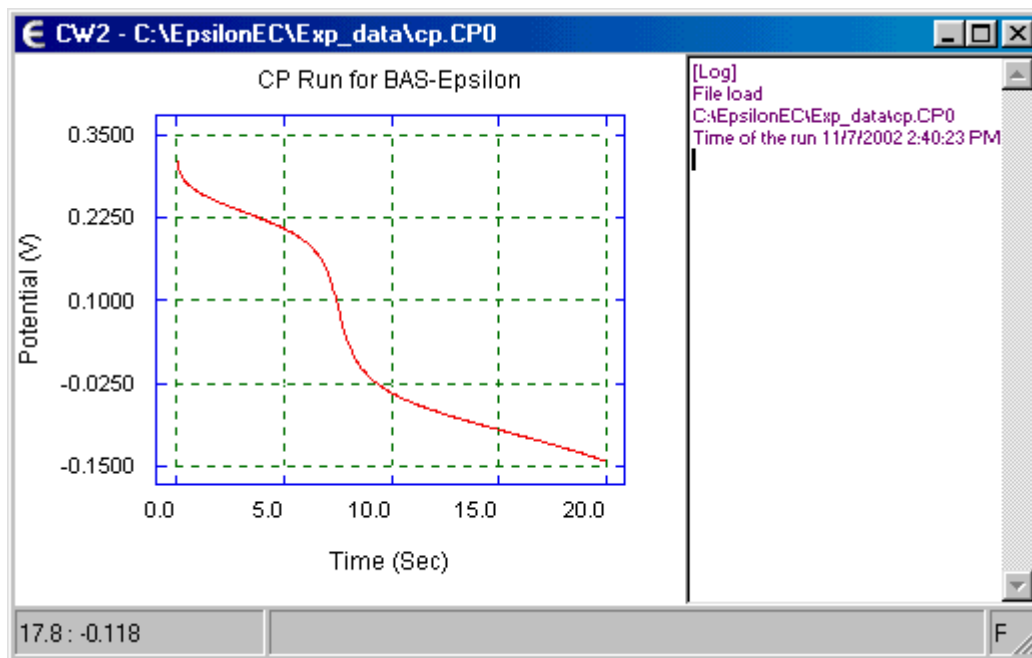
The screenshot shows the "Chronopotentiometry Parameters" dialog box with the following settings:

- Applied Current: 4
- Time: 20
- I - Units: uA
- T-Units: Sec
- Potential Range: +/- 1 V
- Sample Interval: 0.05 sec
- Applied Current Convention: POLAROGRAPHIC
- Noise Filter Value: 100 Hz
- End Condition E Limit:
 - Upper Limit (mV): -1000
 - Lower Limit (mV): 3

At the bottom, there are buttons for "RUN", "Apply", and "Exit".

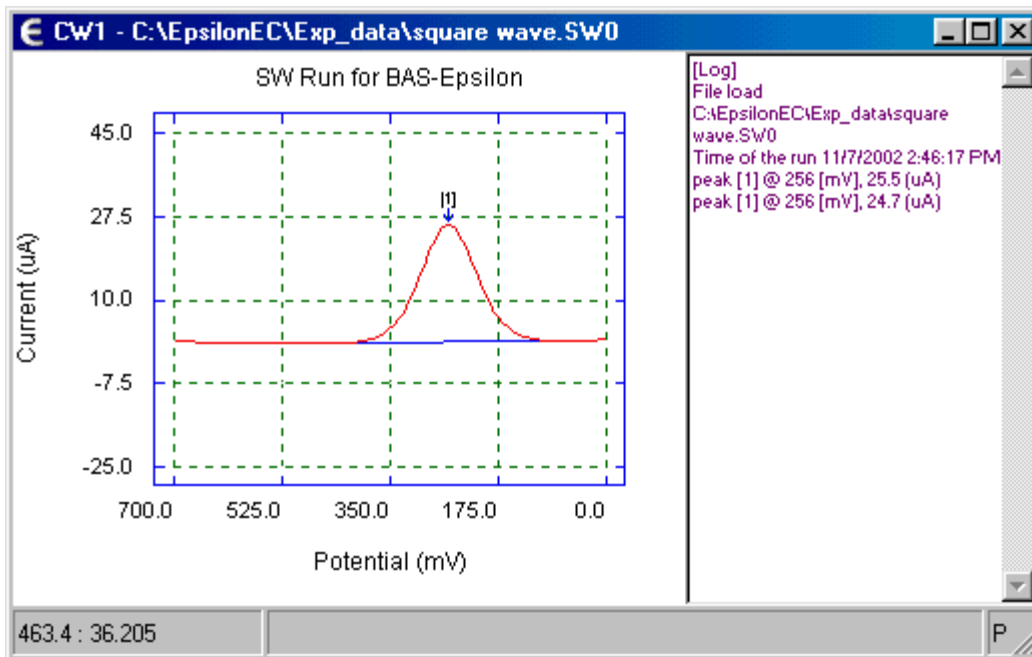
PROCEDURE (CONTD.)

FIGURE 18. CHRONOPOTENTIOMETRY GRAPH



18. Click New and select Square Wave Voltammetry (note that this requires the optional Basic Plus software). A fifth experiment window will be opened, together with the associated Change Parameters dialog box. Enter the parameters shown in Figure 19, then run the experiment. Typical data for a Square Wave Voltammetry experiment is shown in Figure 20.

FIGURE 19. SQUARE WAVE VOLTAMMETRY PARAMETERS

PROCEDURE (CONTD.)**FIGURE 20. SQUARE WAVE VOLTAMMETRY GRAPH**

19. The default plot for a Square Wave Voltammetry experiment is the difference current; that is, the current on the forward cycle less the current on the reverse cycle. The forward and reverse currents are also available under Select Graph in the pop-up menu in the Square Wave Voltammetry experiment window. The forward current data set is shown in Figure 21.

FIGURE 21. SQUARE WAVE VOLTAMMETRY GRAPH – FORWARD CURRENT