Epsilon EClipse™

Potentiostat / Galvanostat / Bipotentiostat

Chemical Test Procedure

August 2022

BASi

Copyright BASi 1997 – 2022
Bioanalytical Systems, Inc.
www.basinc.com

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.

1. INTRODUCTION

The Epsilon EClipse™ is the latest potentiostat designed by Bioanalytical Systems Inc. to enable scientists to conduct cutting edge electrochemistry experiments in the laboratory. It has upgraded hardware, updated software, an expanded applied potential range, and improved design compared to the Epsilon. This manual is intended to aid the electrochemist in utilizing the Epsilon EClipse™ to its utmost potential.

Additional accessories for electrochemistry experiments using the Epsilon EClipse™ are available from BASi. These include the C3 cell stand, the controlled growth mercury electrode (CGME), a rotating disk electrode (RDE2), and many different electrodes. These are mentioned where appropriate throughout the manual.

1.1 TECHNIQUES

Basic Software:

Potentiostat
- Cyclic Voltammetry (CV)
- Linear Sweep Voltammetry (LSV)
- Chronoamperometry/Chronocoulometry (CA/CC)
- Controlled Potential Electrolysis (CPE)
- DC Potential Amperometry (DCPA)

Galvanostat
- Chronopotentiometry (CP)
- Double Step Chronopotentiometry (DSCP)

Other
- Open Circuit Potential Vs Time (OP)

Basic Plus Software:

Pulsed Voltammetry
- Square Wave Voltammetry (SW)
- Normal Pulse Voltammetry/Polarography (NP)
- Differential Pulse Voltammetry/Polarography (DP)
- Sampled Current Polarography (SCP)

Stripping Voltammetry
- Linear Sweep Stripping Voltammetry (LSSV)
- Square Wave Stripping Voltammetry (SWSV)
- Differential Pulse Stripping Voltammetry (DPSV)

Methods Software:

Sequential Techniques

Bipotentiostat Techniques :

Multi-Channel Amperometry (MCA)
Multi-Channel Cyclic Voltammetry (MCCV)
Multi-Channel Chronoamperometry (MCCA)

1.2 FEATURES

Nineteen techniques
±10 V applied potential range
±12V compliance voltage range
Update user friendly software
100 µv potentiostat resolution
Hardware interfaces with BASi Voltammetric Cell Stand, Controlled Growth Mercury Electrode Cell Stand, and Rotating Disk Electrode Cell Stand
Control of magnetic stirring and gas purging with BASi cell stands
Automatic and manual peak fitting
Anson and Cottrell Plots
Digital smoothing
Semi-integration, and semi-differentiation

1.3 SPECIFICATIONS:

Potentiostat:
Channel 1 applied potential: Dynamic DAC: 16 bit, ±3.275 V at 0.1 mV resolution or ±10V at 0.33 mV resolution
Offset DAC: 8 bit, ±2.55 V at 10 mV resolution
Channel 2 applied potential: Dynamic DAC: 8 bit, ±2.55 V at 10 mV resolution
Compliance voltage: ±12 V
Maximum current: 100 mA
Bandwidth: >1012 W
(values for parameters other than applied potential are for both channels)

Current to voltage converter:
Full scale sensitivity: 1 nA*, 10 nA*, 100 nA, 1 µA, 10 µA, 100 µA, 1 mA, 10 mA, 100 mA
(*secondary gain used)
Secondary gain: x1, x10, and x100
ADC resolution: 16 bit
Sampling rate: 50 kHz (20 µs/conversion)
Data length: ≤ 64,000 points – fast

Galvanostat:
Applied current: 50 pA – 50 mA
Voltage range: ±10 V or ±1 V
Measured voltage resolution: 0.02 mV
Maximum leakage current: 30 pA

Minimum PC requirements:
Windows 7 or higher
USB port

Power requirements:
120 or 240 V AC, 50/60 Hz, 60 VA

Dimensions & Weight:
15.75" (40 cm) x 5.25" (13 cm) x 12.75" (32.5 cm)
17.5 lbs (7.4 kg)

1.4 WARRANTY

BASi® warrants equipment manufactured by the company to be free of defects in material and workmanship for a period of one year from the date of shipment, except as provided hereinafter. This assumes normal usage under commonly accepted operating parameters and excludes consumable products. BASi® further warrants that it has clear title to the goods and the goods shall be delivered free of liens and encumbrances. BASi® does not warrant either a good effect or against any ill effect following use of the goods.

All products manufactured by BASi® are tested and inspected prior to shipment. However, in the event a product is delivered to buyer with a defect, upon prompt notification by the buyer, BASi® will correct any defect in products or equipment of its manufacture either, at its option, by return of the item to the factory, or shipment of a repaired or replacement part. BASi® will not be obliged, however, to replace or repair any piece of equipment which it determines has been abused, improperly installed, altered, damaged, or repaired by others. Defects in

equipment do not include decomposition, wear, or damage by chemical action or corrosion, or damage incurred during shipment.

BASi® agrees either to repair or replace, at its sole option and free of part charges to the buyer, any parts of such instrumentation which, under proper and normal conditions of use, prove to be defective within 90 days from the date of shipment.

BASi® neither assumes nor authorizes any person to assume for it any other liability in connection with the sale, installation, service or use of its instrumentation. BASi® shall have no liability whatsoever for special, consequential, or punitive damages of any kind from any cause arising out of the sale, installation, service or use of the goods or services.

THE WARRANTY AND REMEDY PROVIDED HEREIN ARE SOLE AND EXCLUSIVE WARRANTY AND REMEDY MADE BY BASi® AND IN LIEU OF ALL OTHER WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING THE WARRANTY OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE.

Limited Obligations Covered by this Warranty

1. In the case of instruments not of BASi® manufacture, the original manufacturer's warranty applies.
2. Shipping charges under warranty are covered only in one direction. The buyer is responsible for shipping charges to the factory if return of the part is required.
3. This warranty does not cover damage to valves, lamps, seals, or columns due to improper installation by the buyer.
4. Warranty for thin-layer amperometric cells and working electrodes are limited to 60 days.
5. Warranty for valves is limited to 30 days.
6. Expendable items, including but not limited to microdialysis probes, catheters, tubing sets, reference electrodes, chemical standards, prepared solutions, lights, fuses, O-rings, gaskets, glass items, membranes and filters, are excluded from warranty.
7. Failure by the buyer to perform normal and reasonable maintenance on instruments will void warranty claims.
8. If the original invoice for the instrument is issued to a buyer which is not the end user, and not an authorized BASi® distributor, then all requests for warranty must be processed through the company which sold the product to the end user, and not through BASi® or its distributors.

1.5 SERVICE INFORMATION

BASi has skilled staff available to assist with technical support for your BASi equipment. For service, call (765) 463-4527 and ask for Customer Service. Following discussion of your specific difficulties, an appropriate course of action will be described and the problem will be resolved accordingly.

Do not return any products for service until a Return Authorization Number (RA#) has been obtained. The RA# identifies you as the sender and describes the problem you are having in full detail. All correspondence and shipments should be sent to:

Service Department
Bioanalytical Systems, Inc.
2701 Kent Avenue
West Lafayette, IN 47906
RA# _____'

2. SAFETY PRECAUTIONS

The following general safety precautions must be observed during all phases of operation, service, and repair of this instrument. Failure to comply with these precautions or with specific **WARNINGS, CAUTIONS,** or **NOTES** elsewhere in this manual may impair the protection provided by the equipment. Such noncompliance would also violate safety standards of design, manufacture, and intended use of the instrument.

Bioanalytical Systems, Inc. assumes no liability for the customer's failure to comply with these requirements.

- For indoor use only.
- Ground the Instrument. To avoid electric shock, the instrument must be grounded with the supplied power cable's grounding prong.
- DO NOT exceed the operating input power, voltage, current level and signal type appropriate for the instrument. Refer to the Installation Section for further information.
- Electrostatic discharge (ESD) can damage the highly sensitive microcircuits in your instrument. ESD damage is most likely to occur as the instruments are being connected or disconnected. Ground yourself to discharge any static charge built-up by touching the outer shell of any grounded instrument chassis before the I/O connectors are connected or disconnected.
- DO NOT place the instrument in fluid or expose the internal elements or back panel to fluid.
- DO NOT Operate in an Explosive Atmosphere. Do not operate the instrument in the presence of inflammable gasses or fumes. Operation of any electrical instrument in such an environment clearly constitutes a safety hazard.
- Keep Away from Live Circuits. Operators must not remove instrument covers. Component replacement and internal adjustments must be made by qualified maintenance personnel. Do not replace components with the power cable connected. Under certain conditions, dangerous voltage levels may exist even with the power cable removed. To avoid injuries, always disconnect the power and discharge circuits before touching them.
- DO NOT Substitute Parts or Modify the Instrument. To avoid the danger of introducing additional hazards, do not install substitute parts or perform unauthorized modifications to the instrument. Return the instrument to Bioanalytical Systems, Inc. Service Department for service and repair to ensure that safety features are maintained in operational condition.

If you notice any unusual conditions as listed below, immediately terminate operation and disconnect the power cable. Contact the Bioanalytical Systems, Inc. Service Department for repair of the instrument. If you continue to operate without repairing the instrument, there is a potential for hazard or damage to both the equipment and the operator.

- Instrument operates abnormally
- Instrument emits abnormal noise, smell, smoke or a spark-like light during operation
- Instrument generates high temperatures or electrical shock during operation
- Power cable, plug or receptacle on instrument is damaged
- Foreign substance or liquid has penetrated the outer cover of the instrument

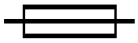
Throughout the course of this manual, the following words and symbols will be used to designate important information:

WARNING – This signifies extreme hazard. Not following the instructions may result in serious injury or death.
CAUTION – Following information relates to a hazard. If instructions are not followed properly, it can result in irrevocable damage to the instrument.

NOTE – This implies that the following instructions are essential for the user to understand in order to operate the

SYMBOLS


Caution: Risk of danger. User's manual must be consulted in all cases where this symbol is marked.


Fuse


Alternating current

On (supply)

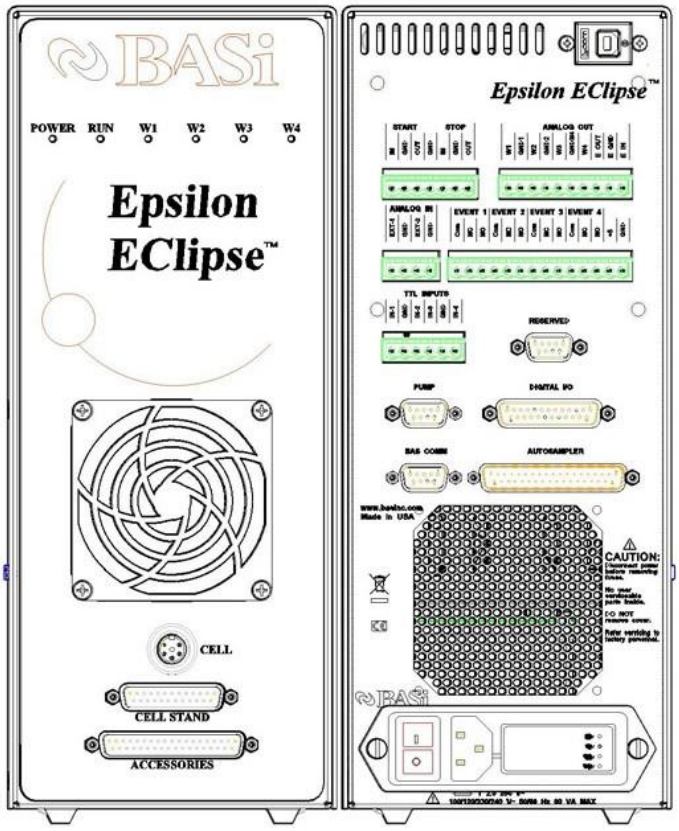

Off (supply)


Complies with European Union directives


The European Waste Electrical and Electronic Equipment (WEEE) Directive

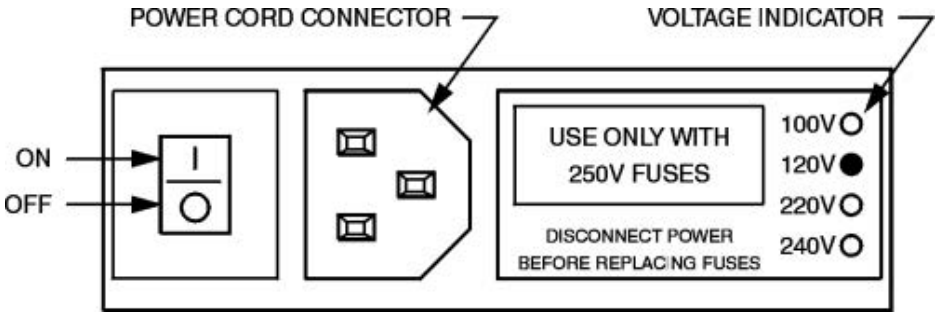
3. INSTALLATION

Connections to the cell and the cell stands (C3, RDE-2, CGME) are made on the front panel of the Epsilon EClipse™ system. All other connections, including the power and USB port, are made on the rear panel.



3.1. POWER

The Epsilon EClipse™ system requires a grounded power supply, providing either 120VAC at 60Hz or 240VAC at 50Hz. Before connecting the supplied power cord, check that the indicator next to the power connection shows the correct voltage.



If you need to change the power input for any reason, please contact BASi for assistance.

3.2. COMPUTER

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 on the back of the Epsilon EClipse™. The Epsilon EClipse software is included on a USB stick with each purchase. For the most up-to-date version of the software, please email ec@basinc.com.

3.3. CELL CONNECTION

The cell connection on the Epsilon EClipse™ can be found on the lower front panel of the instrument. The cell lead cable is the group of wires that connects the Epsilon EClipse™ to the electrodes of the electrochemical cell. The Epsilon EClipse™ has been supplied with a single-channel cell lead cable or a dual-channel cell lead cable, depending on whether it is a standard potentiostat or a bipotentiostat.

WARNING: NEVER CONNECT OR ADJUST THE CELL LEADS DURING AN EXPERIMENT OR WHEN THE CELL IS ON. DOING SO COULD DAMAGE THE SENSITIVE AMPLIFIERS AND VOID YOUR WARRANTY.

The general purpose cell lead cable is terminated with alligator clips that attach directly to the cell electrodes.

SINGLE-CHANNEL CELL LEAD (ER-9861)

There are 3 electrode leads and 1 grounded (shielding) lead.

Black: Working Electrode
Red: Auxiliary electrode
White: Reference electrode
Ring: Ground connector

BIPOT CELL LEAD (ER-9860)

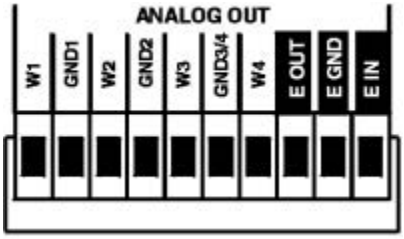
There are 4 electrode leads and 1 grounded (shielding) lead.

Black W1: Working Electrode W1
Black W2: Working Electrode W2
Red: Auxiliary electrode
White: Reference electrode
Ring: Ground connector

CELL STAND CABLE (ER-9862)

This cable is available for direct attachment to the LEMO port on a BASi cell stand.

3.4. ANALOG INPUT/OUTPUT



Analog inputs and outputs can only be used for potentiostatic techniques. An analog output is provided for the W1 and W2 channels (W3 and W4 are unavailable on the Epsilon EClipse™), and must be activated from within the software (see Manual Control in section 7.2). These outputs have a full scale output of ± 10 V and are provided for connection to chart recorders and other data-acquisition devices. The W1 or W2 terminal should be connected to the "high" or "+" input of the peripheral device, and the GND terminal to the "low" or "-" input (do not use any additional grounding that may be available on the peripheral device).

The **E OUT** output is provided to monitor the potential applied to the cell on W1 (or the potential of the working electrode in the Open Circuit Potential technique), and the **E IN** input is provided to apply a potential to W1 from an external source (this external potential is summed to the potential applied by the Epsilon EClipse™). Please be aware that noise may be introduced into the system when **E IN** is activated.

3.5. REMOTE START/STOP

The remote start and stop connections provide several alternatives for sending and receiving signals to and from other instruments. These functions are fixed in time and cannot be modified. For programmable triggers to remote instruments, see Timed Events below.

START IN

Allows an external device to trigger the start of an experiment. Note that this is not the start of data acquisitions, and several hundred milliseconds plus the Quiet Time may elapse from the trigger until data acquisition starts. A switch closure or TTL-low of at least 55ms across the START IN terminal and its ground will trigger the run.

START OUT

Used to trigger other instruments at the start of an experiment. It provides a 1 second TTL-low.

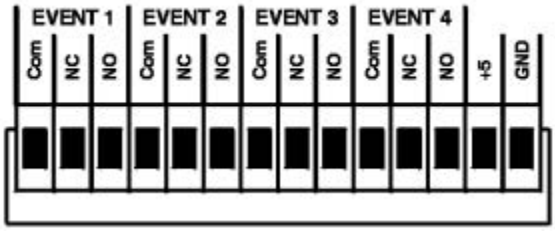
STOP IN

Not applicable for the Epsilon EClipse™.

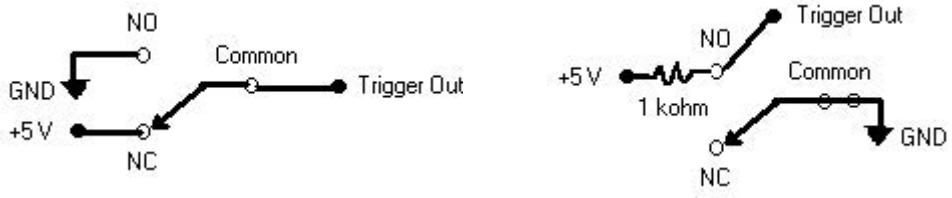
STOP OUT

Used to trigger other instruments at the end of an experiment. It provides a 1 second TTL-low at the end of the run. The time between the last data point acquired and this signal depends upon the technique and its parameters.

3.6. TIMED EVENTS



Timed Events are programmable switch closures that provide exceptional flexibility for controlling peripheral instruments. Four switches are provided, which can be connected in a normally-open (NO) or a normally-closed (NC) configuration. Two possible configurations to create TTL signals are shown below.



With both configurations the trigger line will normally be at 5V and will step to 0V when activated. In the figure on the right, the resistor (1 - 10kΩ) is required to limit the current drawn from the 5V power supply. These switches may be manually activated in the software, or programmed as part of Sequential Techniques (see section 7.4 and section 7.6).

3.7. STARTING THE EPSILON ECLIPSE™

Once the Epsilon EClipse™ is plugged into the USB port on your computer and powered on, you can open the Epsilon EClipse™ software. The software will automatically connect to the instrument. You should see the message "Epsilon Connected" in the bottom left corner of the software. If you receive the following message when you open the software, please check the power and USB connections.

If the connection is broken after it has been established, the PC and the Epsilon EClipse™ can be reconnected using **Reconnect Epsilon** in the **Instrument** menu.

4. EPSILON ECLIPSE™ CHEMICAL TEST PROCEDURE

PURPOSE

The purpose of this test is to perform a final examination of the Epsilon EClipse™ before going to the customer, and to provide the customer with typical output and data. From these outputs, the customer can verify that the instrument is working properly on arrival and can gain some experience in its operation.

INSTRUMENT INSTALLATION

Please follow the installation instructions at the beginning of this manual. If available, a BASi cell stand should be connected to the **CELL STAND** port on the front of the Epsilon EClipse™.

TEST SOLUTION

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

PREPARATION OF THE 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-5B 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 µm alumina following the polishing instructions provided in the polishing kit.

If the RE-5B 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-5B electrodes must be stored in 3M sodium chloride when not in use.

PROCEDURE

1. Turn the Power switch on the Epsilon EClipse™ to on.

2. Open the Epsilon EClipse™ software by clicking the **EpsilonEC** icon. The software will automatically connect to the instrument. You should see the message "Epsilon Connected" in the bottom left corner of the software. If you receive a message that the software is unable to connect to the instrument, please check the power and USB connections. If the link is lost after being established, use **Reconnect Epsilon** in the **Instrument** menu to reestablish the link.

3. Click **New** in the **File** menu to set up a new experiment. The list of available techniques is displayed (**Fig 1**). 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 particular Epsilon EClipse™. 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-Plus and Methods Epsilon EClipse™ software.

Figure 1 - Selecting a New Technique

4. Select **Cyclic Voltammetry**. The **new .etech file** can now be edited. 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**). Various experimental data can be entered into the **Experimental Conditions** section. These notes will also be saved when the experimental data is saved. Once these changes have been entered, an experiment using these parameters can be run by clicking the **RUN** button. An experiment can be run using either **Run** in the **Experiment** menu, or the **RUN** icon on the Tool Bar. This icon will change to **STOP** during the experiment, and can be used to abort the experiment.

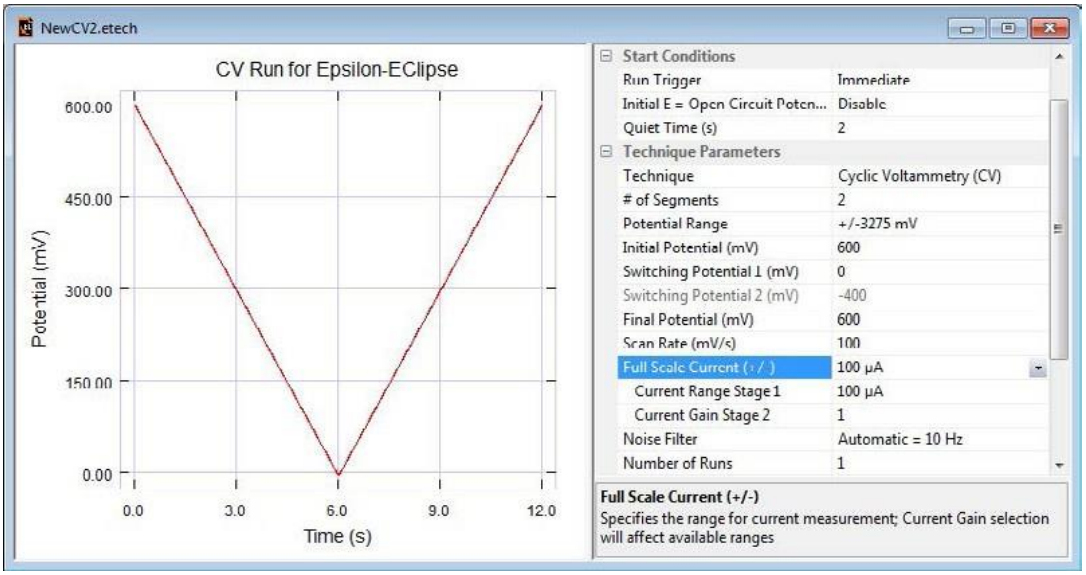


Figure 2 - Editing the .etech for Cyclic Voltammetry

5. After the experiment has been run, the voltammogram will be displayed (**Figure 3**). Note the information about the experiment and the peak parameters on the right side of the graph.

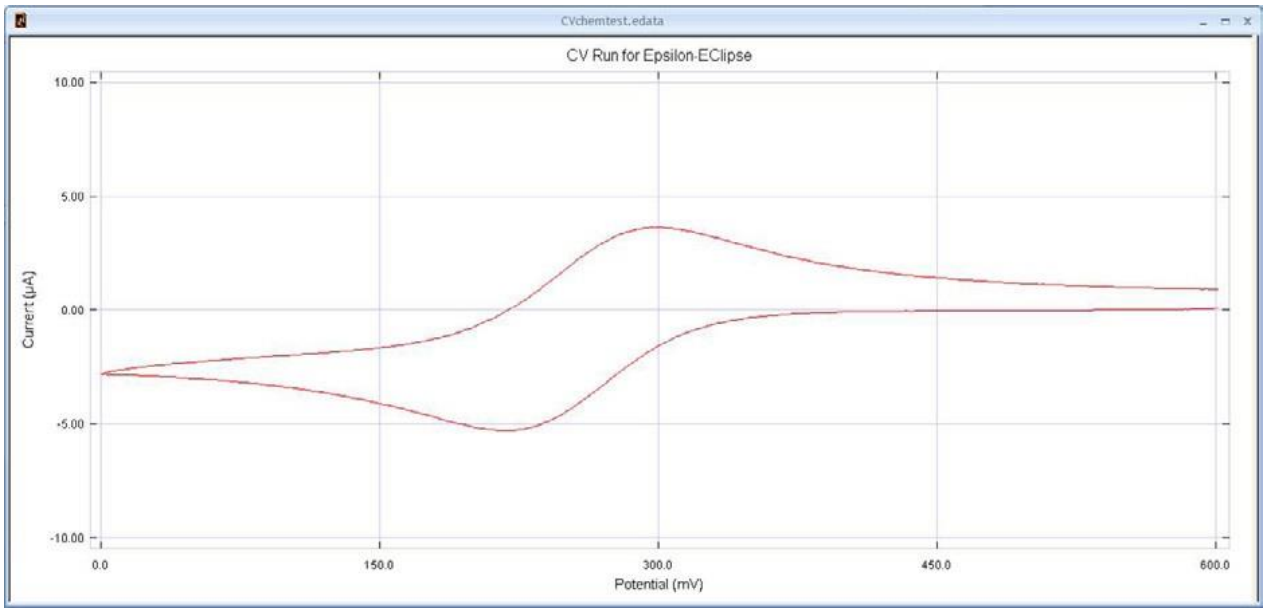


Figure 3 - Cyclic Voltammetry graph

6. A specific area of the graphic can be enlarged by using the mouse cursor (and the left mouse button) to define the area (**Figure 4**), by using the mouse scroll to zoom in and out, or by entering the x and y values in the **Data Display Settings** dialog box in the Graph menu (**Figure 5**). The original graph can be restored using **Zoom Full Range** in the **Graph** menu.

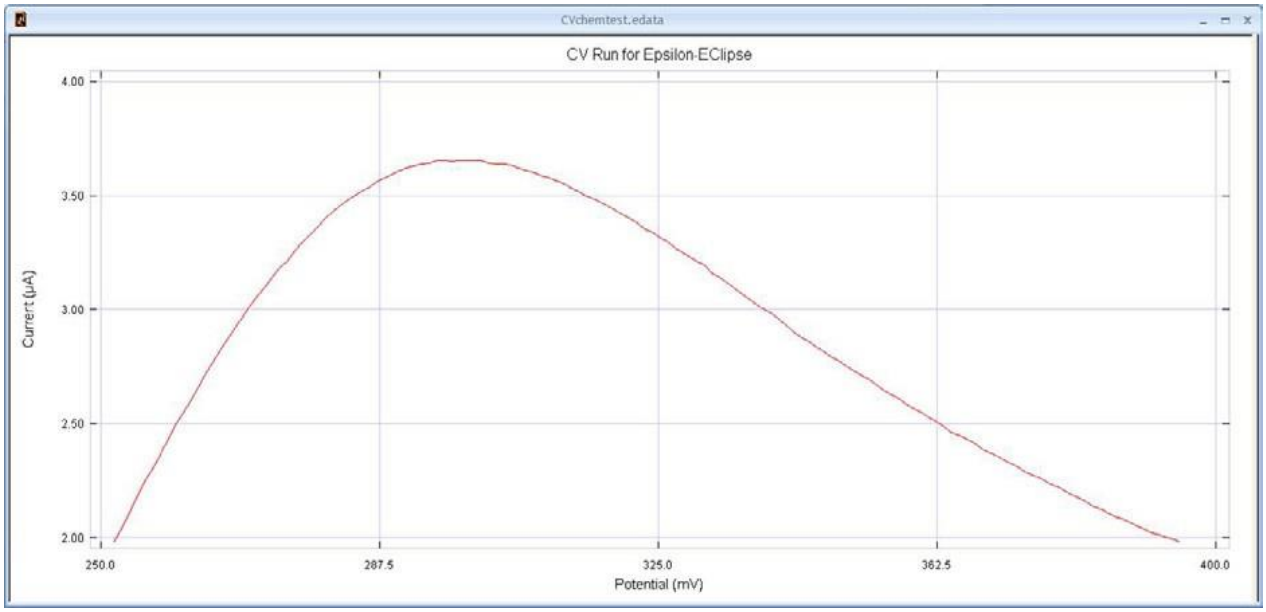


Figure 4 - Enlarged section of the Cyclic Voltammetry graph

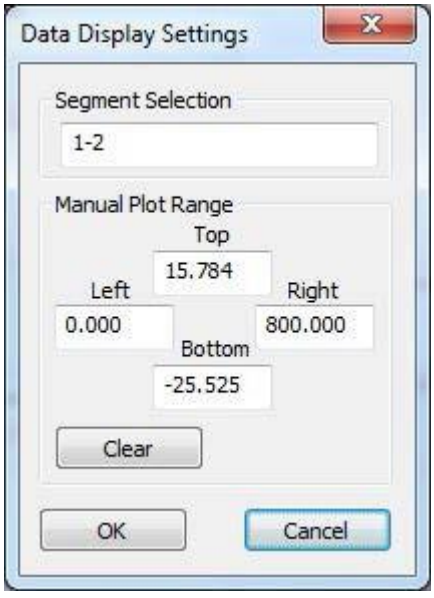


Figure 5 - Data display settings dialog box

7. Use **Save** in the **File** menu to save the data in the active .edata window. The data can be converted to a number of different text formats using **Save Data As** in the **File** menu (**Figure 6**). Select the desired options and the delimiter, and then click **OK** to save the converted Data.

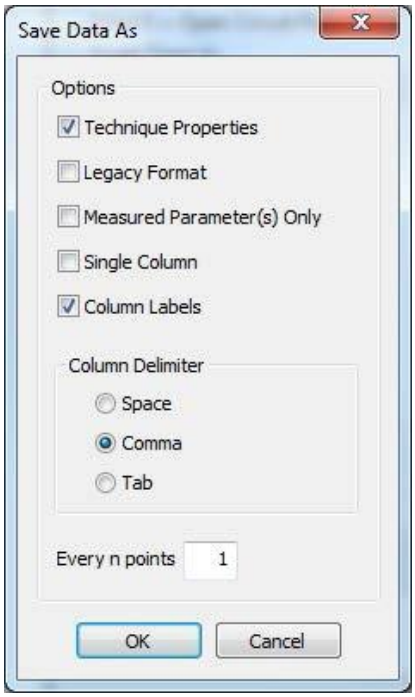


Figure 6 - File conversion

8. Click on the open .etech window. To the right of the graph, find the scan rate parameter box and change the **Scan Rate** to 200 mV/s. Run the experiment again. Note that the new data is displayed in a new .edata window. Save this data. Change the scan rate to 500 mV/s, run the experiment, and save the data.

9. 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** menu. All open *.edata files will be added to the overlay (**Figure 7**).

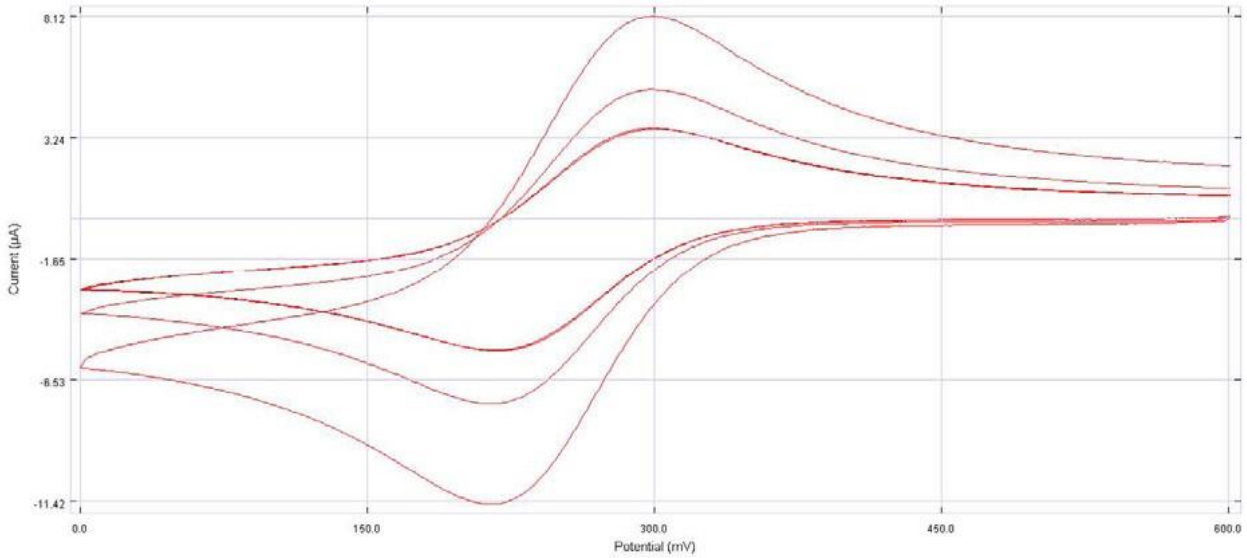


Figure 7 - Overlayed cyclic voltammograms

10. Click **New** in the **File** menu, and select **Cyclic Voltammetry** again. Enter the parameters shown in **Figure 8**, and run this experiment. Since there are 3 segments, 4 potential parameters must be defined (**Initial Potential-Switching Potential 1-Switching Potential 2-Final Potential**).

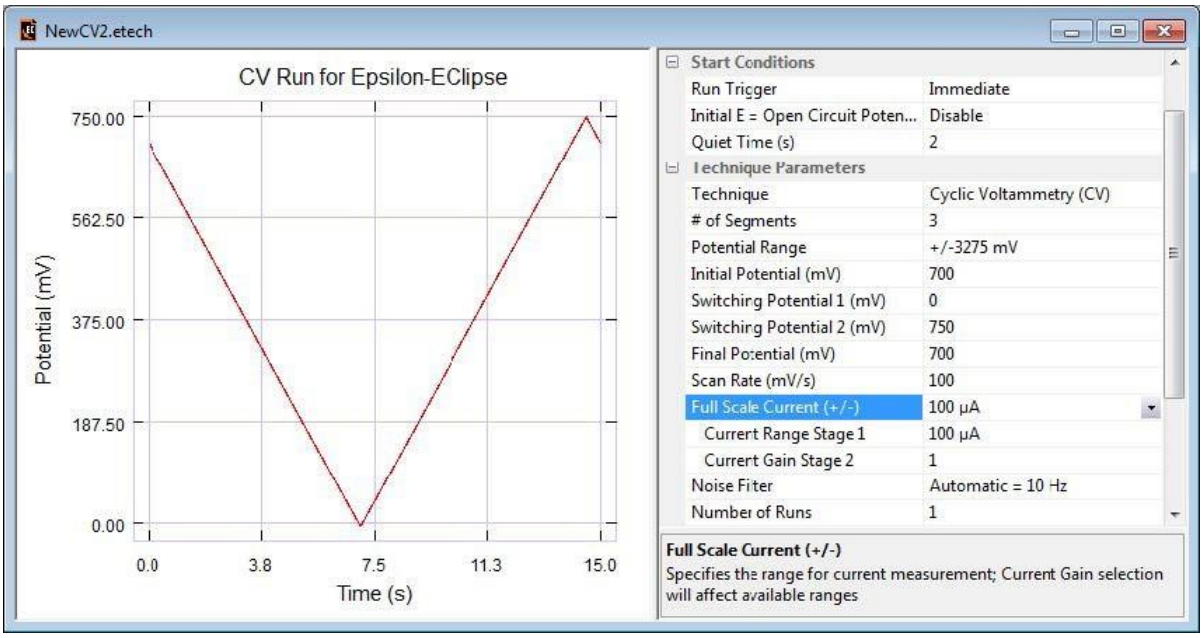


Figure 8 - New Cyclic Voltammetry Parameters

11. Click **New**, and select **Chronoamperometry/Chronocoulometry** from the list of techniques. A third window will appear. Enter the parameters shown in **Figure 9**. Running the experiment generates the plot shown in **Figure 10**.

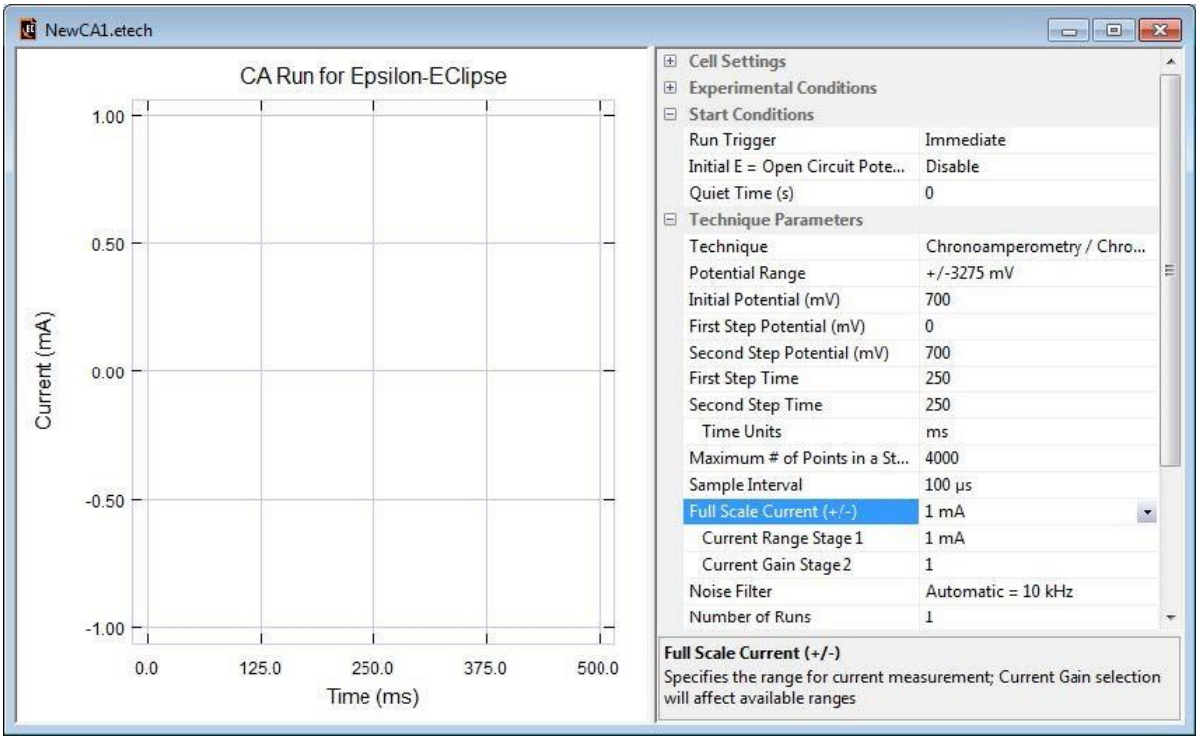


Figure 9 - Parameters for Chronoamperometry/Chronocoulometry

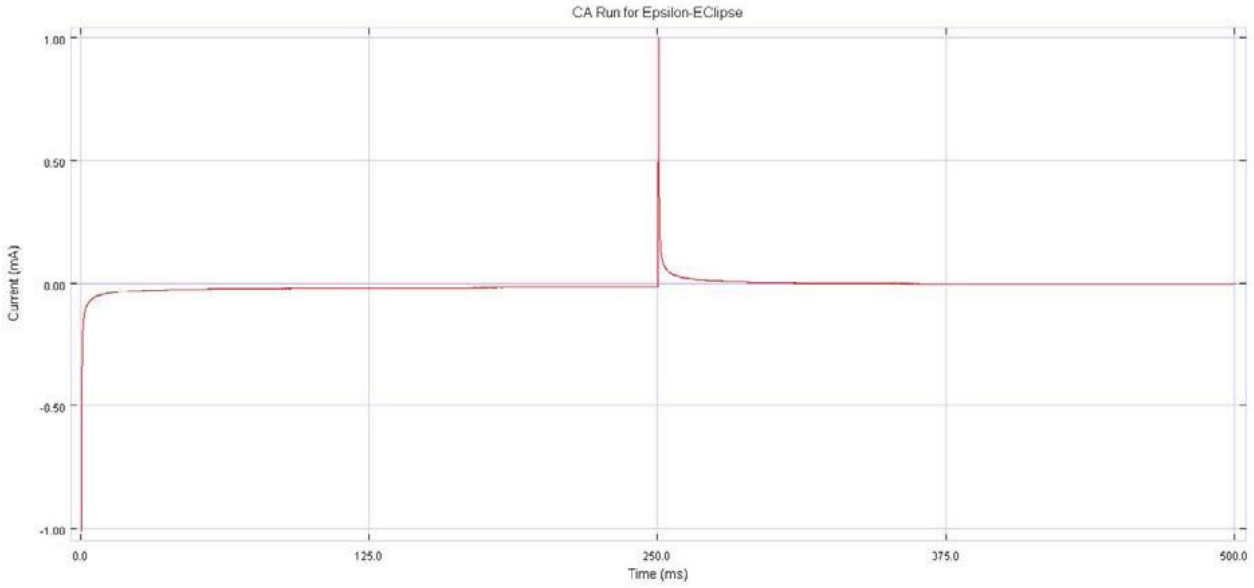


Figure 10 - Chronoamperometry/Chronocoulometry graph

12. The data from a **Chronoamperometry/Chronocoulometry** experiment can be plotted in a number of different formats, which can be selected using **Select Graph Type** in the graph menu, or by right-clicking on the graph. The **Q vs. sqrt(T)** plot is shown in **Figure 11**.

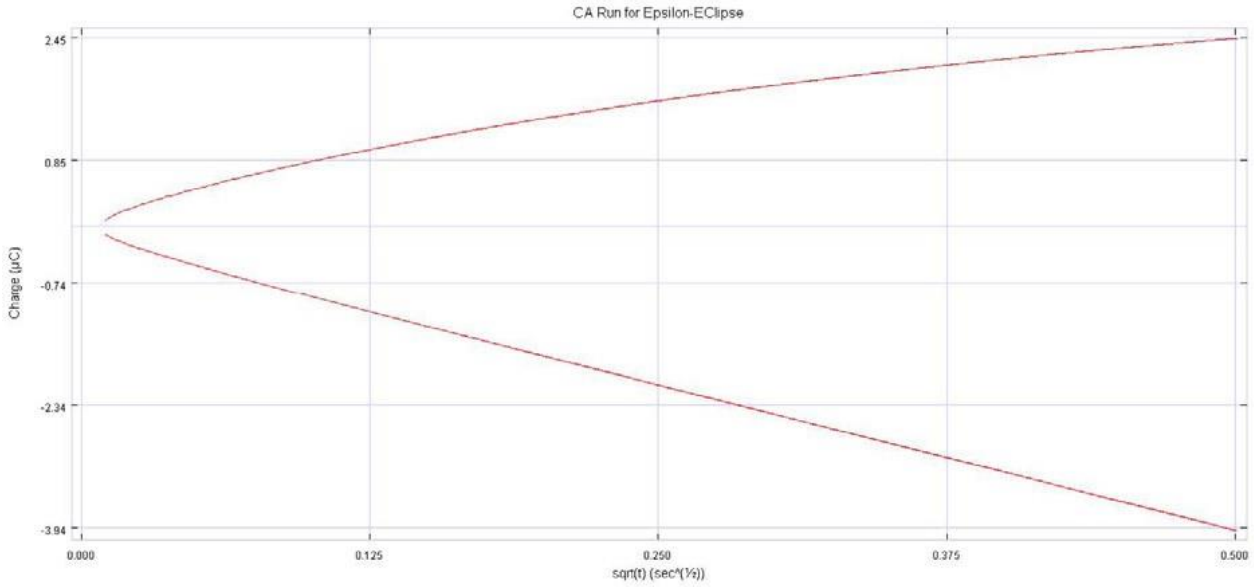


Figure 11 - Q vs. sqrt(T) plot for Chronoamperometry/Chronocoulometry

13. The Epsilon EClipse™ software can calculate the slope and intercept of the linear **Q vs sqrt(T)** plot when it is displayed. Selecting **Calculate CA-SIR** from the Analysis menu generates the information box shown in **Figure 12**. The lines used for the linear fitting are the dashed blue lines in **Figure 12** (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).

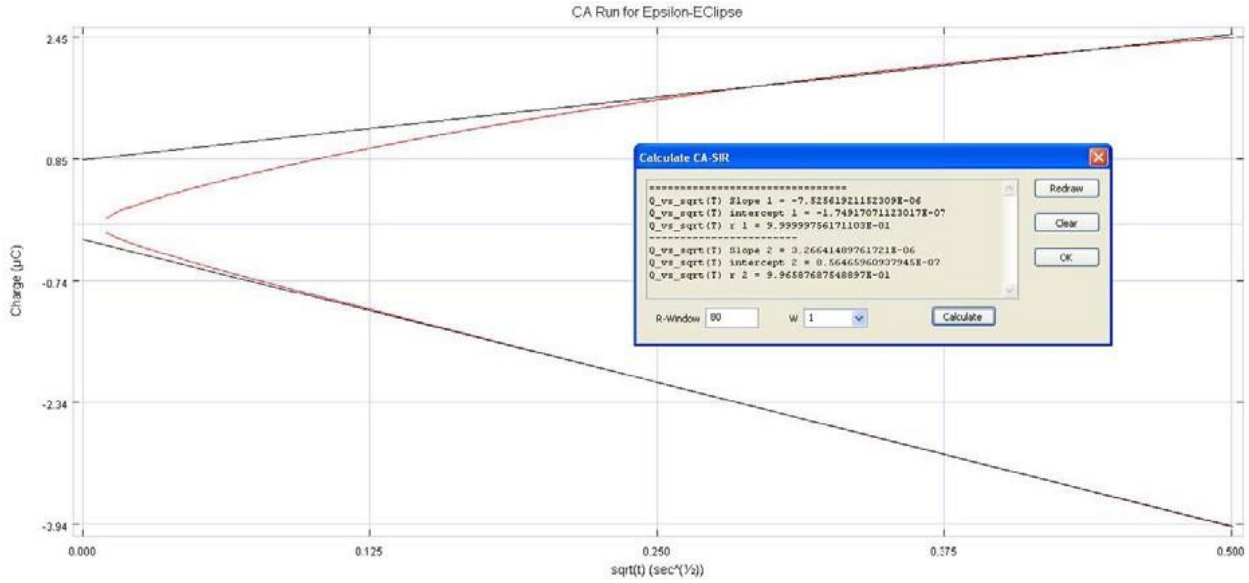


Figure 12 - Calculation of slope and intercept of linear plot

14. Click **New**, and select **Chronopotentiometry.** A fourth experiment window will be opened. Enter the parameters shown in **Figure 13**, 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 14**.

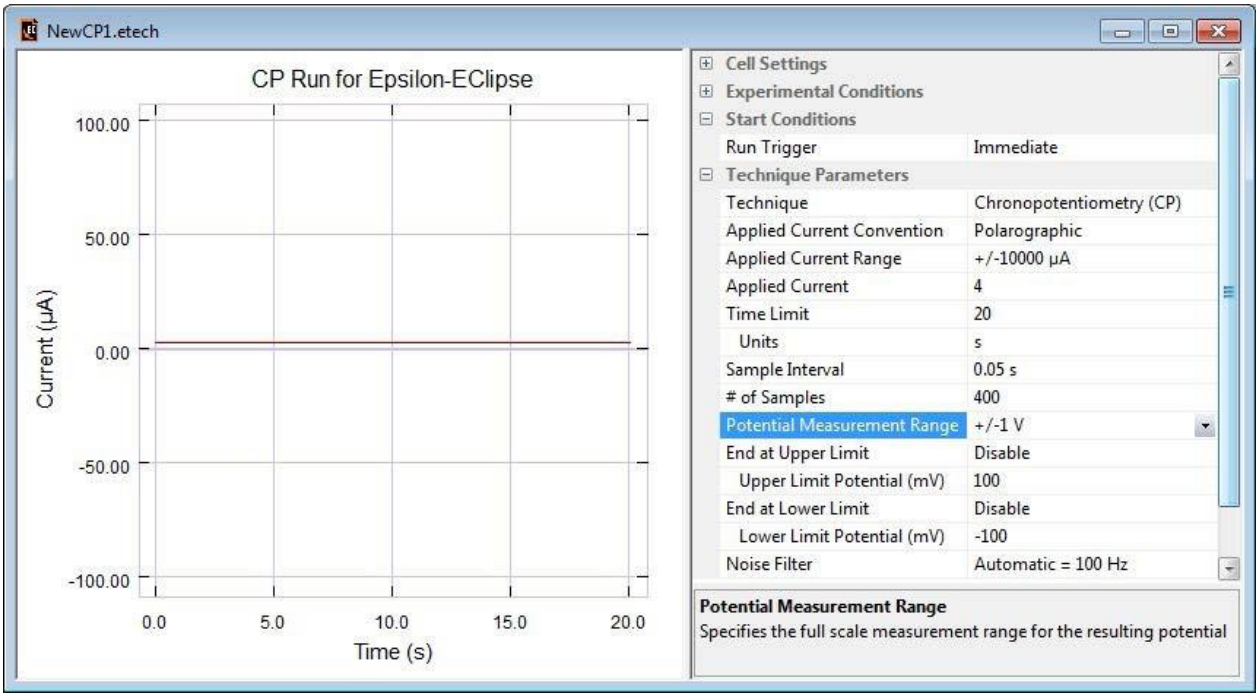


Figure 13 - Parameters for Chronopotentiometry

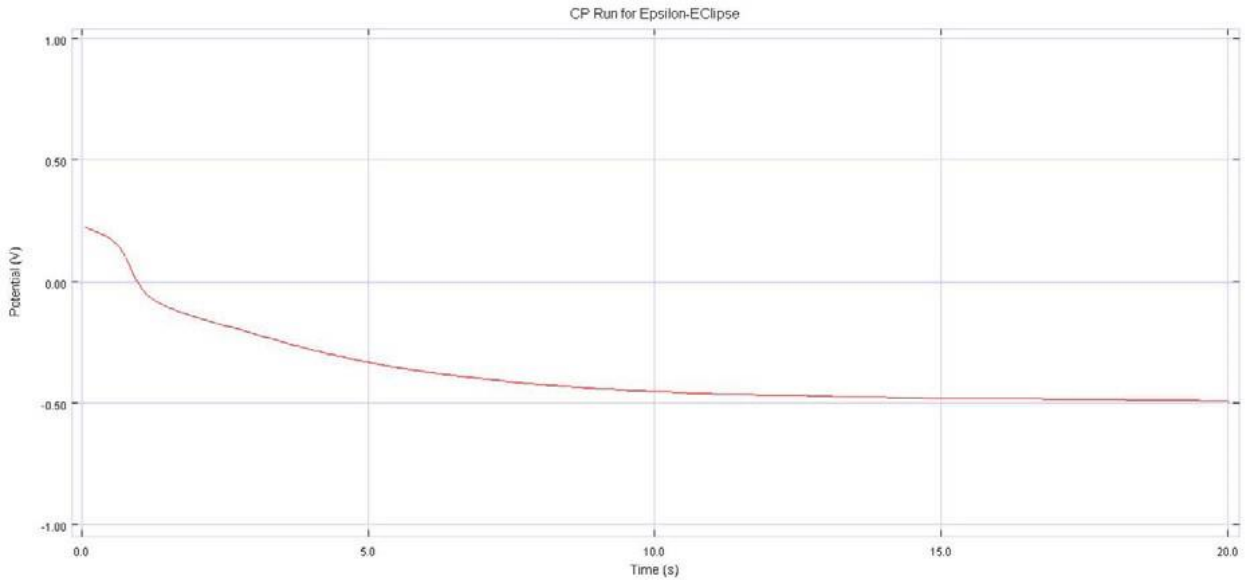


Figure 14 - Chronopotentiometry graph

15. Click **New**, and select **Square Wave Voltammetry** (note that this requires the optional Basic Plus software). A fifth experiment window will be opened. Enter the parameters shown in **Figure 15**, then run the experiment. Typical data for a **Square Wave** Voltammetry experiment is shown in **Figure 16**.

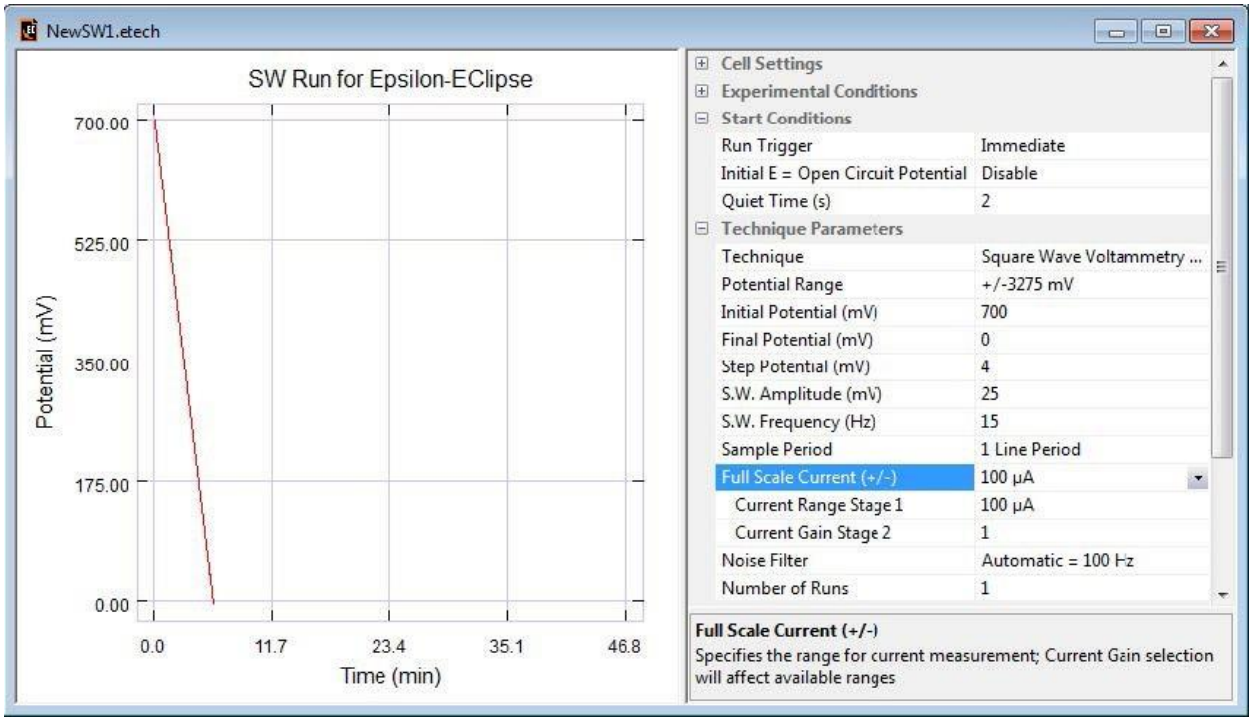


Figure 15 - Change Parameters dialog box for Square Wave Voltammetry

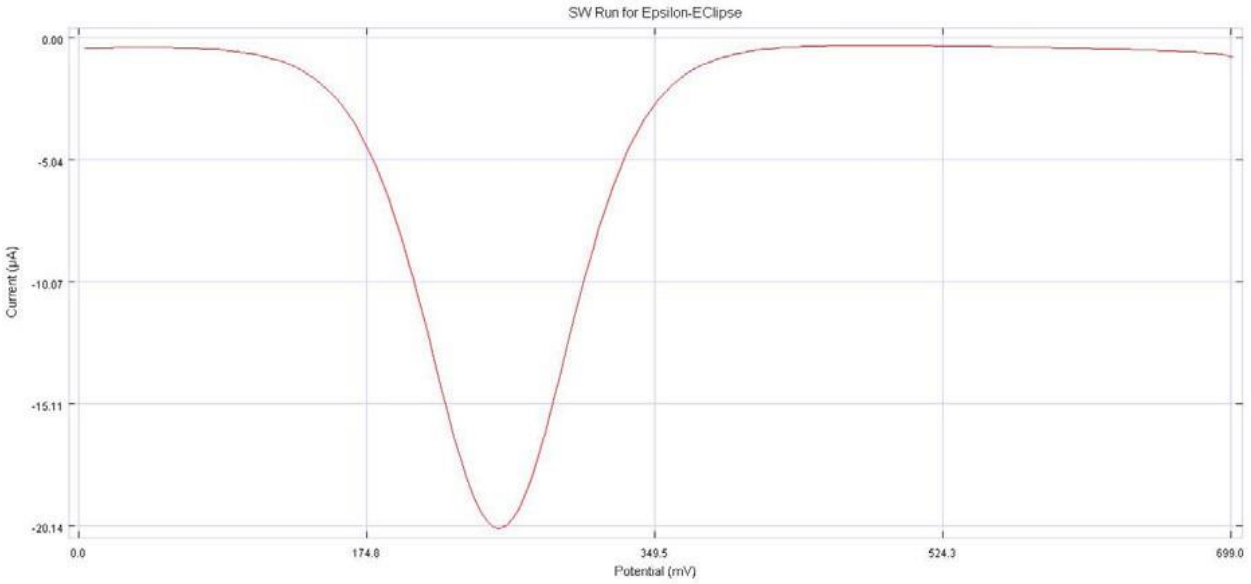


Figure 16 - Default plot for Square Wave Voltammetry

16. 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 by right-clicking on the graph, or by selecting **Select Graph Type** from the **Graph** menu. The forward current data set is shown in **Figure 17**.

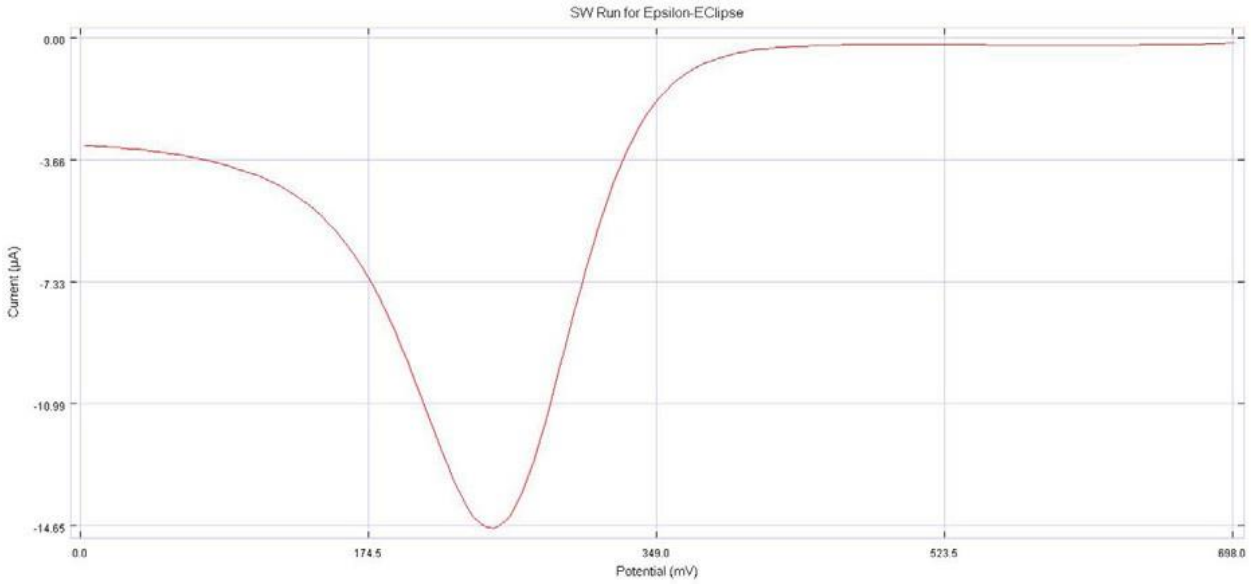


Figure 17 - Forward current plot for Square Wave Voltammetry

This completes the chemical test. All printed output should be shipped with the instrument. The power cord and cell lead (with alligator clips on the cell end) are shipped with the Epsilon EClipse™.