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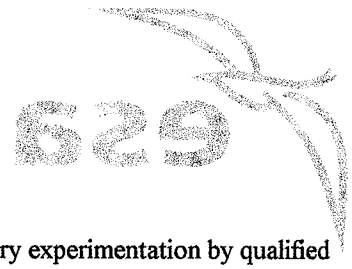
MICROPROCESSOR CONTROLLED

OMNI101 ANALOG POTENTIOSTAT



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Introduction

Potentiostats are used in electrochemical experiments to provide control of the working electrode potential in the cell. The cell usually consists of three electrodes (working, auxiliary and reference) in a container with a solution of electrolyte and the chemical species to be studied. The potentiostat also provides for the measurement of the current flowing through the cell to the working electrode.

The Omni 101 Potentiostat has especially high stability and reproducibility due to the careful selection of components to optimize its performance. It has been designed for high sensitivity measurements of low current levels so critical to the successful use of microelectrodes.

Specifications

Compliance Voltage:	± 15 V
Compliance Current:	± 20 mA
Applied Potential:	± 2 V
Rise time:	20 µs to 200 µs*
Current ranges:	100 nA/V to 100 mA/V
Techniques:	Cyclic Voltammetry
	Linear Sweep Voltammetry
	Chronoamperometry
	Linear Sweep Anodic Stripping Voltammetry
	Controlled Potential Electrolysis
Options:	XYT recorder output
	RS-232 output
	External input
Power:	AC, 115V, 60 Hz

Three-electrode potentiostat: two-electrode functions are accessible by shorting the reference electrode connector to the auxiliary electrode connector.

Seven current sensitivity ranges:

100 mA/V, 10 mA/V, 1 mA/V, 100 µA/V, 10 µA/V, 1 µA/V, 100 nA/V

(10, 100, 1K, 10K, 100K, 1M, and 10 M Ω feedback resistors)

---

* Depending on Current Range set.

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Recorder output accuracy:	± 0.15 % and ± 0.5 mV
Physical Dimensions:	25.7 cm W x 24.1 cm D x 13.3 cm H, 4.5 kg

Accessories

Electrodes, electrode kits, reference electrodes, auxiliary electrodes, polishing kits, Linseis Model 16000 XYT recorder, Faraday Shield Cases...

Call us for details on all our electrochemical accessories.

CAUTIONS

Removing the printed circuit board or meter or attempting repairs to the circuit board may void the warranty on the Omni 101 Potentiostat. If the instrument warranty is still in effect, contact the factory for assistance before attempting service work on your own.

Static electricity: Take proper measures to ensure that static electricity does not damage electronic components by touching a static pad or some grounded object before using the instrument.

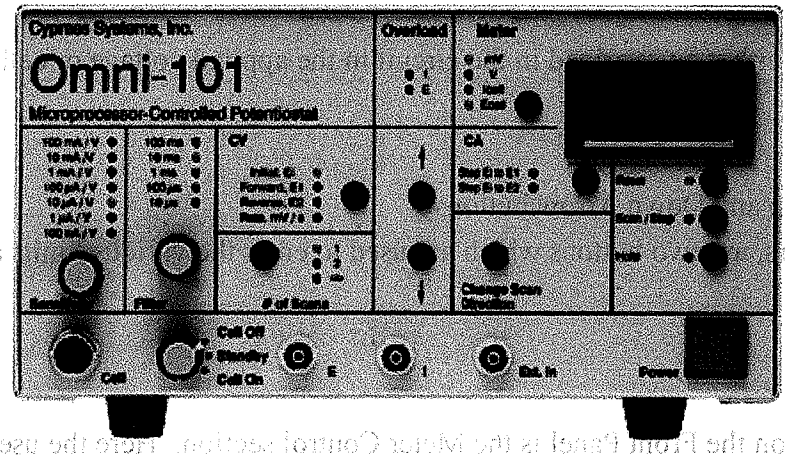
Current/Voltage Overload Signal: The Omni 101 Potentiostat will indicate if either the cell control amplifier or the current-measuring amplifier is driven past 9 V, even briefly as in switching from dummy to external cell or when changing current ranges. If the LED remains lit, switch to a less sensitive current range, check your cell connections and verify that the auxiliary electrode is functioning properly.

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Overview of the Omni 101 Potentiostat

A potentiostat is an instrument that controls the potential (emf) applied to the working electrode (WKG) of an electrochemical cell independently of the current level. For example, if you set the applied potential of a platinum working electrode to +0.10 V vs. a reference Ag/AgCl electrode, the potential will remain at +0.10 V no matter what current flows through the cell (within the power limits of the instrument).

Front Panel Description of Functions



Sensitivity

Located at the far left of the front panel is a column of 7 LEDs, which indicate the sensitivity selected for the experiment. The sensitivity ranges from 100 mA / V to 100 nA / V. The rotary switch controls the sensitivity.

Cell

Below the sensitivity LEDs is the socket for the electrode cable.

Filter

To the right of the Sensitivity LEDs are the Filter Setting LEDs. There are 5 selections to choose from; ranging from 100 ms to 10 µs. A rotary switch controls the selection of the sensitivity.

Cell On-Off Switch

Below the Filter Setting LEDs is the rotary switch to switch from internal dummy cell, **Cell Off**, to the open circuit position, **Standby**, and then to external cell, **Cell On**.

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CV (Cyclic Voltammetry) Section

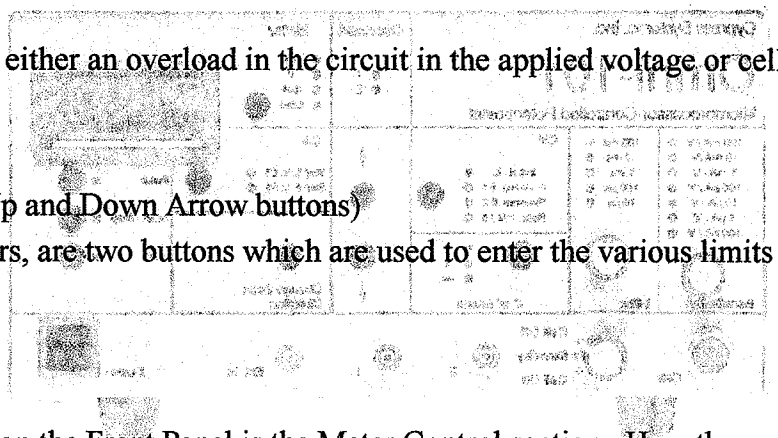
To the right of the filter section is the block, which is used to set up the parameters to do a Cyclic Voltammogram experiment. Below the parameter limit block is the number of scans selection block.

Output/ Input BNC

Below the CV section of the front panel are three BNC jacks, which allow connection of the Omni-101 to a choice of recording devices. Also the External Input BNC allows incorporation of an external signal to the Omni-101.

Overload LEDs

The Overload LEDs indicate either an overload in the circuit in the applied voltage or cell current portion of the circuit.



Parameter Adjust Buttons (Up and Down Arrow buttons)

Below the Overload indicators, are two buttons which are used to enter the various limits and scan rate for the experiment.

Meter

Next to the overload section on the Front Panel is the Meter Control section. Here the user has a selection to make as to what is displayed, Ecell or Icell, and in what units, mV or V, on the LCD meter.

CA (Chronoamperometry) Section

The Chronoamperometry Section sets up what potential step is performed in the experiment.

Scan Direction

When pressed, this button will reverse the scan direction during a CV experiment.

Experimental Control Section

The three switches here are used to control the experiment **Reset**, **Scan/ Step** or **Hold** at a potential.

Power Switch

This button turns on the AC power to the Omni-101.

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Cell Cable

Connector for cell cable. The working electrode has the Blue boot, the reference electrode has the White boot and the auxiliary electrode has the Red boot.

WKG = Blue
REF = White
AUX = Red

Cell On/Standby/Off

The rotary switch can be rotated from connection to an internal dummy cell to a position where the connection to the external cell allows the rest potential to be measured and displayed on the meter. The final position completes the connection to the external cell.

Power

Turns the power to the instrument On and Off.

E

BNC connector to X channel of an XY recorder. This is actually -E applied because of the display convention used.

I

BNC connector to Y channel of an XY recorder.

Ext. In

BNC connector for input of an External source, for example a waveform generator.

Back Panel Description

RS-232 Connector

9-pin socket for connection via RS-232 cable to collect data from the Omni-101 to a computer.

Power cord socket

Fused socket for connection of AC power cord.

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Ground Jack
Banana jack for grounding connection to Faraday Case, if applicable.

Electrochemical Cell

The electrochemical cell consists of a working electrode (WKG), auxiliary electrode (AUX) and reference electrode (REF).

Connectors

Connect each of the cell cables to the cell cable socket located on the front of the potentiostat using the cable provided. Check that the switch is switched to the **Cell Off** position.

BNC outputs are located at the front of the potentiostat for connecting to a X-Y recorder: the Y-axis is conventionally for the current and the X-axis is for the potential. Use only the Y-axis connectors with an Y-T strip-chart recorder.

Power On the Potentiostat

The Omni 101 Potentiostat is operated from AC power. The power switch is located in the lower right corner of the front panel of the Omni-101.

Setting Switches (Parameters) on the Omni 101 Potentiostat

Description of Button Functions

Initial, Ei

Press the button under the CV on the front panel until the LED next to the **Ei** is lit. The meter will display the default value for the **Ei**, 100 mV. The unit digit will be flashing. This can be changed using either the up or down arrow. To change to the next digit over, press and hold one of the arrow buttons and then press the other arrow button to move to the next digit to change. Any value between ± 2000 mV is acceptable for this parameter.

Forward, E1

Press the button under the CV on the front panel until the LED next to the **E1** is lit. The meter now will display the default value for the **E1**, 1000 mV. The units' digit will be flashing. This can be changed using either the up or down arrow. To change to the next digit over, press and hold one of the arrow buttons and then press the other arrow button to move to the next digit to change. Any value between ± 2000 mV is acceptable for this parameter.

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Reverse, E2

Press the button under the CV on the front panel until the LED next to the **E2** is lit. The meter now will display the default value for the **E2**, 0 mV. One digit will be flashing. This can be changed using either the up or down arrow. To change to the next digit over, press and hold one of the arrow buttons and then press the other arrow button to move to the next digit. Any value between ± 2000 mV is acceptable for this parameter.

Rate

Press the button under the CV on the front panel until the LED next to the **Rate** is lit. The meter now will display the default value for the **Rate**, 100 mV/sec. One digit will be flashing. This can be changed using either the up or down arrow. To change to the next digit over, press and hold one of the arrow buttons and then press the other arrow button to move to the next digit. Any value between ± 2000 mV is acceptable for this parameter.

# of Scans

Press the button next to the **# of Scans** until the desired number of scans is shown. There are three choices: one scan, two scans or infinity number of scans. The one and two scans will automatically stop the scanning at the end of the selected scans. When the infinity choice is selected, the scans will continue until either the **Hold** or **Reset** buttons are pressed.

Experimental Control Section

Reset

Pressing the **Reset** button will immediately cause the applied potential to go to the **Ei**. The **Reset** button is also used as an Enter key to enter a value into the experiment parameters. Therefore, anytime a parameter is changed the **Reset** button must be pressed to enter the new value(s) set.

Scan / Step

Pressing this button will start the experiment. The scan will begin at the **Ei** go to the **E1**, reverse direction and go to the **E2**, then if applicable go back to the **Ei**.

Hold

Pressing this button during the scan will stop the sweep and hold the Cell potential at the applied potential when the button was pressed. Pressing the **Scan/Step** button causes the scan to continue from the held potential.

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Change Scan Direction

During the course of a CV experiment you may reverse the scan direction at anytime during the scan by pressing this button. Pressing the button will not edit the limits setup initially, however. Subsequent scans will follow the parameters setup before starting the experiment.

Meter

I Cell	Meter displays the cell current during an experiment.
E Cell	Meter displays the cell potential during an experiment.
mV or V	Changes the units of the Meter.

Two of the four LEDs are lit at all times. Pressing the meter button scrolls through the display possibilities. These can be changed while an experiment in progress.

Sensitivity

Rotation of the knob to the right moves the selection to decreasing sensitivity.

Filter

Rotation of the knob to the right moves the filter selection to greater filtering time constants.

Overload

An LED is lit when either the Voltage or Current is out of range.

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Example Set-up Procedure for Cyclic Voltammetry (CV)

Assuming that we wish to do CV on a solution containing 0.001 M Ferrocyanide in 0.05 M KCl at a Pt or glassy carbon minielectrode (1 mm diameter), a reasonable set of parameters is:

scan range:	-0.100 V to +0.600 V and return to -0.100 V
scan rate:	50 mV/s
sensitivity:	10 μA/V and set Y-axis of recorder on 100 mV/in or
	100 mV/cm. Thus, recorder sensitivity = 1.0 μA/in or 1.0 μA/cm
filter:	20 ms
# scans:	1 (2 or continuous if you wish to see how successive CV scans look!).

Set these values using the procedures previously described in the Button Function Section, on page 5.

With the **Cell Off** and the Power On, set the **Meter** to display **Ecell**, V or mV and push the **Reset** button.

Running the Experiment with the Omni 101 Potentiostat

Go through the checklist below to see if everything is set properly:

* X-Y recorder connected, both X and Y-axis at 100 mV/in or 100 mV/cm;
  Also adjust both X and Y to zero points and mark this location
* Electrodes connected properly
* Power On
* **Cell Off**
* **Reset** LED on
* FILTER at 20 ms
* SENSITIVITY at 10 μA/V
* **Ei** = +0.000 V
* **E1** = +0.600 V (same as +600 mV)
* **E2** = +0 V
* **Scan Rate** = 50 mV/s
* **# Scans** = 1
* **Meter** at Ecell, V or mV

Checking Parameters with the Dummy Cell

Pushing the **Scan/Step** button will initiate the scan from 0.000V to +0.600V and then return to -0.000 V.

Push the **Reset** button. Now repeat the experiment with the Dummy cell, except set **# of Scans** to 2.

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At the end of each experiment, push the **Reset** button. If you were actually running an experiment with the **Cell On**, you should switch back to **Cell Off** before disconnecting the electrodes.

Ready to Run a CV Experiment with the Real Cell?

Set # of Scans to 1 and switch **Cell On**. Be sure your X-Y recorder is ready to record the CV current-voltage curve. Push the **Scan/Step** button. If the recorder pen goes off-scale, either change the recorder sensitivity setting or the Sensitivity on the Omni 101 Potentiostat. You can stop the scan at anytime during the scan by pushing the **Hold** button.

Congratulations, you have successfully run your first CV experiment! You can change the scan rate to see the dependence of the peak current on scan rate, or change the filter setting to see the effect of the response time on the noise level.

Things to be Aware of with the Omni 101 Potentiostat

* The Omni 101 Potentiostat gives you voltammograms (i-E curves) using the Polarographic convention: anodic scan direction is from right to left with anodic current negative and downward. You can reverse these directions (signal polarity) by reversing the connectors to the X-Y recorder from the banana plugs located on the back panel of the potentiostat. If you reverse the connectors, the recorder inputs must be truly differential and floating, or the signal will be grounded.

* For measuring very low currents below 100 nA or so, you will need to place the cell inside a Faraday Case, and connect the instrument GRD to the case. At a setting of 100 nA/V, it is possible to measure currents less than 100 pA with the recorder Y-axis set at 10 mV/in. It may be necessary to use the highest setting of the noise **Filter** (100 ms).

* The discharge of static electricity can damage the electronics of most high-sensitivity instruments such as the Omni 101 Potentiostat. In a low-humidity environment (cold days in winter), discharge any static electricity prior to touching the potentiostat or any other electronic device in the laboratory.

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Amperometry and Chronoamperometry (CA)

Amperometry or sometimes referred to as **Controlled Potential Electrolysis** or **Bulk Electrolysis** is a method where the potential to the cell is kept at a constant value and the cell current is monitored as a function of time. Usually, a strip-chart recorder is used rather than an X-Y recorder with its limited time span in the Y-T mode. With **Ei**, you can set the desired potential (WKG vs. REF). Leave the **Reset** LED button lit. Select an appropriate current sensitivity value and recorder span. Activate the recorder. Switch from the **Cell Off** to **Cell On.** Everything is ready.

Chronoamperometry is a method where the potential is stepped from an initial value to a final value, and the resulting cell current is measured as a function of time, t. You can step back to the initial value, if you wish, after the initial step for Double-Potential-Step Chronoamperometry. The CA Section buttons control the cell functions. For example, selecting the **Ei** to **E1** choice and pushing the **Scan/Step** button, switches the applied potential from **Ei** to **E1** and gives you the initial step (e.g., 0.000 V to +0.600 V). This may be followed after time, t, by switching back to initial by pushing the **Reset** button. The length of the step is determined by when you push the buttons. If you do not wish to do the switching manually, you can connect an external signal generator to the Omni 101 Potentiostat via the **EXT. In** connector located on the front panel. This external voltage is added to the voltage being applied by the internal signal generator of the potentiostat. If you wish the internal signal to be 0.000 V, set **Ei** to 0.000 V.

Chronoamperometry Experiment Setup

1. Plug in the Omni-101 and turn on the power.

2. Enter (Initial Potential), **Ei**
Select **Ei** by pressing the parameter select button until the LED next to the **Ei** is lit. The meter will display the default value for the **Ei**. Enter a new **Ei** as described previously.

3. Enter **E1**, **or E2**
Select **E1** by pressing the parameter select button until the LED next to the **E1** is lit. The meter will display the default value for the **E1**. Enter a new **E1** as described previously. You may also enter another potential step value for **E2**. Enter a new **E2** as described previously.

4. Select the step needed. There are two choices, i.e., step from **Ei** to **E1** or Step from **Ei** to **E2**.

5. Sensitivity
Adjust sensitivity knob to the desired value.

6. Filter
Adjust filter knob to the desired value.

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7. **After checking that the cell cables are connected to the appropriate electrodes and selecting the Meter to display the Cell Current, turn the cell on.**
8. **After the cell current has settled down, start the Step by pressing the Scan/Step button.**

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Stripping Voltammetry for Analysis of Trace Metals with the Omni 101 Potentiostat

Stripping Voltammetry concentrates metal ions, such as Pb(II), Cd(II), Cu(II) or Zn(II), onto an electrode surface by electrodeposition and then "strips" the metal from the surface by anodic oxidation. Because the metal is concentrated at the electrode, the stripping current is much larger than if one had looked directly at the voltammetric current for the metal-ion reduction.

The initial set-up is similar to that for amperometry except that the INIT potential usually has a negative value such as -0.6 V if one is analyzing for Pb(II) and Cd(II). After maintaining the voltage of -0.6 V (WKG vs. REF) for 5 minutes, stirring is stopped and the potential is then scanned from -0.6 V to 0.00 V.

Stripping Experiment: Assuming that you have a solution of 0.01 mM Pb(II) in 0.01 M KNO_3, a suggested check list of instrument settings is as follows:

* Current sensitivity 1 μA/V and Y-axis of recorder set at 100 mV/in
* Filter 20 ms
* Scan Rate at 500 mV/sec
* **Reset** Lit
* **Cell Off**
* Set **E1** and **E2** to 0.000 V

With the Meter set to Ecell, the DVM should display -0.600 V. This potential will be applied to electrodeposit metal ions that can be reduced at this potential. Turn the stirrer on; it is important to stir at the same rate for all the electrodepositions in order to obtain reproducible results. Deoxygenate the solution by bubbling nitrogen through it for 2-3 minutes.

Switching from **Cell Off** to **Cell On** connects the cell so that you are now electrodepositing metals onto the working electrode surface. When you have completed the time duration desired for the deposition, stop the stirrer and allow the solution to quiet (ca. 1 minute), and push the **Scan/Step** button. The potential will scan from -0.6 V to 0.00 V at the preset scan rate of 50 mV/s. Be certain to switch the toggle from **Cell On** to **Cell Off** prior to disconnecting the connectors to the electrodes.

WARNING NOTES:

* Use of high-purity water is important.
* Avoid high concentrations of supporting electrolyte since these reagents often contain heavy-metal contaminants at ppb to ppm levels.
* It may be necessary to use longer deposition times if the metal ion concentration is very small (e.g., < 10^{-7} M).
* The stirring rate, the deposition time period, and the resting period before the anodic scan all must be constant in order for the electrodepositions to be reproducible.

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* Choice of supporting electrolyte is very important, e.g., the presence of chloride ion will affect the results for Pb analysis.
* Metals can form intermetallic compounds which complicate the oxidative behavior; thus, co-deposition of several metals simultaneously can cause variations in the results.

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Initial Set-up

A. Power

Connect AC power cord to power connector on back of Omni-101.

B. Connections to electrodes

Be sure that electrodes are carefully inserted into the solution of electrolyte and analyte, positioned so the stirring bar will not strike them. Attach each electrode to the corresponding terminal on the top of the Omni 101 Potentiostat, using the electrode cable supplied:

Red terminal	to Auxiliary electrode
White terminal	to Reference electrode
Blue terminal	to Working electrode

C. Connections to recorder

1. Two BNC jacks on the Front panel are labeled Ecell and Icell. For cyclic voltammetry, both connections to the X-Y recorder are used. For chronoamperometry, only the I_{CELL} is used for the Y-axis of the recorder; the X-axis is a time function set on the recorder which is used in the Y-T mode. If a strip-chart recorder is used, only the Icell connection is used.

2. BE SURE THAT GROUND OF THE Omni 101 Potentiostat IS CONNECTED TO GROUND OF THE RECORDER!

3. **Conventions:** The Omni 101 Potentiostat will present voltammograms in the Polarographic convention with anodic potentials to the left and the anodic current toward the bottom of the paper. To have voltammograms displayed in the IUPAC convention, switch the connections at the XY recorder. However, be sure that the recorder inputs are truly differential in nature if you wish to reverse the signal polarity.

D. Grounding

1. The Omni 101 Potentiostat may be grounded by running a wire from the banana GRD jack on the instrument case to a water pipe or other good earth ground.

2. For special cases, other grounding concerns may arise:
   If it is desired to "float" the instrument in order to avoid cross talk with other apparatus connected to the same cell, care must be taken to avoid grounding via connections to recording devices.

E. Connections to external signal source

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An external signal source can be connected to the BNC jack on the front panel labeled **Ext. In,** to provide more complex signals than available from the internal generator of the Omni 101 Potentiostat.

The composite waveform resulting from use of an external source may have voltages that exceed those the Omni 101 Potentiostat can generate internally. Note that the sum of the voltages cannot exceed ± 5 V.

The potential applied to the cell may be thought of as the sum of two components: an internally-generated base consisting of the step, ramp or plateau determined by the choices made with the front-panel controls and an externally generated waveform applied to the **Ext. In** jack from some other signal generator.

For full waveform fidelity, restrict sine-, square-, or triangle-wave inputs to 1 kHz. The input impedance at the **Ext. In** jack is 50 kilo-ohms. Signal generators with output impedances of 600 ohms or less should work well.

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Experiments for the Omni 101 Potentiostat

1. **Cyclic Voltammetry**

2. **Chronoamperometry**

3. **Linear Sweep Stripping Voltammetry**

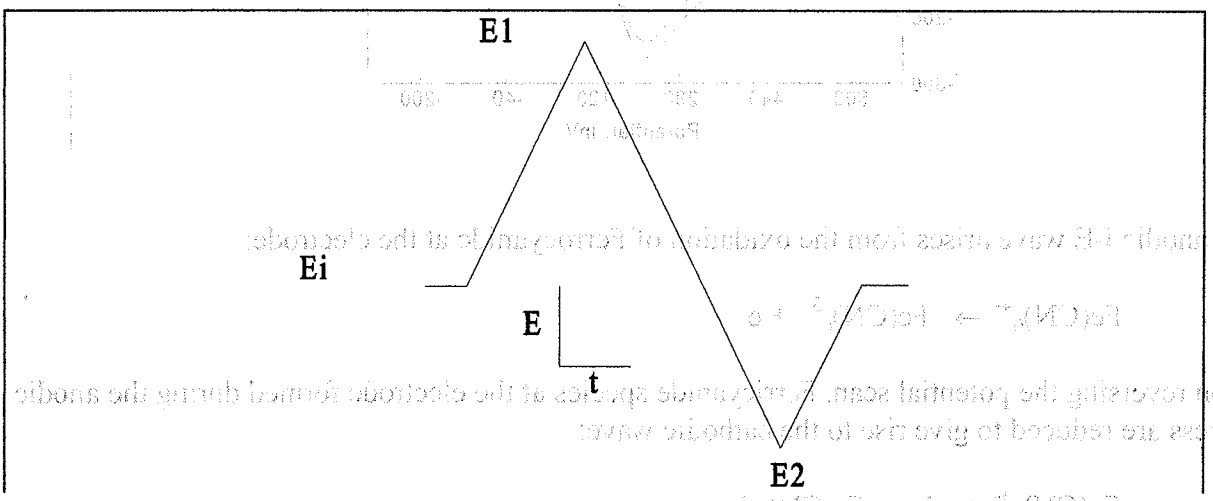
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Cyclic Voltammetry

I. Waveform and a typical Cyclic Voltammogram

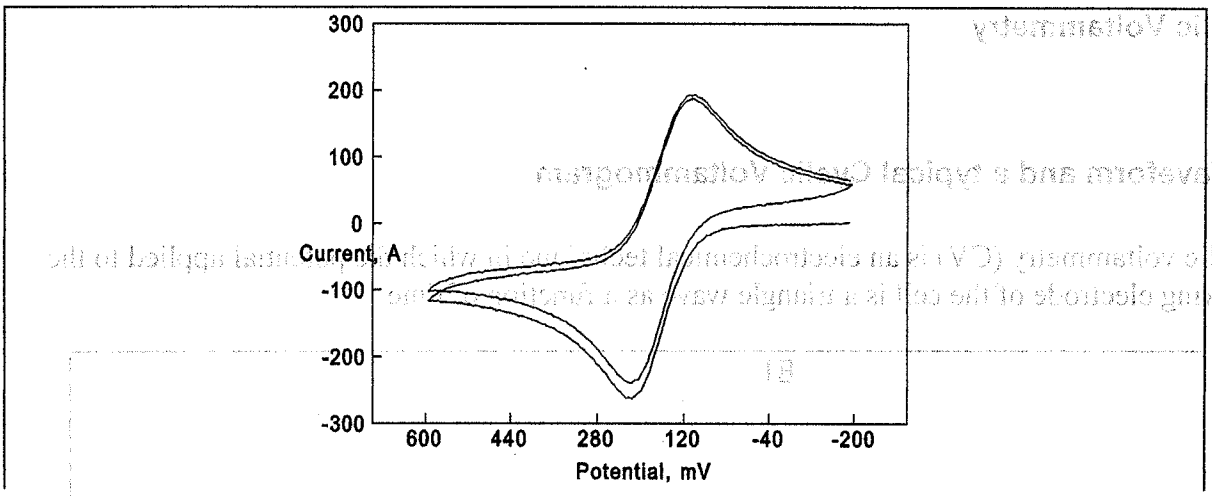
Cyclic voltammetry (CV) is an electrochemical technique in which the potential applied to the working electrode of the cell is a triangle wave as a function of time.



Typically, the signal has a uniform rate of potential change (scan rate), and the potential scan direction is reversed when the predetermined anodic and cathodic limits are reached. This technique also is called linear-scan or linear-potential-sweep voltammetry.

The nature of the current, which flows through the working electrode, is observed as a function of the scanned potential. The reversal of the potential scan direction can give rise to current flows due to reaction of products formed in the preceding scan period. A voltammogram is the plot of the cell current as a function of the electrode potential. A typical voltammogram for Ferrocyanide reacting at a platinum electrode appears in Figure 2.

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The anodic i-E wave arises from the oxidation of Ferrocyanide at the electrode:

$$Fe(CN)_6^{4-} \rightarrow Fe(CN)_6^{3-} + e^-$$

Upon reversing the potential scan, Ferricyanide species at the electrode formed during the anodic process are reduced to give rise to the cathodic wave:

$$Fe(CN)_6^{3-} + e^- \rightarrow Fe(CN)_6^{4-}$$

In CV, you usually choose the initial potential to be a value where little or no electrode reaction occurs (near-zero current). The anodic and cathodic scan limits are determined by the redox potential of the electrochemical reaction and by the background limit which is dependent on the solvent, electrode and supporting electrolyte.

For convenience, the scan rate can be set to higher values to shorten experimental time, but its selection plays an important role in studying electrode reaction rates as well as chemical reactions involving species which are oxidized and reduced at the electrode. Finally, whether one, two or more successive scans are needed is determined by interest in studying behavior of electrode reaction products. In simple electrode reactions such as the Ferrocyanide-Ferricyanide conversion, successive voltammograms differ little from the first. In a more complex case such as the oxidation of p-aminophenol, chemical conversion following the electrode oxidation gives rise to multiple cathodic waves as more than one species is reduced when the potential scan is reversed. For more information on this technique, see A.J. Bard and L.R. Faulkner, "Electrochemical Methods," Wiley, New York, 1980, Chapter 6.

II. Summary of procedure

1. Plug in the Omni-101. Make certain that **Cell On/Standby/Cell Off** switch is in the **Cell Off** position. Turn on the power.

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2. Enter (Initial Potential), **Ei**
   Select **Ei** by pressing the parameter select button. The LED next to **Ei** will remain lit and the meter will display the default value for the **Ei** with the units digit flashing. Enter a new **Ei** as described in Button Function Section.

3. Enter **E1** limit
   Select **E1** by pressing the parameter select button until the LED next to the **E1** is lit. The meter will display the default value for the **E1**. Enter a new **E1** as previously described.

4. Enter **E2** limit
   Select **E2** by pressing the parameter select button until the LED next to the **E2** is lit. The meter will display the default value for the **E2**. Enter a new **E2** as previously described.

5. Enter Scan **Rate** (mV/sec)
   Press the button under the CV on the front panel until the LED next to the **Rate** is lit. The meter now will display the default value for the **Rate**. Enter a new **Rate** as previously described.

Once the parameters are entered as desired it is important to press the Reset button before proceeding. The Reset button in this instance acts as an ENTER key. It signals the microprocessor that new parameters have been entered and will update the microprocessors program memory for the upcoming experiment.

6. Select the desired number of scans.

7. Sensitivity
   Adjust sensitivity knob to the desired value.

8. Filter
   Adjust filter knob to the desired value.

9. After checking that the cell cables are connected to the appropriate electrodes and selecting the Meter to display the Cell Current, turn the cell on by rotating the cell on/off switch to the cell on position. There is an intermediate position between **Cell Off** and **Cell On**, the **Standby** position. If the knob is left in this position the Open Circuit potential can be monitored on the meter or through the XY recorder.

10. After the cell current has settled down, start the scan by pressing the **Scan/Step** button. The meter will by default show the cell potential. This can be changed during the run by pressing the meter button.

III. The Cyclic Voltammetric Experiment

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A. Connections <u>DO NOT TURN ON THE POTENTIOSTAT YET!</u>

1. The recorder should be connected to the jacks on the front the of the Potentiostat. For CV, both pairs of jacks are used: the Cell current to the Y axis of the recorder, and the Cell voltage to the X axis.

2. Electrodes should be clean and carefully inserted into a cell containing the species to be studied in an electrolyte solution. De-oxygenate the solution if oxygen can interfere with the experiment. If the solution is stirred, be careful that the stirring bar does not hit the electrodes.

3. Connect the electrode cable between the electrodes and the connector on the front of the Omni 101 Potentiostat [**Red** to AUXiliary; **White** to REFerence; **Blue** to WorKinG]. Keep the electrode leads as short as possible to minimize resistance. For high-sensitivity, low-current measurements, place the cell inside a Faraday shield case which is connected to the ground of the potentiostat.

4. BE SURE THAT GROUND OF THE OMNI 101 Potentiostat IS CONNECTED TO GROUND OF THE RECORDER!

B. Check before turning on the Omni 101 Potentiostat

Disconnect the external cell by switching the toggle from CELL On to CELL OFF.

C. Turn on the potentiostat using the power switch on the front panel

D. Set the electrochemical parameters for your electrodes and solution

1. **Determine number of scans desired (1, 2, ∞):**

   a. A single scan starts at the Initial Potential, **Ei**, proceeds to the **E1** limit and then to the **E2** limit. The scan logic is set for two reversals of scan direction.

   b. For a single scan, set the **# of Scans** toggle to the position labeled "1"; for successive scans, set to "2". Continuous scanning (∞) is used for obtaining a steady-state voltammogram, for pretreating electrodes, or for studying species generated in the first scan.

2. **Set an appropriate scan rate to determine how quickly the potential changes.**

   a. First set the LED to **Rate** so the scan rate is read.

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b. To change the numbers shown on the meter display, press the button under the CV on the front panel until the LED next to the **Rate** is lit. The meter now will display the default value for the **Rate**, 100 mV/sec. One digit will be flashing. This can be changed using either the up or down arrow. To change to the next digit over, press and hold one of the arrow buttons and then press the other arrow button to move to the next digit to change. Any value between ± 2000 mV is acceptable for this parameter.

d. Voltammograms at small solid electrodes often are obtained at scan rates of tens of millivolts per second.

4. **Set the Ei, E1, and E2 limits**

Following the previously described procedure, enter the values for **Ei**, **E1**, and **E2**.

5. **Set the filter time constant value.**

a. The switch marked **Filter** determines how much noise is removed from the current output to give a smoother curve.

b. Events that occur more rapidly than about twice the selected time constant will be distorted. For highly precise readings, allow a five time constant period for the current readings to settle down. The 100 nA/V setting has an effective time constant greater than the labeled values.

6. Set the Sensitivity.

This determines how much the output to the panel meter and the recorder will be amplified to work with different levels of current generated in various experiments.

a. If the setting is 10 mA/V, then a 1-volt displacement will appear on the recorder whenever a current of 10 mA flows through the working electrode.

b. This is used with the recorder ranges (volts per inch or per centimeter) to determine the scale of the plot.

c. The setting of 100 nA gives maximum sensitivity but requires careful shielding of the electrochemical cell and the electrode leads. You will find it necessary in most cases to house the cell in a Faraday shield case connected to the potentiostat ground.

d. When setting the desired current sensitivity with the **Cell Off**, the Current overload light may be continuously lit. This will occur when the initial voltage applied to the 1 MΩ dummy cell results in the current-measuring amplifier output exceeding ca. 9 V. For example, an initial potential of 1 V applied to the dummy cell resistance will

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result in a 1-μA current. If the current sensitivity is set at 100 nA/V, the current-measuring amplifier is overloaded, i.e., 10-V output is demanded, and the overload light will be lit.

E. Display

1. Set the **Meter** to **Ecell**, either V or mV, so that you can watch the potential excursions.

2. Notes on scanning: You can change the scan direction at any time during the experiment by pressing the **Change Scan Direction** push button.

F. Switch from the dummy cell to the external cell

1. The overload LED may light briefly. If it does not go out, reduce the current sensitivity.

2. You may wish to run a test scan without the pen down to check the current sensitivity setting. If so, stir the solution before you repeat the scan.

G. Start the scan(s)

1. Be sure the recorder is on, with paper and the pen down.

2. Push the **Scan/Step** push button.

   a. The voltage will scan until the selected limit is reached. Then, the scan direction will be reversed until the other limit is reached. At any time during the scan the direction may be changed by pressing the Change Scan Direction push button.

H. Use of Reset or Hold to stop the scan

1. Scanning will stop automatically at the end of a scan if it is set for 1 or 2.

2. To stop during a scan, push either:

   Reset where the potential is returned to the initial value

   or

   Hold where the potential stays at the value it had when the switch was made. The potential may gradually drift toward zero over time. This drift also is seen at the end of the scan when the potential is at its final value (about 2 mV in first minute,

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then 4 mV/hr). Generally, the **Hold** position is used only for momentary holds during preliminary experiments.

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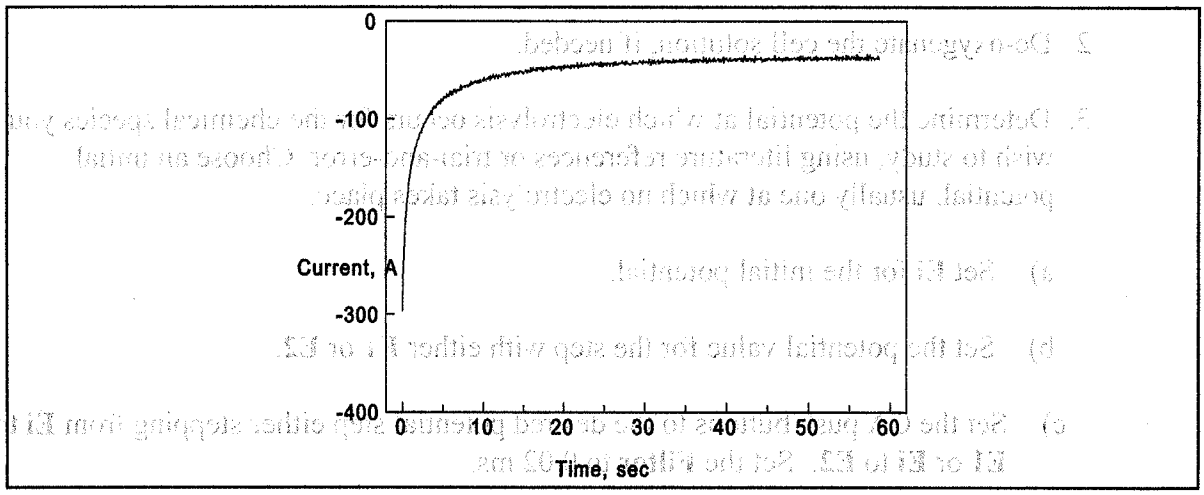
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Chronoamperometry

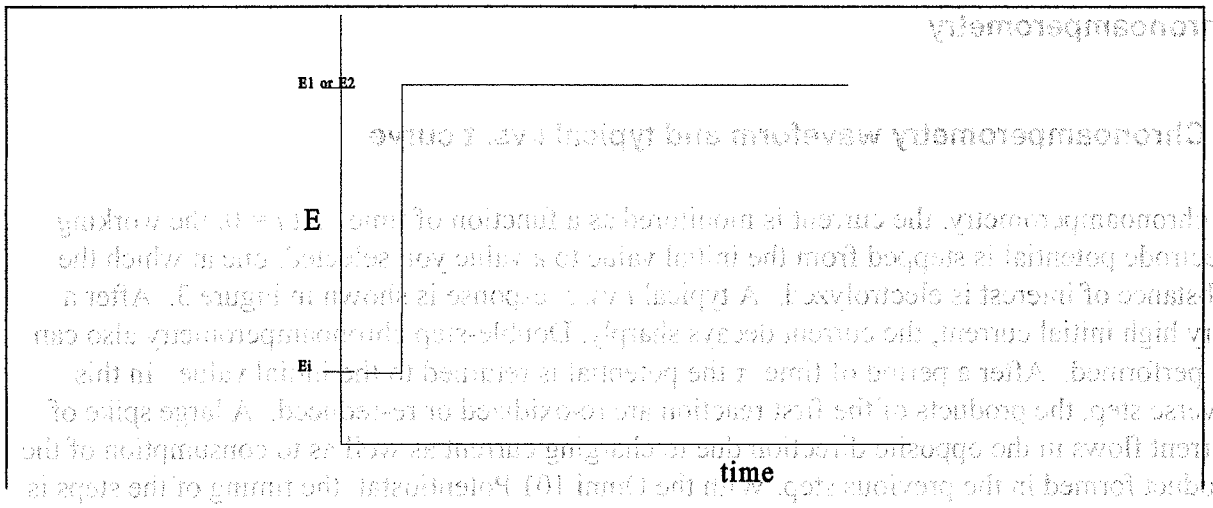
I. Chronoamperometry waveform and typical i vs. t curve

In chronoamperometry, the current is monitored as a function of time. At $t = 0$, the working electrode potential is stepped from the initial value to a value you selected, one at which the substance of interest is electrolyzed. A typical i vs. t response is shown in Figure 3. After a very high initial current, the current decays sharply. Double-step chronoamperometry also can be performed. After a period of time, τ the potential is returned to the initial value. In this reverse step, the products of the first reaction are re-oxidized or re-reduced. A large spike of current flows in the opposite direction due to charging current as well as to consumption of the product formed in the previous step. With the Omni 101 Potentiostat, the timing of the steps is controlled by the user. The waveform for the applied potential is shown in Figure 4.

For more information on this technique, see A.J. Bard and L.R. Faulkner, "Electrochemical Methods," Wiley, New York, 1980, Chapter 5.



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II. Summary of Chronoamperometry procedure

1. Check cell, electrodes, power and recorder connections.

2. De-oxygenate the cell solution, if needed.

3. Determine the potential at which electrolysis occurs for the chemical species you wish to study, using literature references or trial-and-error. Choose an initial potential, usually one at which no electrolysis takes place.

   a) Set **Ei** for the initial potential.

   b) Set the potential value for the step with either **E1** or **E2**.

   c) Set the CA push buttons to the desired potential step either stepping from **Ei** to **E1** or **Ei** to **E2**. Set the **Filter** to 0.02 ms.

4. Choose the **Sensitivity** values suitable for the conditions. This may require trial-and-error. Start with less sensitive values to avoid overdriving the current-measuring amplifier and making the recorder pen go off-scale.

5. Set up the recorder in a Y-T mode, with the cell current to be recorded as a function of time.

6. Usually, you will start recording current vs. time before applying the potential step to ensure that current flow at the initial potential has reached a steady-state value.

7. After checking that the cell cables are connected to the appropriate electrodes and selecting the Meter to display the Cell Current, **Icell**, and turn the cell on.

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8. Stir the solution briefly, allow the solution to come to rest (ca. 1 min), then activate the recorder. When the current reaches a steady-state value, you are ready to apply the potential step. Apply the Step by pressing the **Scan/Step** button.

9. If you want a double-potential step, wait the appropriate period of time, then press the **Rest** push button.

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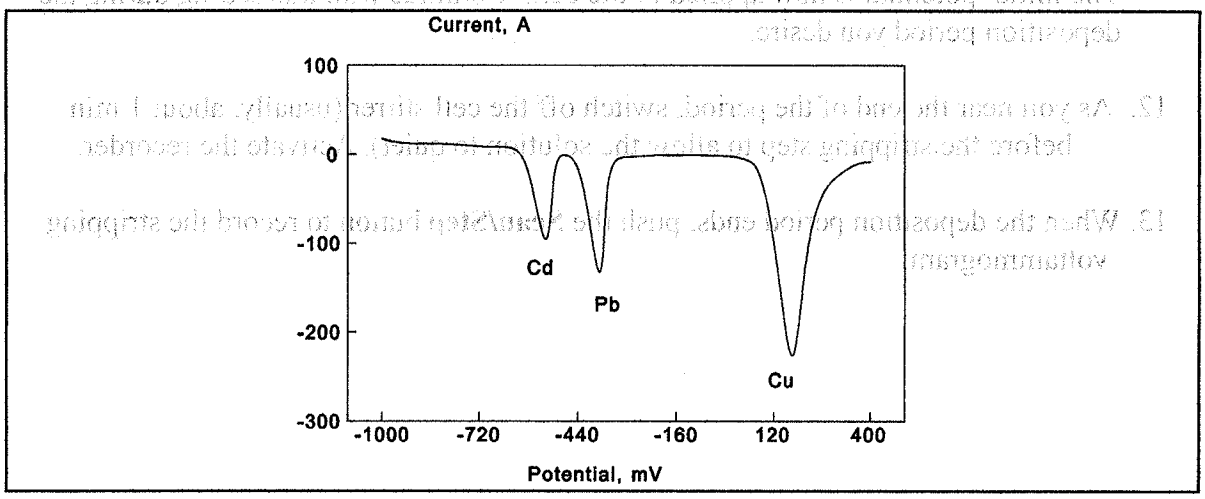
Linear-Potential-Scan Stripping Voltammetry

I. Waveform and typical curve

Stripping techniques electrochemically concentrate metal ions from the solution and therefore are particularly useful for analyzing trace amounts easily and rapidly. To deposit metals on the electrode surface, the electrode potential initially is held at a constant value a few tenths of a volt more negative than the half-wave potential of the ions of interest. The solution is stirred during the deposition. The electrolysis period is measured and should be longer for more dilute solutions. Five minutes is often sufficient.

After this electrolysis period, the electrode potential is slowly scanned to more positive potentials (linear-potential sweep). This causes the deposited metals to be redissolved and stripped off the electrode. Different metals oxidize off the electrode at different potentials. The resulting anodic current is recorded as a function of applied potential.

Metal-ion concentrations are proportional to the peak currents after correction for the background current. Concentration also is dependent on electrolysis time. If more than one metal is present, peaks past the first peak must also be corrected for tailing from previous peaks. Since solutions are not exhaustively stripped, care must be taken that deposition time, potentials used, stirring rate, and electrode size be carefully matched to those used in calibration.



Brief summary of procedure

1. Check cell, electrodes, power and recorder connections.

2. De-oxygenate the cell solution, if needed.

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3. Determine the **Ei**, **E1**, and **E2** limits to be used. Set potentials appropriate to the chemical species you wish to determine. You may want to obtain a cyclic voltammogram first to check the conditions.

4. Choose the **Sensitivity** suitable for your experiment. Again, this may require trial-and-error. Start with less sensitive Sensitivities to avoid overdriving the current-measuring circuit.

5. Adjust the **Ei** for the value at which the deposition takes place.

6. Select the appropriate scan rate for the stripping portion of the experiment and set this value.

7. Set the scan limit to the same value with <u>both</u> the **E1** and **E2** so the scan proceeds from the initial deposition potential to the final stripping potential limit.

8. Set the **# of Scans** switch to 1 and push the **Reset** button.

10. Turn on the stirrer in the electrochemical cell.

11. Activate your stop watch or lab timer at the same time that you switch to **Cell On**. The initial potential is now applied to the cell. Continue with this setting during the deposition period you desire.

12. As you near the end of the period, switch off the cell stirrer (usually, about 1 min before the stripping step to allow the solution to quiet). Activate the recorder.

13. When the deposition period ends, push the **Scan/Step** button to record the stripping voltammogram.

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Acquire 101 Software

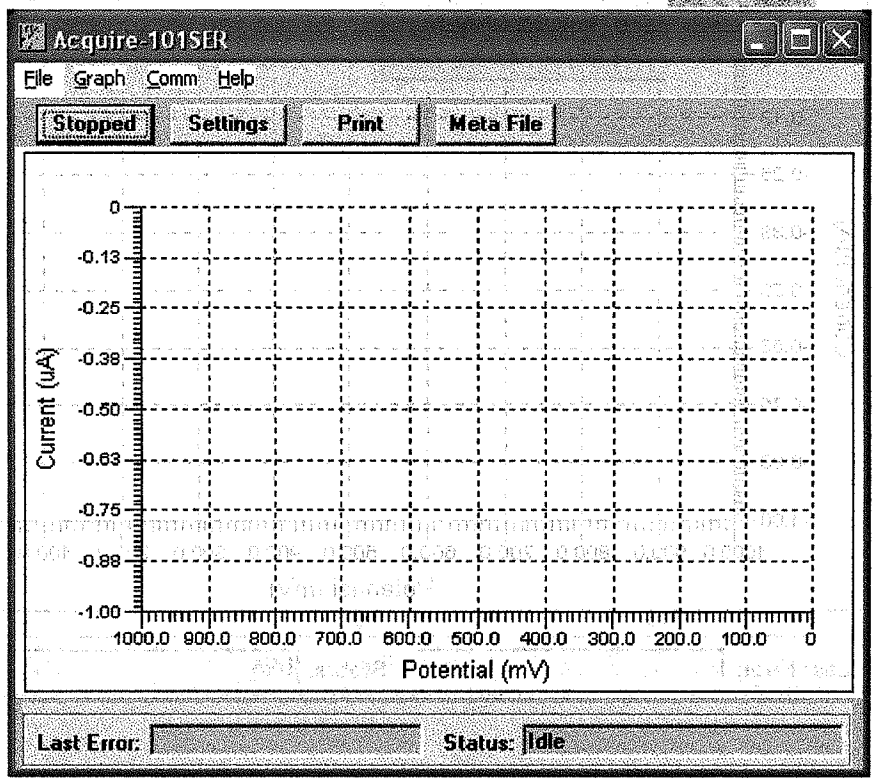
By connection to the 9-pin RS-232 jack at the back of the Omni-101 you may collect data from your Omni-101 with your computer. The software covers two modes of experiments, for cyclic Voltammetry and Chronoamperometry.

The RS-232 kit contains the following items:

1 Serial Data Collection Software disk
1 Serial Communication cable

Getting Started

Select the appropriate Com Port for the connection to the computer in the Comm Menu. Select the type of experiment to be performed either Cyclic Voltammetry or Chronoamperometry in the Graph Menu. Also X and Y-axis Scaling here. In the Current Scaling, make sure the sensitivity setting follows the setting of the Omni-101 to ensure that the current data is scaled correctly.



Once the plot is setup to collect data for the particular experiment, press the "Stopped" button and the system will be setup and waiting for the data. The button will change from "Stopped" to "Running" and the Status message window at the lower right on the screen should change from "Idle" to "Waiting for data".

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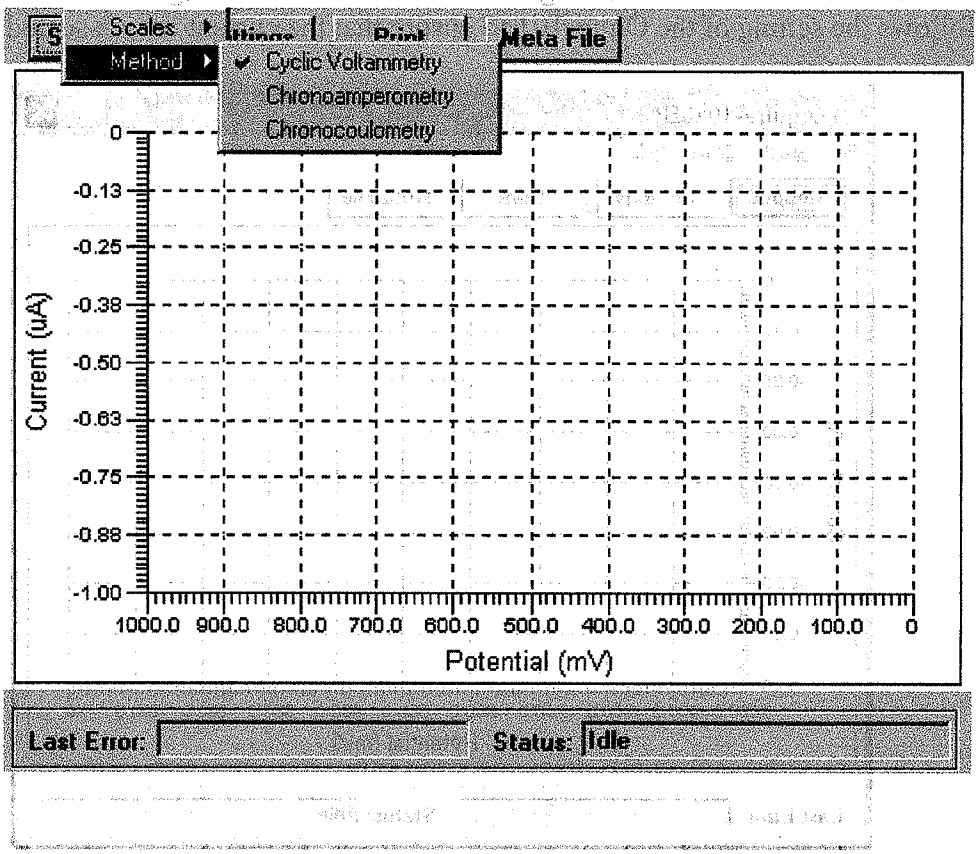
Data will start to be displayed when the Scan/Step button on the Omni-101 is pressed. The number of data points will start to be displayed in the Status window.

Experiment Setup

There are several steps to follow to setup both the Omni-101 and the Acquire software to run experiments. Since the software cannot control the Omni-101, the Omni-101 should be setup as described in the Omni-101 manual for the experiment desired. Next the software must be configured to communicate correctly with the computer by choosing the appropriate technique, either CV or CA, the COM port connection and Baud Rate speed.

Setting the technique or method

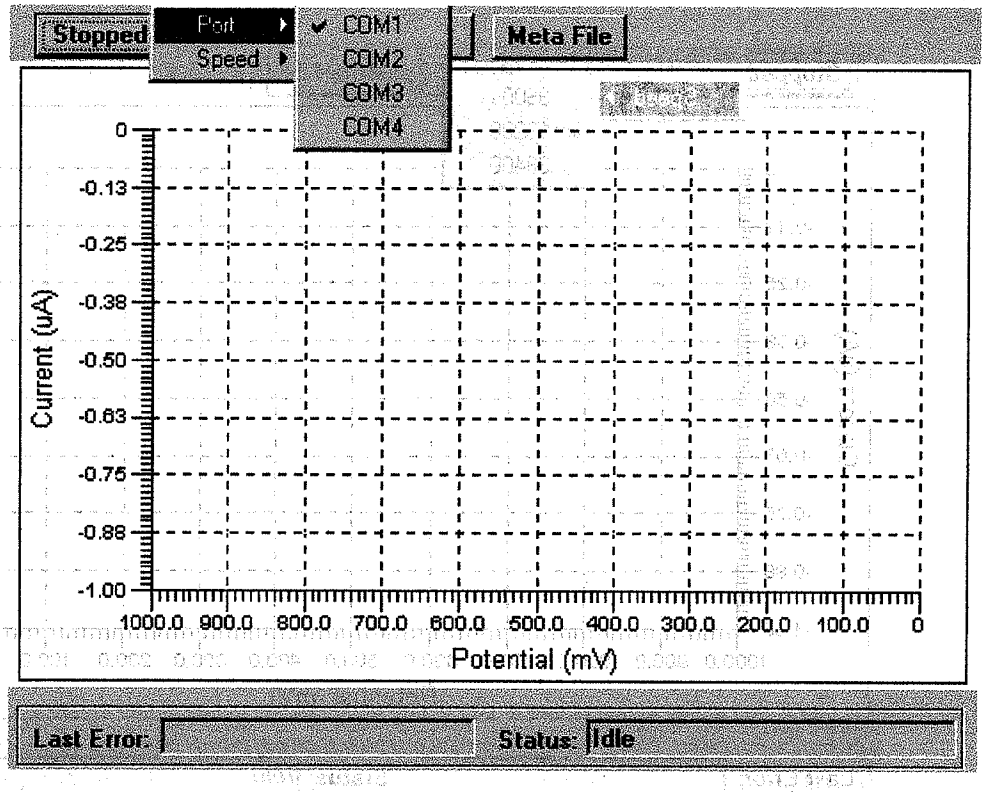
Under the Graph Menu, choose Method, then select either Cyclic Voltammetry, Chronoamperometry, or Chronocoulometry.



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Setting the COM port

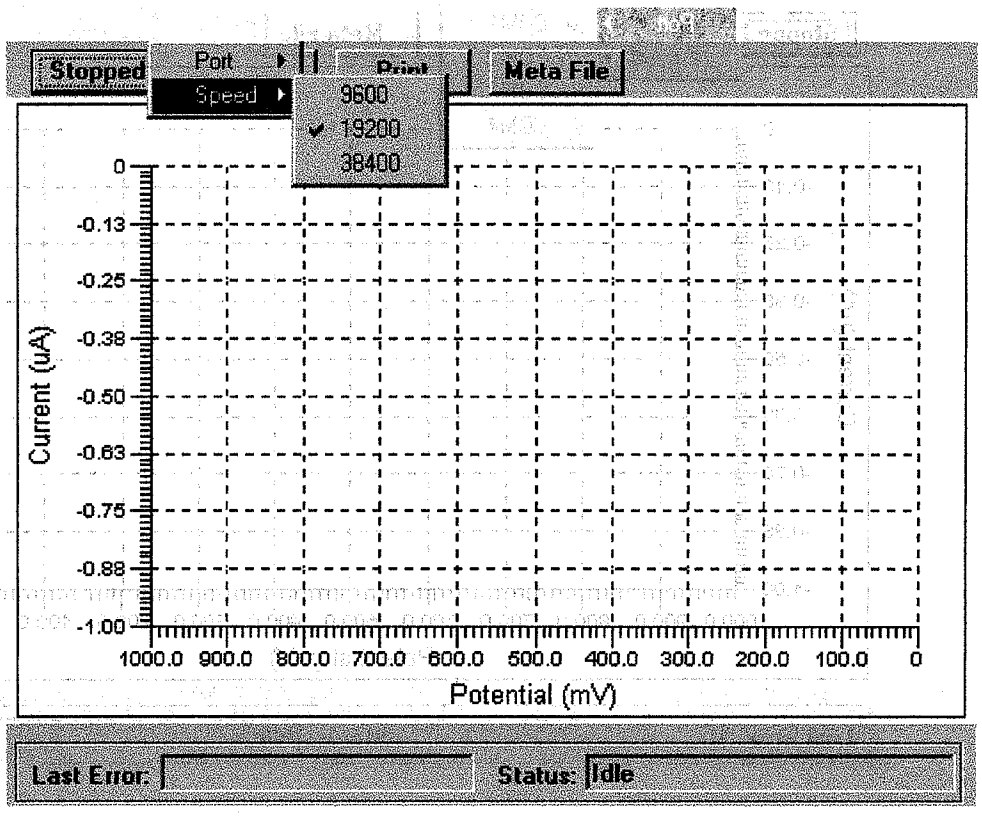
Under the Comm Menu, choose Port, select the communication port that you are connected to on your computer.



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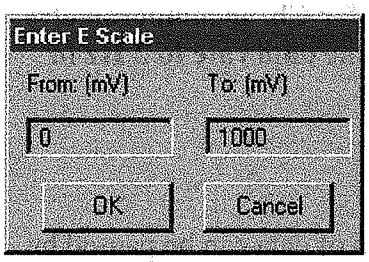
Setting the communication speed

Under COMM select Speed to set the baud rate. For the standard system, the 9600 baud rate should be selected. In the case of the new microprocessor for the high speed data acquisition, select the 19200 baud rate.



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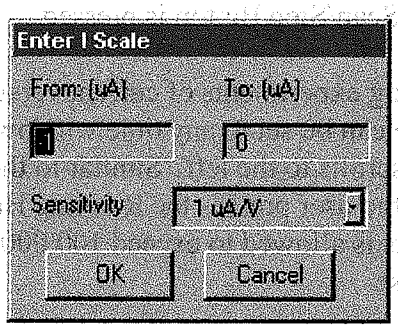
Setting the Potential Range



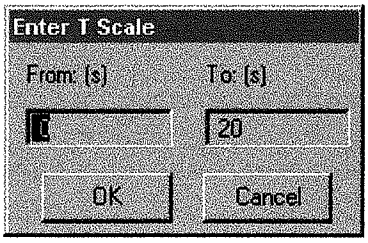
Under the Graph Menu select Scales, choose E Range. This Menu is used to set the plot screen for the minimum and maximum potentials. It is not necessary that these be exactly what is set on the Omni-101. **From** corresponds to the left hand corner of the plot and **To** the right hand corner of the screen.

Setting Current Range

Under the Graph Menu select Scales, choose I Range. The From/To boxes can be filled to any scales desired. However, the Sensitivity field must be identical to the setting of the Omni-101 to make sure the numbers displayed are correct.



Setting the Time Range



If Chronoamperometry is selected as the method, the X-axis will be time not Potential. Therefore, when Scales is selected, the Time scale of the X-axis can be set. The From/To limits can be set to cover the entire experiment or be setup to view smaller portions of the experiments. During the experiment as the data reaches the maximum limit for the Time range, the window display shifts to the left to always show the live data.

Data Point Limit

In the Chronoamperometry or Cyclic Voltammetry when Scans is set to infinity, the number of data points will be collected up to 100,000 points, at which time, the data in RAM will be overwritten by the new data coming in. At any time the RUN button can be clicked and the data collection is stopped and the data can be saved to the hard disk.

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Note

For the high speed data acquisition, the data rate is fixed at 300 Hz. This speed allows adequate data resolution for the faster scan rates in CV, however, for Chronoamperometry this data rate will quickly fill the graph buffer. We have reduced the data rate to 2 Hz in the Acquire 101 software.

Pausing the Data Collection

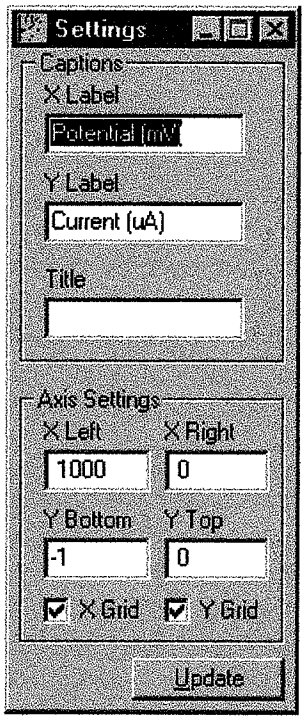
During a current versus time data collection experiment, the collection of data can be paused by pressing the Initial button on the Omni-101. The applied potential will now step to the value setup for the Initial Potential, Ei. If you do not wish the potential to change during a pause make sure the value setup for Ei is the same as the value of E1 or E2. Data collection will resume when the Scan/Step Button is pressed.

During long experiments of current versus time for example FIA or HPLC experiments. The time scale can be set to any time window desired. When the data reaches the X-axis maximum the chart scrolls to the left. Similar to the way an oscilloscope screen looks. It will continue like this until the user presses the Running Button to stop the collection of data. If the number of data points is allowed to exceed 100,000, the data will continue to scroll but all previous data will be lost.

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Editing Plot Window

There is a second way to change the plot screen coordinates as well as label the axes. The menu to the right will appear. The X- and Y-axis labels and a title can be added or changed in this menu. X- and Y-axes coordinates can be changed . You can also turn on or off X- and Y-axis grid lines. When you are finished, click Update and the plot will change according to your parameters. The screen will repaint so any previous data will not be redrawn, however, the data is still saved in RAM for later data file storage.



Grid lines can also be added from this menu.

Saving Data Files

Once the data collection is sopped the data collected can be saved as a text file. Select Save under the File Menu.

Loading Data Files

Select Load from the File Menu and select the data file desired.

Printing Graphs

Click the Print Button to send the plot to the print manager to be printed.

Screen Image Capture

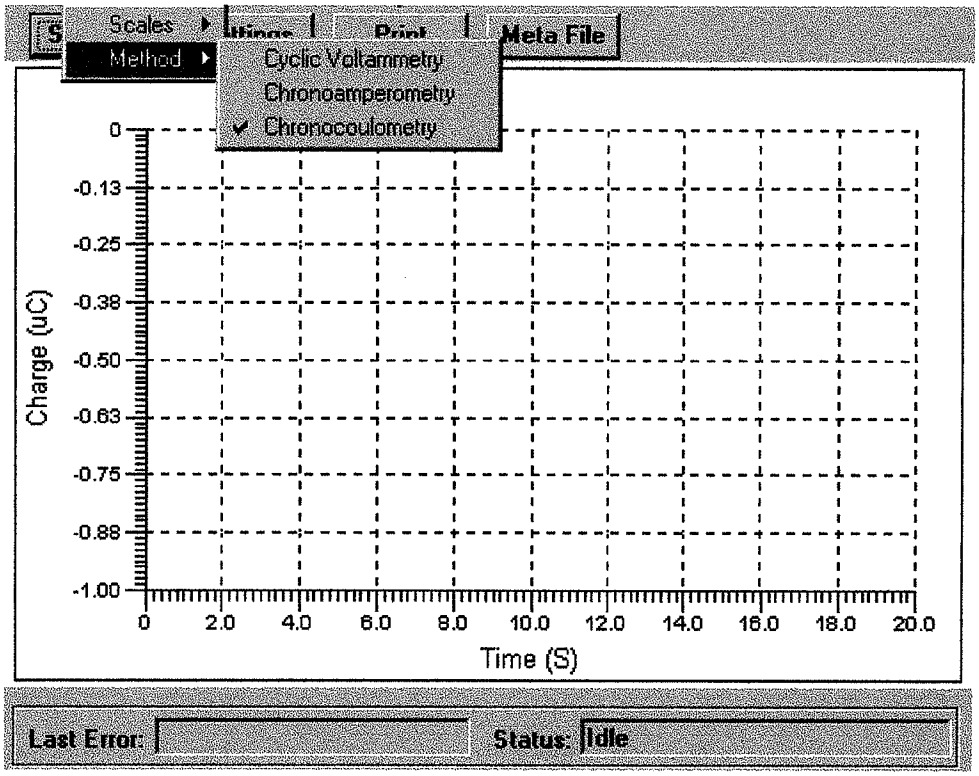
To capture the onscreen image to import to a word processing program use the Meta button to create a Windows enhanced meta file, emf, which can be imported into a word processing program.

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Chronocoulometry

A third option has bee added to the Graph / Methods Menu called Chronocoulometry



The data collection operates as in the Chronoamperometry method in all ways except instead of current versus time; the plot is the calculated accumulated charge, $\int i \cdot dt$, versus time.
The calculated charge is displayed as it is calculated and displayed on the plot. The display is as close to real time as can be achieved due to the fact that the plotted data pointed are calculated not direct signals.

Some other notes:

- The data files are saved with the .qc extension for later use and to distinguish them from other file types.
- As with all other techniques, the other menu setting must match the Omni-101 parameters for the calculated and displayed values to be correct.
- During the experiment, the charge axis can be expanded through adjustment of the y-axis in the Graph Settings Menu.
- The data will not start to be recorded until the Scan/Step button on the front panel of the Omni-101 is pressed.

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Precaution

Quantization Error

There have been some cases of our customers reporting problems with apparent noise or poor resolution in data collected via the RS-232 port and the Acqure-101 software. To this end we have been continually working on reducing noise and improving the performance of the Omni-101. However, we have found some cases where the problems reported were due to improper setting of the current axis scale in the -Acquire-101 software.

The data from the RS-232 port is 12 bit data from the ADC inside the Omni-101. So if for example the sensitivity of the Omni-101 is set to 1 µA/V the full-scale range of the current data is divided into 4096 digital numbers. Therefore, with the Acquire-101 y-scale is set to ± 1 µA the full scale range is 0 to 4096. However, if you set the scale of the current axis to ± 0.030 µA (± 30 nA) you are looking at only 123 digital numbers out the 4096. In this case the data displayed in the Acquire-101 plot will exhibit quantization error. The noise observed is actually the bit level transitions because you have zoomed in on the digital data. The Omni-101 is still set to the 1 µA/V.

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Below is data taken from the Acquire-101 software where the Omni-101 sensitivity is set to 10 µA/V and the y-axis scale is set comparable values.

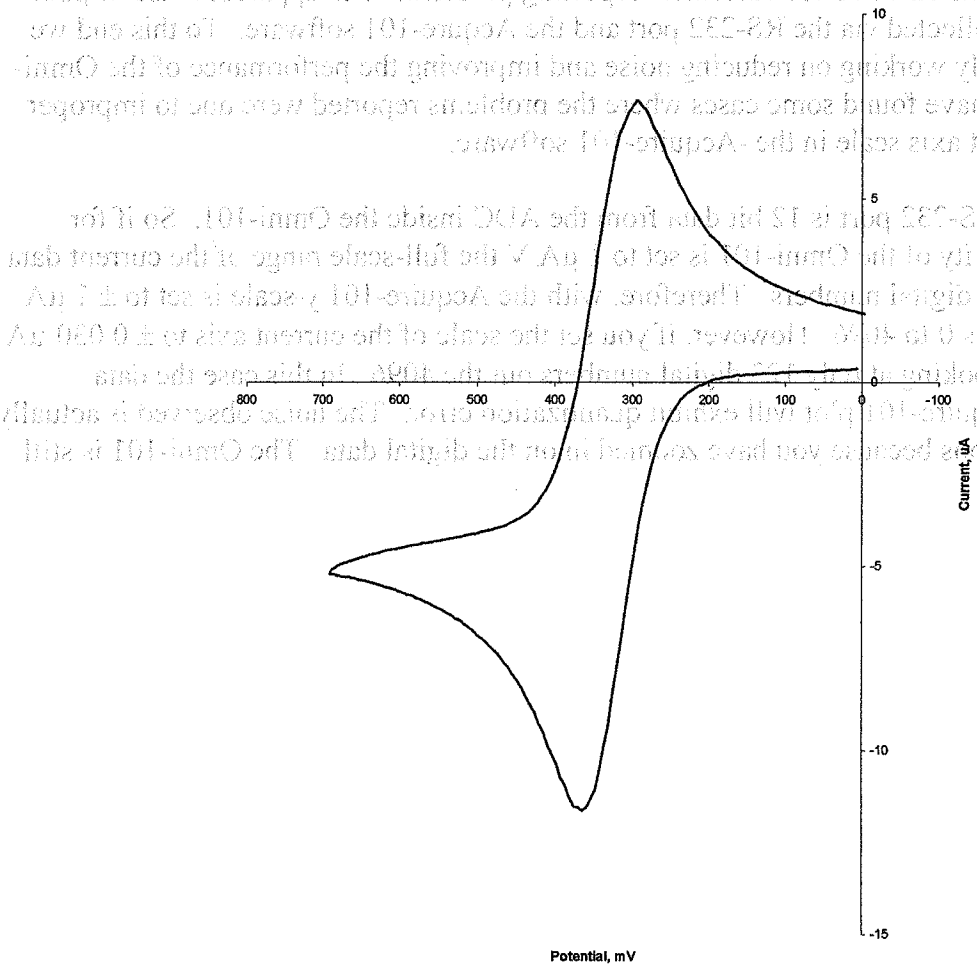


Figure 1. Ferrocene with 1 mm platinum electrode (EE006). Omni-101 set at 10 µA/V, 1 ms filter, scan rate 100 mV/sec.

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In contrast, the picture below is the same experimental setup but the Omni-101 is set to 1 mA/V sensitivity and we tried to see the same picture by adjusting the current scale in the Acquire-101 to the same range as the picture above.

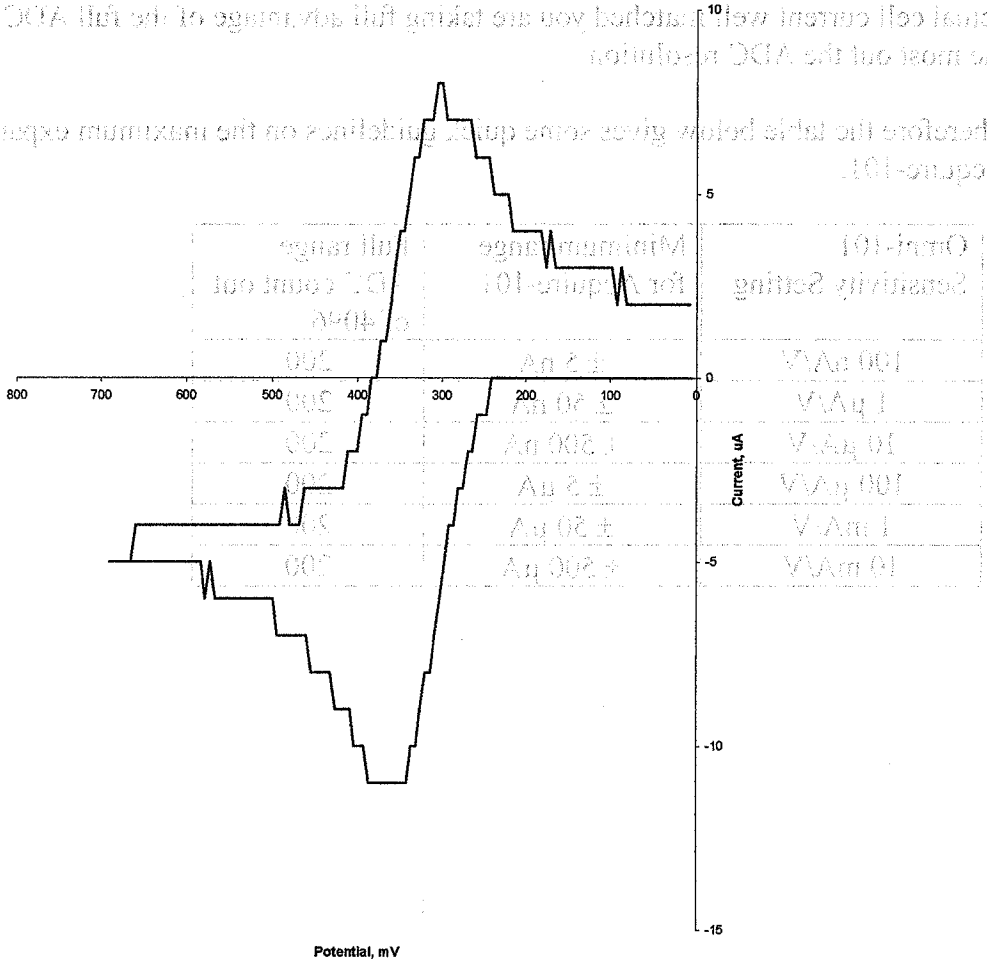


Figure 2. Ferrocene with 1 mm platinum electrode (EE006). Omni-101 set to 1 mA/V, 1 ms filter, scan rate 100 mV/sec.

As you can see, the output on the Acquire-101 plot shows that at a sensitivity of 1 mA/V is not a good match for the ADC resolution. The software expanded the current axis so much that the bit-to-bit transitions can be observed. At the 1mA/V range the bit-to-bit transitions have a higher value than if the sensitivity of the Omni-101 were changed to the 10uA/V range.

In the second case again the Omni-101 was set at 1 mA/V but the actual cell current was in the 10 µA range. With the Acquire-101 settings we are looking at only 1% of the total ADC range. Another way of looking at it is out of the 4096 bits we are only looking at approximately 40 ADC counts in the plot displayed in Figure 2.

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It is our suggestion that the most expansion one should set is 5% but even in this case the Acquire-101SER plot is looking only at about 200 counts out of the total 4096. It is always a good practice to match as closely as possible the sensitivity of the Omni-101 and the actual cell current and not use the Acquire-101SER to zoom in too much. With the Omni-101 and the actual cell current well matched you are taking full advantage of the full ADC range and getting the most out the ADC resolution.

Therefore the table below gives some quick guidelines on the maximum expansion (5%) for the Acquire-101.

Omni-101 Sensitivity Setting	Minimum range for Acquire-101	Full range ADC count out of 4096
100 nA/V	± 5 nA	200
1 µA/V	± 50 nA	200
10 µA/V	± 500 nA	200
100 µA/V	± 5 µA	200
1 mA/V	± 50 µA	200
10 mA/V	± 500 µA	200

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