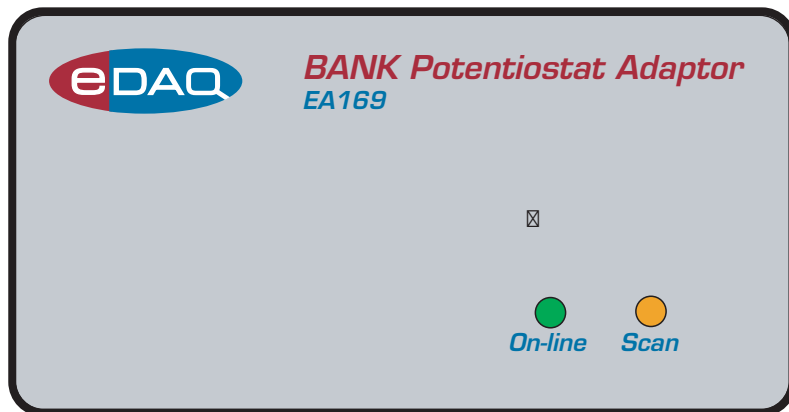


# *BANK Elektronik Potentiostat Adaptor*



This document was, as far as possible, accurate at the time of printing. Changes may have been made to the software and hardware it describes since then: eDAQ Pty Ltd reserves the right to alter specifications as required. Late-breaking information may be supplied separately. Latest information and information and software updates can be obtained from our web site.

### **Trademarks of eDAQ**

e-corder and PowerChrom are registered trademarks of eDAQ Pty Ltd. Specific model names, such as e-corder 201, PowerChrom 280, Picostat and QuadStat are trademarks of eDAQ Pty Ltd. Chart and Scope are trademarks of ADInstruments Pty Ltd and are used under license by eDAQ. EChem is a trademark of eDAQ Pty Ltd.

### **Other Trademarks**

Mac OS, and Macintosh, are registered trademarks of Apple Computer, Inc. Windows 98, Windows Me, Windows 2000, and Windows XP are trademarks of Microsoft Corporation.

PostScript, and Acrobat are registered trademarks of Adobe Systems, Incorporated.

All other trademarks are the properties of their respective owners.

Products: eDAQ BANK Elektronik Potentiostat Adaptor (EA169)

Document Number: UM-EA169-0707

Copyright © July 2005  
eDAQ Pty Ltd  
6 Doig Avenue  
Denistone East, NSW 2112  
Australia  
<http://www.eDAQ.com>  
email: [info@eDAQ.com](mailto:info@eDAQ.com)

All rights reserved. No part of this document may be reproduced by any means without the prior written permission of eDAQ Pty Ltd.



# Contents

## 1 Overview 1

- How to Use this Manual 2
- eDAQ Amps 2
- Checking the unit 3

## 2 The Bank Elektronik Adaptor 5

- The Front Panel 6
  - The Online Indicator 6
  - The Scan Indicator 6
- The Back Panel 7
  - E Out, I Out and E In Connectors 7
  - I<sup>2</sup>C Connectors 7
  - Potentiostat Interface Port 8
- Connecting the Adaptor 8
- First Use 10
- Adaptor Control Window 11

## 3 Techniques 17

- Introduction 18
- Linear Scan Techniques 18
  - Fast Cyclic Voltammetry 19
- Chronoamperometry with Chart 21
  - On Windows computers 21
  - On Macintosh 22
  - Analysis of Chronoamperometry 25
- Chronoamperometry with Scope 28

Chronocoulometry 31

## A Troubleshooting 33

## B Specifications 39

Index 41

License & Warranty 43



# 1

## CHAPTER ONE

# Overview

The eDAQ BANK ETeKtronik Potentiostat Adaptor is designed to work with any e-corder data acquisition system and a BANK Potentiostat, model:

- LPG03
- LPG03\_2A
- POS2
- POS2\_2A
- PGS95
- HP96\_10A
- HP400\_10A
- HP96\_20

Direct software control over many of the potentiostat front panel functions is provided. The eDAQ Chart, Scope, and EChem software all can make use of the Adaptor to provide a variety of electrochemical techniques. Please read the eDAQ *Potentiostats Manual* and the *EChem Software Manual* for an outline of the experimental settings for these techniques.

Please read your BANK Potentiostat manual to familiarise yourself with its front panel controls. Some BANK Potentiostats generate large voltage/current combinations which can be dangerous to the user!

# How to Use this Manual

This manual describes how to set up and begin using your Adaptor ([Chapter 3](#)).

The appendices provide technical and troubleshooting information.

See the *EChem Software Manual* for a description of the use of these potentiostats with the optional EChem software.

## eDAQ Amps

The Adaptor is one of a family of preamplifiers known as eDAQ Amps which are designed to be operated under full software control and are automatically recognised by Chart, Scope or EChem software which control their gain range, signal filtering, and other settings.

The eDAQ Amp family includes the:

- Potentiostat (model EA161). Single channel, three electrode potentiostat/galvanostat with gain ranges of 20 nA to 100 mA;
- Picostat (model EA162). Single channel, three electrode, high sensitivity potentiostat with gain ranges of 10 pA to 100 nA; and
- QuadStat (model EA164). Four channel, three electrode potentiostat with gain ranges of 2 nA to 10 mA with current signal offset.
- Quad pH Amp (model EA168), suitable for connection of up to four pH, ion selective, and potentiometric (ORP) electrodes. Each channel is electrically isolated.
- Quad Tc Amp (model EA150), suitable for connection of up to four thermocouples. Each channel is electrically isolated.
- C<sup>4</sup>D Amp (model EA120). Capacitively coupled contactless conductivity detector for capillary, and microfluidic channel, electrophoresis.
- GP Amp (EA142), suitable for high output sensors requiring a high impedance DC Wheatstone bridge. Also provides DC excitation.

See our web site at [www.eDAQ.com](http://www.eDAQ.com) for more information.

## Checking the unit

Before you begin working with the Adaptor please check that:

- all items described in the packing list are included; and that
- there are no signs of damage that may have occurred during transit.

Contact your eDAQ distributor if you encounter a problem.

You should also become familiar with the basic features of your e-corder system, which are discussed in the *e-corder Manual* which will be installed as a pdf file on your computer when you install the software.





# 2

## CHAPTER TWO

# The Bank Elektronik Adaptor

**▲ WARNING!**

*Some models of BANK potentiostat are capable of producing large currents and high voltages. Please read the BANK potentiostat manual to familiarise yourself with any possible hazard and correct operating procedure.*

This chapter describes how to connect and use your Bank Elektronik Potentiostat Adaptor.

**IMPORTANT:** Always make sure that the e-corder is turned off before you connect or disconnect the Potentiostat Adaptor unit. Failure to do this may result in damage to the e-corder and/or the Adaptor unit.

# The Front Panel

The front panel of the Adaptor is shown in [Figure 2-1](#)

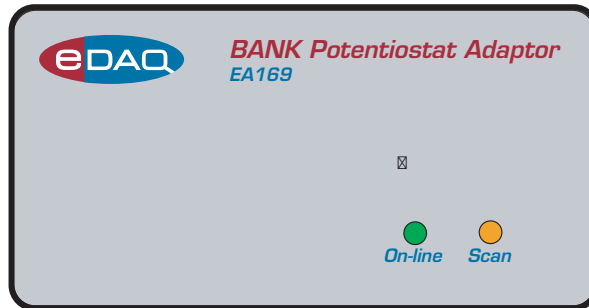
## The Online Indicator

When lit, the green Online Indicator, [Figure 2-1](#), shows that the software (such as EChem, Chart or Scope) has located and initialised the Interface. If the light does not go on when the software is run, check that the Adaptor is properly connected. If there is still a problem, please refer to [Appendix A, page 33](#).

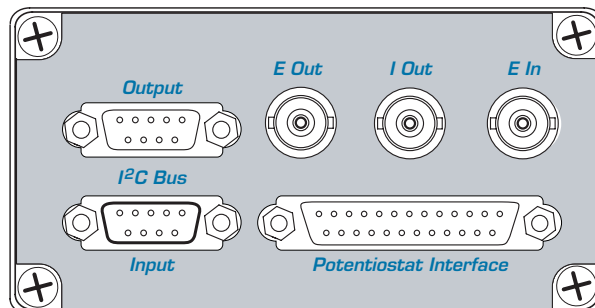
## The Scan Indicator

When lit, the yellow Scan indicator, [Figure 2-1](#) shows when the sweep generator in the Bank Elektronik potentiostat is active. Note that not all models of Bank potentiostat contain a sweep generator.

**Figure 2-1**  
The Bank Elektronik Potentiostat Adaptor, front panel.



**Figure 2-2**  
The Bank Elektronik Potentiostat Adaptor, back panel.



# The Back Panel

The back panel of the Adaptor is shown in [Figure 2-2](#).

## E Out, I Out and E In Connectors

The Adaptor back panel has three BNC connectors labelled E Out, I Out, and E In.

The Adaptor provides two signals from the potentiostat: the current signal (I Out) indicating the current flow between the working and auxiliary/counter electrodes, and the potential signal (E Out) indicating the potential difference between the working and reference electrodes. For most situations I Out is connected to e-corder Input 1, and E Out to e-corder Input 2, [Table 2-1](#). The description of operation in this manual assumes you have followed this convention. However, when you are using Chart software and recording data from various sources on more than just two channels, you may want to connect the Adaptor to other e-corder input channels.

The Adaptor E In is connected to the Output of the e-corder, usually Output + is used. To reverse the polarity of the Adaptor and potentiostat use e-corder Output -.

## I<sup>2</sup>C Connectors

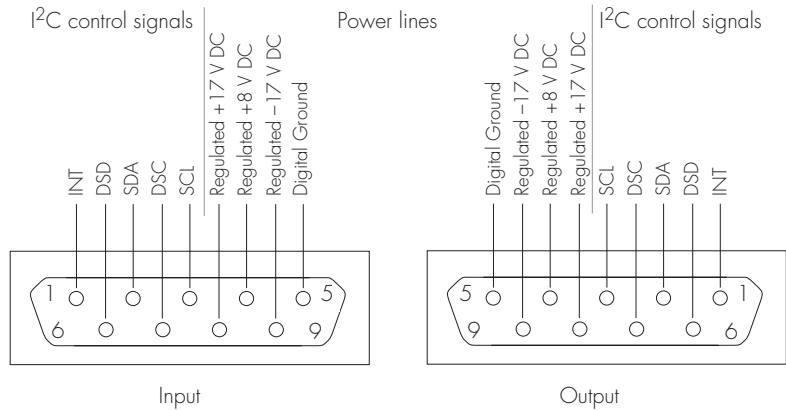
The Adaptor back panel, [Figure 2-2](#), has two DB-9 pin 'I<sup>2</sup>C bus' connectors labelled Input and Output. The Input connector provides power to the Adaptor and carries the various control signals (for mode, gain range, and filter selection) to and from the e-corder. A cable is

**Table 2-1**  
Adaptor to e-corder connections, analog signals.

<i>Adaptor back panel</i>	<i>e-corder front panel</i>
I Out	Input 1
E Out	Input 2
E In	*Output +

\* Use Output - to reverse the polarity of the applied potential

**Figure 2-3**  
The pin assignments for the I<sup>2</sup>C DB-9 connectors.



provided for this purpose. The pin assignments are shown in [Figure 2-3](#).

The Output connector can be used for the attachment of a second Adaptor or for attachment of eDAQ Amps.

More information about the I<sup>2</sup>C connector can be found in your *e-corder Manual*.

## Potentiostat Interface Port

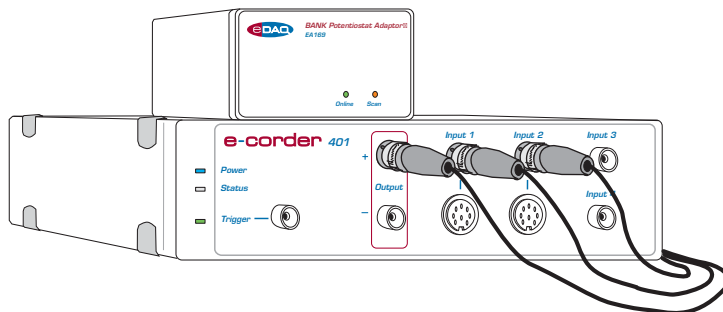
The back panel, [Figure 2-2](#), has a female DB25 pin Potentiostat Interface port. This provides digital (TTL) control signals to be sent to, and received from, the potentiostat.

## Connecting the Adaptor

First make sure that the e-corder and potentiostat are switched off. Use the cable supplied to connect the Potentiostat Interface Port to the back panel of the Bank Elektronik potentiostat. Then connect the back panels of the Adaptor and e-corder with the I<sup>2</sup>C cable. Next use the three BNC cables to connect the back panel of the Adaptor to the front panel of the e-corder as shown in [Table 2-1](#).

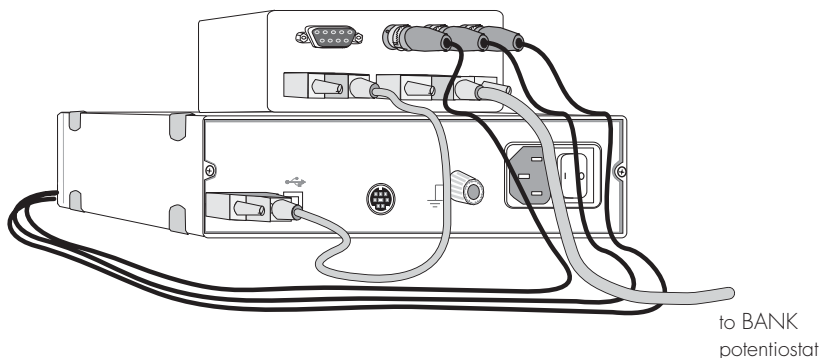
**Figure 2-4**

The Adaptor shown connected to an e-corder, front view, as described in Table 2-2.



**Figure 2-5**

The Adaptor shown connected to an e-corder, rear view, as described in Table 2-2.



With these connections, when you use the software to set a more positive voltage, a more oxidising potential will be applied at the working electrode. Such an arrangement is shown in Figure 2-4 and Figure 2-6.

To operate the potentiostat with reverse polarity connect Adaptor E In to e-corder Output -. With these connections, when you use the software to set a more positive voltage, a more reducing potential will be applied at the working electrode.

Check that all connectors are firmly attached. Loose connectors can cause erratic behaviour, or may cause the potentiostat to fail to work.

The Adaptor uses two e-corder input channels during normal operation for the current (I Out) and potential (E Out) signals. The remainder of this chapter assumes that you have connected the current signal to e-corder Input Channel 1 and the potential signal to e-corder Input Channel 2. (It is possible when using Chart or Scope software to

connect the Potentiostat to other e-corder input channels, in which case the description that follows would change accordingly).

When using EChem software, Channel 1 is always set to be the current signal (the I channel), and Channel 2 is automatically set to be the potential signal (the E channel). Thus when using EChem software you *must* always connect the current signal (I Out) to e-corder Input Channel 1, and the potential signal (E Out) to e-corder Input Channel 2. Channel 2 normally displays the applied potential, and its settings are controlled using the standard Input Amplifier dialog box, described in the *Chart Software Manual* and *Scope Software Manual* on the eDAQ Installer CD.

## First Use

After you have installed the software, connected the e-corder and computer as described with the booklet supplied with the *e-corder* system, and connected the Adaptor and potentiostat as described above, you are ready to begin.

Switch the range knob on the potentiostat to REM (remote). Turn the potentiostat on and allow it several minutes to warm up.

When the e-corder is turned on, and Chart (or Scope or EChem) software started, the Adaptor Online indicator (green) should light, [Figure 2-1, page 6](#).

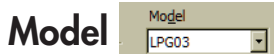
From the Channel 1 Function pop-up menu, select the 'Bank Elektronik' command, which opens the Control window, [Figure 2-6 \(Windows\)](#).

This window allows you to preview the current signal without actually recording the signal to your computer's hard disk. (If the menu says 'Input Amplifier' instead of 'Bank Elektronik' or 'BANK' then the software has not recognised the Adaptor. Exit the software, check all your connections and try again).

By default, the control window opens with the potentiostat in Standby mode, that is with the counter electrode isolated so that no current will flow through your electrodes. To connect to the Potentiostat lead wires you must select Real mode. When you click Cancel or OK the Potentiostat will revert to Standby mode until recording is started.

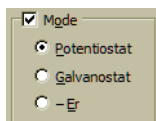
# Adaptor Control Window

With Chart software, the Adaptor Control window is accessed from the Bank Elektronik command in the Channel Function pop-up menu. [Figure 2-6](#) shows the control window on a Windows computer. This window controls the various current ranges, filters, and operating modes of the Potentiostat.



The correct potentiostat model, [page 1](#), must be selected from the Model menu. Some models may not offer all the controls described below.

## Modes of Operation



While the Mode box is unticked,  Mode, the potentiostat is in a resting state equivalent to the 'O' position of the front panel Operation knob. While in this state gain ranges cannot be selected.

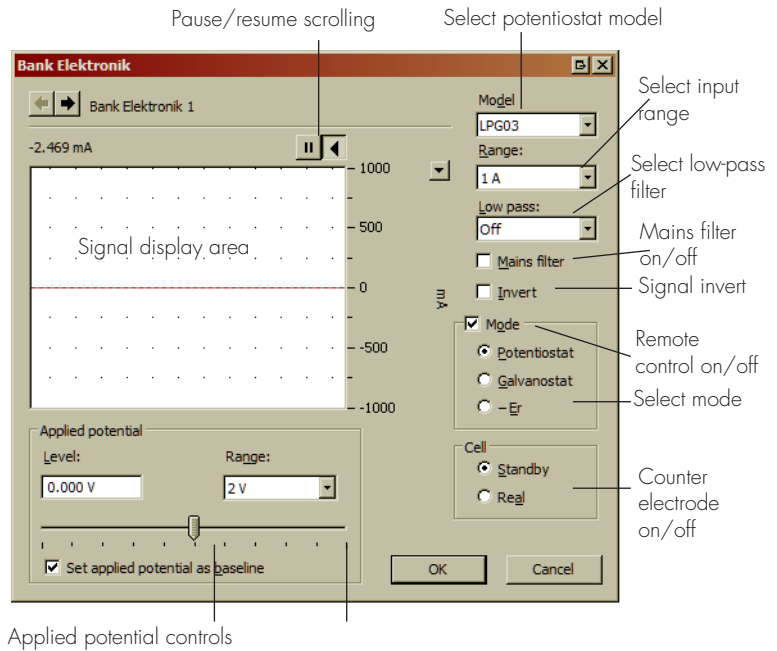
When the box is ticked,  Mode, the Adaptor becomes fully active and the potentiostat can now be operated in several different modes by selecting the appropriate radio button:

- **Potentiostat** (Chart, Scope, and EChem software). This is equivalent to setting "Operation P" on the potentiostat front panel. The current signal is provided at I Out, and the potential signal is provided at E Out on the Adaptor back panel, [Figure 2-2](#).
- **Galvanostat** (Chart and Scope software only). This is equivalent to setting "Operation G" on the potentiostat front panel. The current signal is provided at I Out, and the potential signal is provided at E Out on the Adaptor back panel, [Figure 2-2](#).
- **-Er**, high impedance voltmeter, (Chart and Scope software only). This is equivalent to setting "Operation -Er" on the potentiostat front panel. The high impedance potential signal is supplied at E Out, on the Adaptor back panel, [Figure 2-2](#);

Please make sure the electrode lead wires are connected appropriately before operating in any of these modes. In particular, incorrect placement of leads may damage the reference electrode.

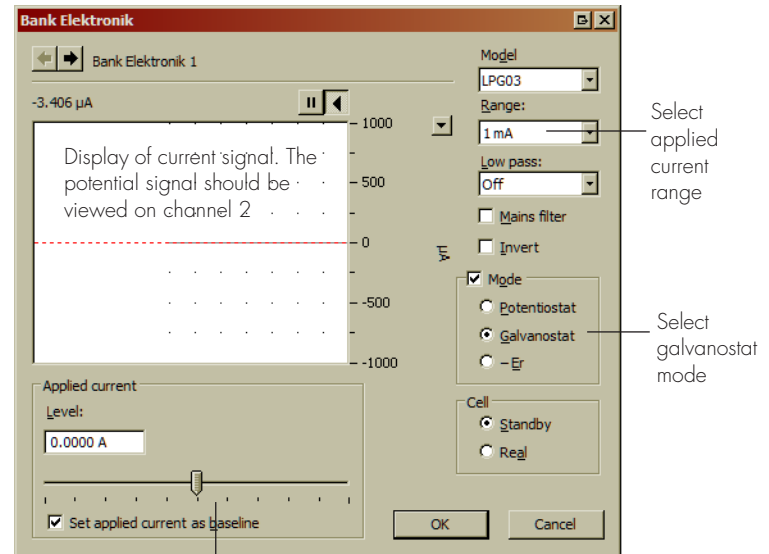
**Figure 2-6**  
 Potentiostat Adaptor controls with Chart software. Potentiostat mode.

Select Bank Elektronik in the Channel pop-up menu



Applied potential controls

**Figure 2-7**  
 Potentiostat Adaptor controls with Chart software. Galvanostat mode.



Applied current control (when in galvanostat mode)




Note that not all modes may be available on all models of BANK Potentiostat. Also Galvanostat and -Er modes are not available when using EChem software (which is designed for voltammetric experiments requiring potentiostat mode only)

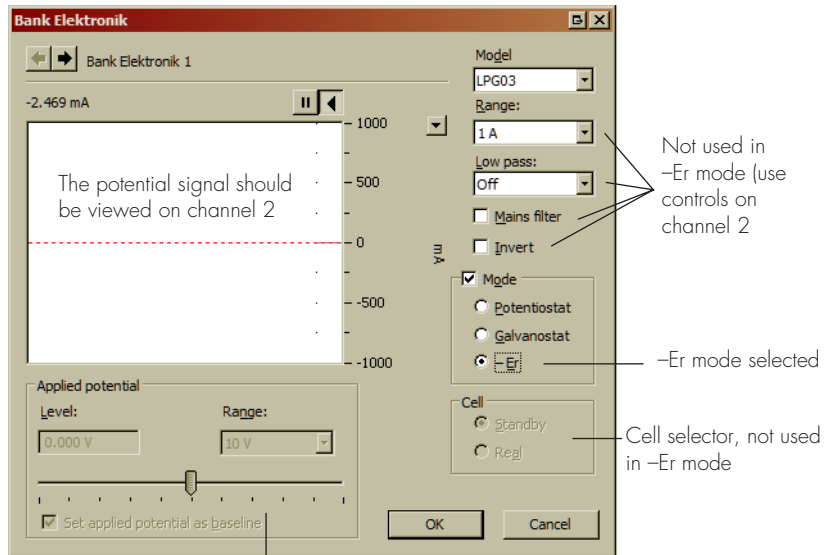
## Signal Display

The current signal is previewed in the scrolling display area. Note that the signal is not being recorded to hard disk at this stage, and that when the window is closed the signal trace will be lost.

By using Real cell, the counter electrode is activated and you can investigate the effect of the [Applied Potential](#), [page 15](#), on the current signal.

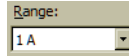
You can stop/start the signal scrolling by clicking the Pause/Resume button .

You can shift or stretch the vertical Amplitude axis to make the best use of the available display area.



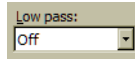
Applied potential control, not used -Er mode

## Setting the Range



Use the Range pop-up menu to select the input current range (channel sensitivity). The resolution within each range is 16 bits or 0.0015%. You should set the range so that it is larger than the biggest current that you expect to encounter during your experiment. If, during the experiment the current signal exceeds the range, then the signal will go off scale and be lost.

## Filtering



The e-corder provides filter settings from 1 Hz to 2000 Hz (Chart software). When filtering is Off the bandwidth of the e-corder is about 20 kHz. Always examine the signal without filtering before applying a low pass filter. Excessive noise can indicate a problem with shielding of the experiment, inadequate earthing of the power socket, or a problem in locating the experiment to close to nearby 'noisy' equipment.

As a general rule a 10 Hz filter setting is highly effective for the removal of mains hum (50 or 60 Hz interference) and should be employed whenever possible. A 10 Hz filter should not be used for pulsed experiments where the pulses are shorter than 100 ms, or for voltammetric experiments where rapid scan rates (greater than about 100 mV/s) are used, because it will distort the signal.

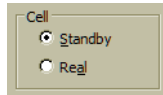
With Chart software, there is an additional Mains Filter checkbox,  Mains filter. If this is ticked, then the e-corder will apply a mains filtering algorithm to the incoming signal which removes repetitive signals occurring at 50 or 60 Hz which are typical of mains interference. Lower and higher frequency components in the signal will be unaffected. Note that the mains filter is not a notch filter, and that it can remove a 50 or 60 Hz interference even if it is not a pure sinusoidal function. However, the mains filter does take a couple of seconds to 'learn' the pattern of the interference so that you will need to record for longer than this for it to have full effect. The mains filter can be employed even for experiments in which there are sudden potential jumps.

**Figure 2-8**  
Accessing the  
Potentiostat controls with  
Scope software.

## Inverting the Signal Invert

The Invert checkbox, allows you to reassign the direction (up or down) of an anodic (or cathodic) current. Please note that this affects the display of the signal only — it does not reverse the direction of actual current flow at the electrodes!

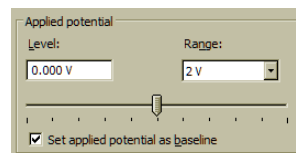
## Cell Control



When in Potentiostat or Galvanostat mode you can select:

- **Standby:** the counter electrode is unconnected (the 'CE ON' light on the front panel of the potentiostat will be unlit). However, the counter electrode will be connected when Potentiostat Control window is closed and the Start button is clicked to begin the experiment. Note that the Applied Potential (or Applied Current) controls are disabled in this mode.
- **Real:** the counter electrode is connected (the 'CE ON' light on the front panel of the potentiostat will light). Note that the Applied Potential (or Applied Current) controls are enabled in this mode, [Figure 2–6, page 12](#) and [Figure 2–7](#), and can be used to set the potential (or current) applied to the electrodes while the Potentiostat control window is open. When you close the control dialog (using EChem or Scope software) the potentiostat will revert to Standby mode until the Start button is clicked to begin a scan. If you are using Chart software, the potentiostat will remain in Real mode (CE ON) when the dialog is closed — so that when you start and stop recording data the electrodes will remain active. This allows periodic recording of the signal from, for example, amperometric biosensors without disturbing the environment around the electrodes.

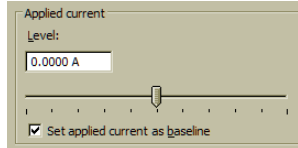
## Applied Potential



The Applied Potential controls are enabled when Real cell is selected in Potentiostat mode. It allows you to adjust the voltage applied to the electrodes. The Range control limits the range over which the applied potential can be set. Smaller ranges offer finer control with the slider bar.

Check the  Set applied potential as baseline box to remember the value of the applied potential when the control window, [Figure 2–6, page 12](#), is closed. (The value is transferred to the Stimulator baseline control, [Figure 3–5, page 23](#)).

## Applied Current



The Applied Current controls are active when when Real cell is selected in Galvanostat mode (replacing the Applied Potential controls). The maximum applied current is determined by the setting of the Range control.

Check the  Set applied current as baseline box to remember the value of the applied current when the control window, is closed. (The value is transferred to the Stimulator baseline control).

# 3

## CHAPTER THREE

# Techniques

The Chart and Scope software supplied with your e-corder can be used to perform many different electrochemical techniques. This chapter provides an overview of these techniques, but you will need to also refer to the *Chart Software Manual* and *Scope Software Manual* (which are installed on the computer hard disk, with the software, or can be found on the eDAQ Installer CD).

Additional experiments such as current-sampled staircase linear sweep, differential pulse, normal pulse, square wave, staircase cyclic voltammetry, and pulse amperometry can be performed with the optional EChem software — see the *EChem Software Manual*, or contact eDAQ for more details.

# Introduction

A potentiostat applies a potential difference across a pair of working and reference electrodes whilst monitoring the current flow between the working and auxiliary electrodes.

This potential difference is determined by a 'command voltage' which is sent from the e-corder output to the 'E In' connector of the Adaptor, [Figure 2-7](#). The potentiostat can also generate a constant command voltage internally, and normally this should be set to zero (or it will be added to the external command voltage).

Chart and Scope software control the e-corder output via the 'Stimulator' controls in their Setup menus. For a full description of these controls, and the waveforms that can be produced, you should consult the *Chart Software Manual* and *Scope Software Manual*. You can use your e-corder and potentiostat with Chart and Scope software to perform the following experiments:

- Linear Scan techniques, [page 18](#) — use Scope software with the Potentiostat to provide a potential ramp (up to 500 V/s) and to subtract charging current contributions.
- Chronoamperometry [page 21](#) & [page 28](#), Amperometry, and Constant Potential Electrolysis, — monitor the current signal at fixed potentials
- Chronocoulometry [page 31](#) — monitor and integrate the current signal at a fixed potentials
- Chronopotentiometry, and Constant Current Electrolysis — monitor the potential signal when in Galvanostat mode, [page 11](#), to maintain a constant current at the working electrode.
- high impedance voltmeter, with some BANK potentiostat models using '-Er' mode, [page 11](#).

## Linear Scan Techniques

Linear sweep or cyclic voltammetry are usually best performed with EChem software. However, Scope also incorporates Stimulator... and Output Voltage... commands which can be used to control the analog

output of the e-corder to create a waveform suitable for these techniques (albeit less conveniently than with the EChem software). With Scope software up to 2560 points can be collected on one channel at a rate of up to up to 100 kHz on both channels.

## Fast Cyclic Voltammetry

Scope software has a number of features that can perform Fast Cyclic Voltammetry (FCV). These are accessed via the Stimulator... and Output Voltage... commands in the Setup menu, [Figure 3-1](#).

The base potential is set by using the Stimulator Constant Output dialog box while the waveform is generated using the Stimulator Up & Down, [Figure 3-2](#), or Triangle, [Figure 3-3](#), waveforms. See the *Scope Software Manual* for further details.

In [Figure 3-2](#) the base potential has been set to  $-1.00$  V and a potential ramp has been set up to go from  $-1.00$  to  $+1.00$  V and back again over a period of 40 ms using the Up & Down waveform option. This corresponds to a scan rate of 50 V/s.

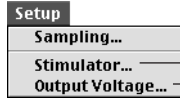
[Figure 3-3](#) shows an example of a positive and negative cyclic waveform. The base potential has been set to  $+0.20$  V and a potential ramp has been set up to go from  $+0.20$  to  $+0.80$  V back again to  $-0.40$  V and finally return to  $+0.20$  V, over a period of 20 ms using the Triangle waveform option. This corresponds to a scan rate of 120 V/s.

The Triangle waveform option always starts a scan in the centre of the potential limits, while the Up & Down waveform option starts a scan at one, or the other, of the potential limits.

Ideally, FCV requires a smooth analog ramp, and so it is desirable to make the steps in the applied waveform output as small as possible. The e-corder output is controlled by a DAC (digital-to-analog convertor) is the value of which is updated whenever a new data point acquired. Thus, by setting the data acquisition rate to be as fast as possible for a particular experiment, you will also ensure that the waveform has steps that are as small as possible.

When performing FCV, a large background charging current is often recorded. This can be many times larger than the signal you are

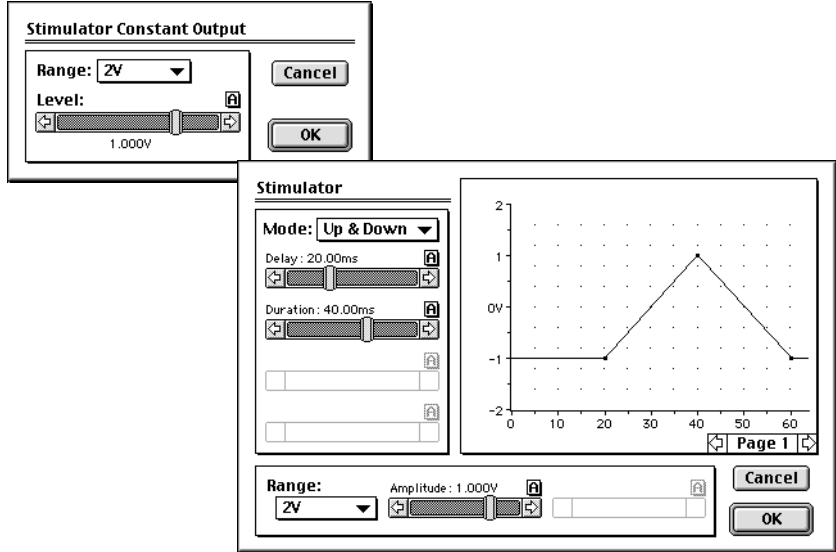
**Figure 3-1**  
Scope Setup menu.



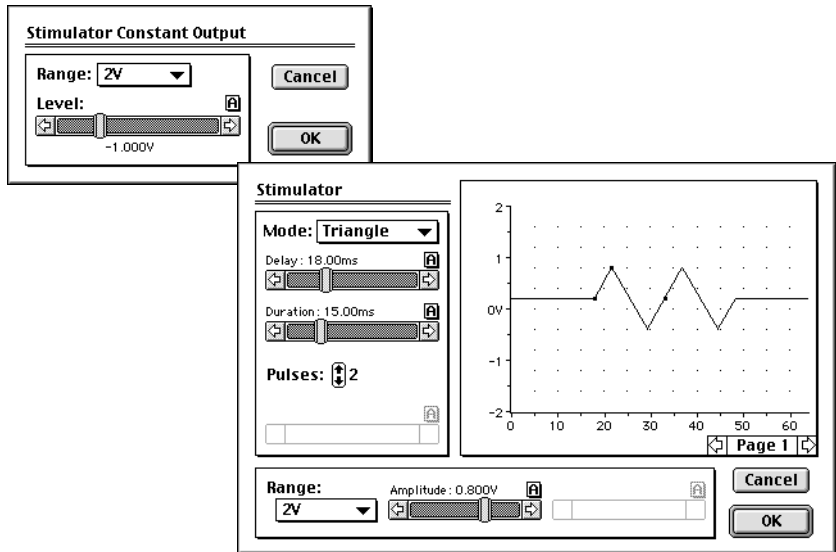
Use the Stimulator... command to access the Stimulator (applied potential waveform) controls

Use the Output Voltage... command to set the baseline potential

**Figure 3-2**  
Using the Scope Stimulator Up and Down command.



**Figure 3-3**  
Using the Scope Stimulator Triangle command.





looking for. Fortunately the charging current is usually reproducible between scans and can be subtracted from the final result:

1. first a background scan is obtained with a 'blank' solution (that is, a solution containing only the background electrolyte);
2. next a scan is performed of the solution in which the substrate is present; and finally
3. the Scope 'page' that contains the background scan is selected using the Display > Set Background command which will subtract this scan from all other pages in the file. Use the Display > Don't Subtract Background command to cancel subtraction, and Clear Background to cancel the subtraction and clear the background sweep.

The background scan must be run under the same conditions (sweep width and stimulator settings) as the substrate solution to be effective. See the *Scope Software Manual* for more information.

## Chronoamperometry with Chart

Chronoamperometric techniques require that a constant potential is maintained for a defined period while the current is monitored. If the current signal is integrated with respect to time then the total charge transferred at the electrode can be calculated ([Chronocoulometry, page 31](#)).

Single, double and even multi-step chronoamperometry, can be performed with Chart or Scope software, in a time frame from a millisecond to hours, or even days, if need be. For experiments involving sudden changes in potential you should use the full bandwidth of the potentiostat (that is do not use low-pass filters) or the response of the current signal may be modified by the low-pass filter time response characteristics.

## On Windows computers

Chart software can be used to set a constant voltage of up to  $\pm 10$  V (which is known as the command voltage) from the e-corder Output. This is sent to the potentiostat, via the 'E In' input cable, which then applies this potential across the reference and working electrodes. The

software controls are accessed through the Stimulator command in the Setup menu, [Figure 3–4](#).

To adjust the command voltage you will first need to select the range by adjusting the Output Range control in the Stimulator controls [Figure 3–5](#). The smaller the selected output range, the finer the control that you will have when you adjust the potential with the Baseline control. To monitor the current signal at a constant potential:

1. set the **Stimulator** to Off;
2. set the **Baseline** control to the desired voltage;
3. adjust the current input range to an appropriate value, [page 14](#);
4. set the speed of recording (that is the number of data points to be collected per second) to an appropriate value — you will usually require at least several hundred data points over the lifetime of your experiment. The *Chart Software Manual* has detailed descriptions on setting the recording speed; and finally
5. begin the experiment by clicking the Start button in the main Chart window.

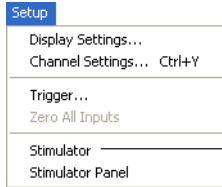
The Stimulator command can be used to alter the applied potential with a precision better than 1 ms. Pulses up to 30 s long may be created by this method. For further details on using the Stimulator in Chart refer to the *Chart Software Manual*. An example using the Chart Stimulator is shown in [Figure 3–6](#). Note that the pulse amplitudes are added to any value set by the Baseline control.

Chart macros can be used to keep the potential constant for a fixed period of time, or to drive the reaction backwards by first applying one potential and then subsequently applying a second potential to perform an oxidation/reduction cycle. Refer to the *Chart Software Manual* for more information.

## On Macintosh

Chart software can be used to output a constant potential (up to  $\pm 10$  V) which the potentiostat will apply at the working electrode. These controls are accessed through the Setup menu, [Figure 3–7](#).

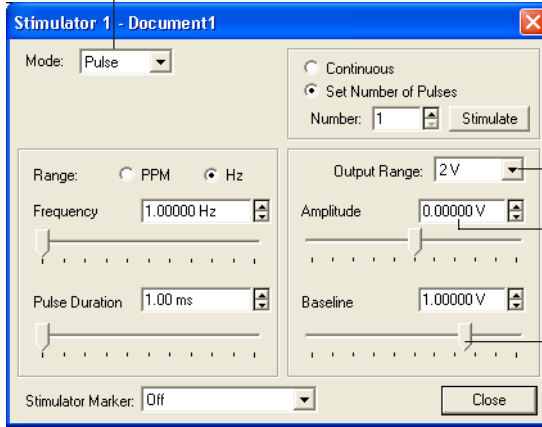
**Figure 3-4**  
Chart Setup menu  
(Windows).



Use the Stimulator command to access the Stimulator (applied potential waveform) controls, Figure 3-5 & Figure 3-6

**Figure 3-5**  
Chart Stimulator  
(waveform output)  
controls (Windows).

Select stimulator mode

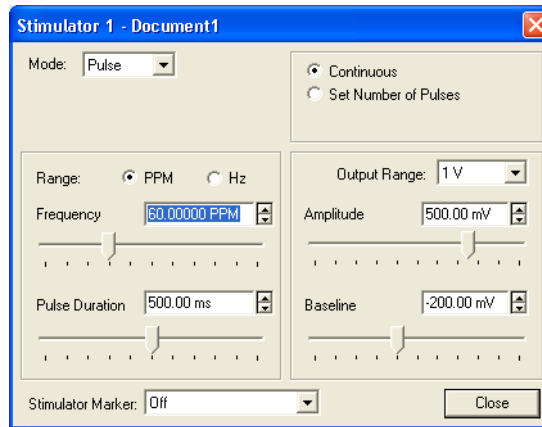


Select output range

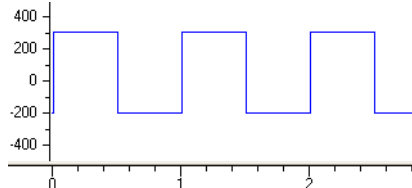
Enter exact values as text.  
Use zero amplitude for  
constant output

Baseline control can be used for constant  
output — drag slider  
controls to adjust  
value

**Figure 3-6**  
Stimulator controls for  
multiple step  
chronoamperometry  
(Windows).



Settings to produce a 500  
mV amplitude 1 Hz square  
wave, on a base potential  
of -200 mV



Potential waveform generated by  
the Stimulator settings above

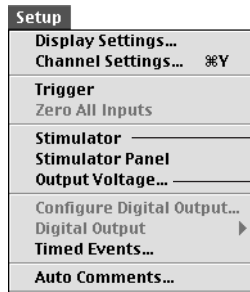
To adjust the potential you will first need to select the range by choosing the Output Voltage command in the Setup menu to activate the Stimulator Constant Output dialog box, [Figure 3–8](#). The smaller the selected output range, the finer the control that you will have when you adjust the potential.

The Timed Events (Setup menu) feature and macros can be used to keep the potential constant for a fixed period of time, or to drive the reaction backwards by applying an opposite potential to perform an oxidation/reduction cycle.

The Chart software Stimulator command (in the Setup menu) can also be used to set up pulses, stepped pulses, and staircase ramp waveforms for the potentiostat. See the *Chart Software Manual* for more details.

You can also use the Timed Events feature (Setup menu) to adjust the current at predetermined time intervals after the start of recording. For example, the settings shown in [Figure 3–9](#) will maintain a potential of

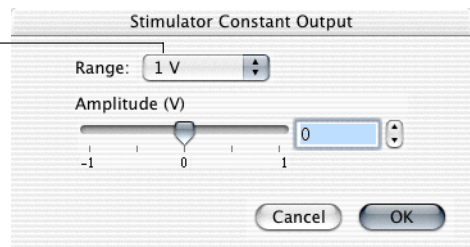
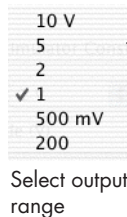
**Figure 3–7**  
Chart Setup menu  
(Macintosh).



Use the Stimulator command to access the Stimulator (applied potential waveform) controls

Use the Output Voltage... command to set the baseline potential, [Figure 3–8](#)

**Figure 3–8**  
Chart Stimulator  
Constant Output voltage  
controls (Macintosh).



1.25 V for 30 minutes after which the potential will be decreased to 0.50 V in order to reverse the reaction, resulting in a Double Step Chronoamperometric experiment. Any number of steps at any potential could be set up with this feature. However, Timed Events can be slightly delayed (by up to 0.1 s) depending on the model of computer and what else is happening in the computer operating system. For accurate timing it is better to use the Chart Stimulator which is fully under the control of the internal e-corder clock.

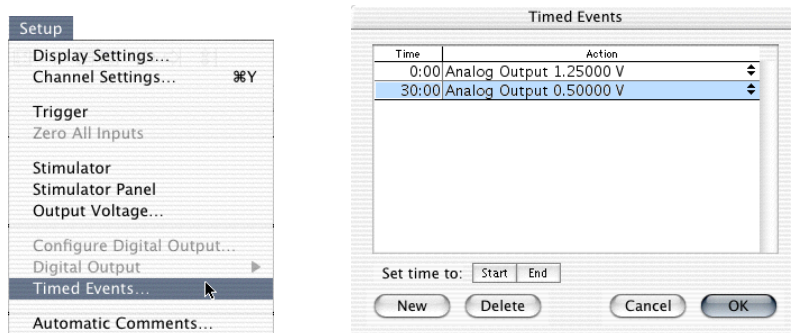
Using the Stimulator command (Setup menu) it is possible to send and monitor pulses with a precision better than 1 ms, [Figure 3–10](#). Pulses up to about 30 s may be created by this method. For further details on using the Stimulator in Chart refer to the *Chart Software Manual*.

## Analysis of Chronoamperometry

Chronoamperograms obtained from a planar disk electrode should exhibit a current/time relationship governed by the Cottrell equation, (see Appendix D for definition of terms)

$$i = \frac{nFAC\sqrt{D}}{1000\sqrt{\pi t}}$$

**Figure 3–9**  
Adjusting potential with the Chart Timed Events feature. (Macintosh only)

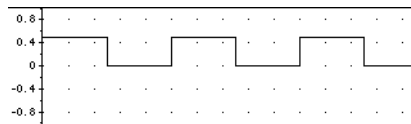
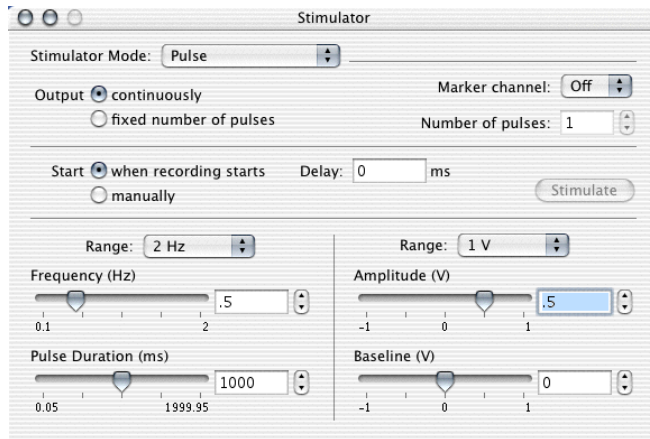


Access Timed Events from the Setup menu

Changing the potential after a period of 30 s

**Figure 3–10**

Simulator controls for multiple step chronoamperometry (Macintosh).



Stimulator settings to produce a 500 mV high, 0.5 Hz square wave

It is usually easier to first simplify the Cottrell equation thus:

$$i = \frac{a}{\sqrt{t}}$$

where  $a$  is treated as an empirical constant to be determined.

You can use the Chart's Arithmetic channel calculation (see the *Chart Software Manual*) to create 'data' on an unused channel (usually Channel 3) according to the equation:

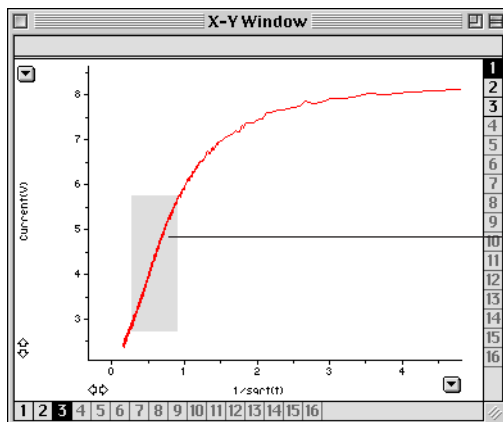
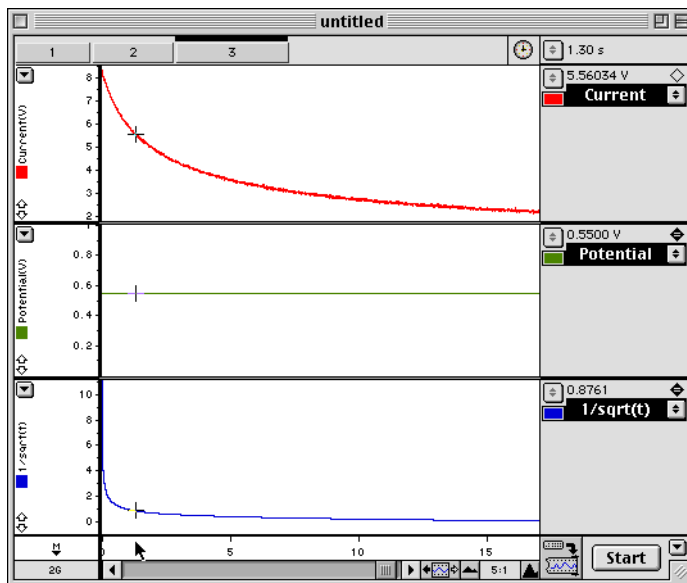
$$\text{Channel 3} = 1/\text{Sqrt}(\text{SampleTime})$$

You can now use the X–Y window to plot the current signal (usually on Channel 1) versus Channel 3, and a near straight line should be obtained with a slope of  $a$ , [Figure 3–11](#).

With the Curve Fit extension installed, you can use the Curve Fitting command (Windows menu) to fit your current signal data with an idealised curve. The Curve Fit analysis extension and accompanying documentation can be downloaded free from the eDAQ web site, [www.eDAQ.com](http://www.eDAQ.com). The extension file should be put into the Chart Extensions folder, inside the Chart folder on your hard disk.

**Figure 3–11**

Typical chronoamperometric data, analyzed using the Arithmetic channel calculation and the X–Y window.



Typical experiment with  $1/\sqrt{t}$  data calculated on Channel 3 using the Arithmetic channel

Region of ideal linear response

Cottrell graph of current versus  $1/\sqrt{t}$  prepared in the XY Window. Deviations from linearity typically occur at very small and very large  $t$  values.

It is unlikely that the current signal will decay to exactly zero (due to convection currents in the vessel causing non-ideal diffusion) so a small non-zero constant,  $b$ , is added. Also the equation is very sensitive to the exact instant when  $t$  is zero which is a problem if you have selected data very close to zero time (in the Curve Fitting window, it is usually best to use the 't=0 at start of block' option as shown in Figure 3–12). In this case, to get a well behaved equation when the data selected start at very close to zero time, initially add a small time correction,  $c$ . This allows for the errors in the estimation of time zero, and the fact that the Potentiostat has a finite bandwidth (and so the current signal will

take a small amount of time to register a true value after a sudden change in potential).

Thus a better form of the equation to be fitted is:

$$i = \frac{a}{\sqrt{t+c}} + b$$

This must be entered as a New Equation in the Curve Fit extension the very first time you use it, [Figure 3-12](#).

Because of the discontinuity that occurs when the denominator is zero, first try fixing  $c$  to a small positive value, and iterating for better values of  $a$  and  $b$ . Some trial and error will be involved while you make guesses that are suitable for the initial values to iterate. After the better estimates for  $a$  and  $b$  have been determined, and iterated,  $c$  can be unlocked so that it refines during the next iteration, [Figure 3-12](#).

Your data selection should not include points very close to zero time (the Potentiostat will require some time to settle), nor points taken at very long times when convection aided diffusion will produce non-ideal behaviour. The exact time period that is suitable will depend on the size of the electrode, and some trial and error to determine the time interval to give best results will probably be required.

After the file is saved, the Curve Fit Cottrell equation will be available whenever you open Chart data file on the same computer or even when you transfer the data file to another computer on which Chart software and the Curve Fit extension are installed.

## Chronoamperometry with Scope

For chronoamperometric measurements, where you need to overlay the results of successive experiments, Scope will generally be the program of choice.

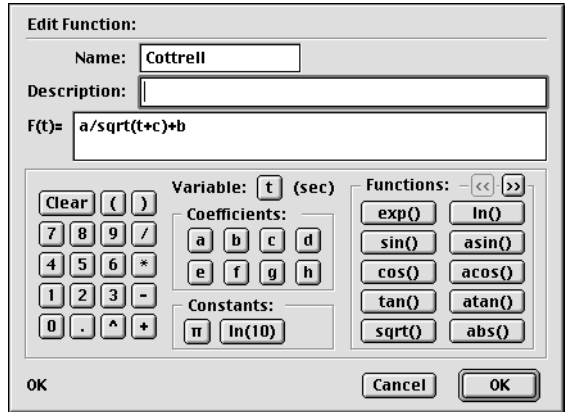
The total length of the experiment is chosen in the Time Base panel. Up to 2560 data points can be collected in a period of up to 128 s long. You should normally use the full bandwidth of the potentiostat (that is, do not use any low-pass filters) or the response of the current signal may be dominated by the low-pass filter time response characteristics.



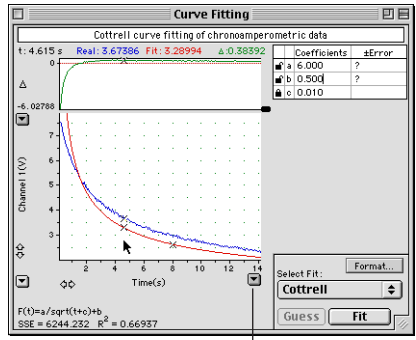
**Figure 3-12**

Fitting a Cottrell equation to data with the Curve Fit extension.

At the time of writing the Curve Fit extension is only available for Chart for Macintosh.



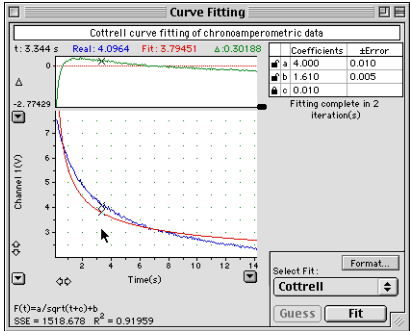
Define the Cottrell relationship as a new equation



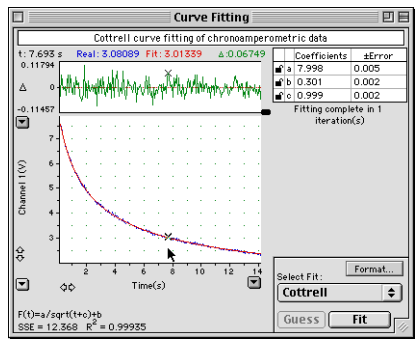
First lock in a value for c, then try guesses for a and b until an approximate fit is obtained

Usually use 't=0 at start of block'

Set Scale...  
 Auto Scale  
 Full Range  
 t=0 at start of selection  
 t=0 at start of block  
 Channel 1  
 Channel 2



Iterate by clicking the Fit button, refining a and b only. If the initial guesses are close enough, then a curve of better fit with new values for a and b will be obtained.



Unlock c, and iterate again. A curve of best fit should be obtained.

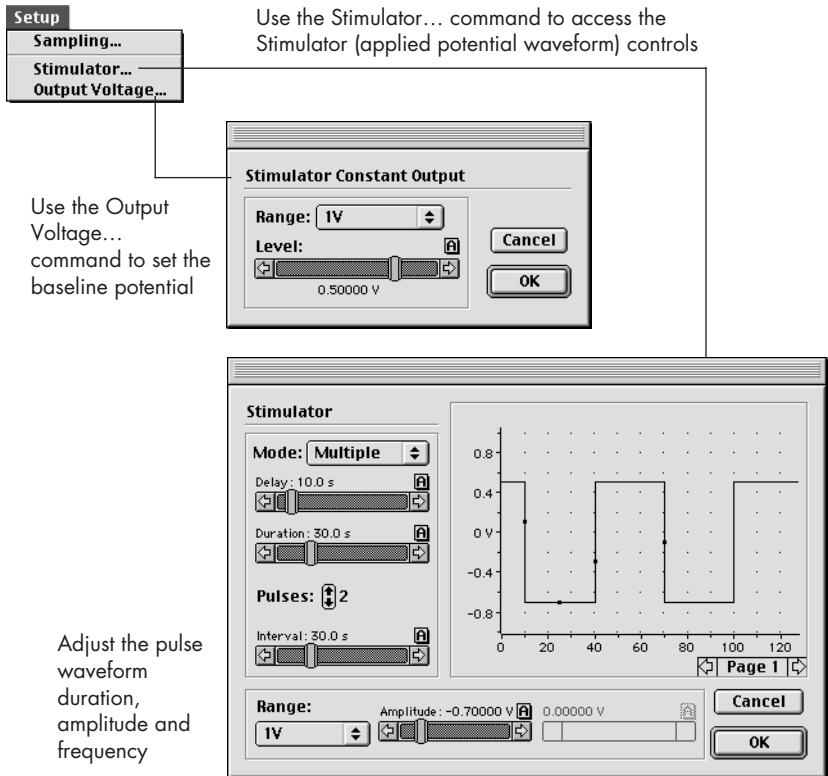
The base potential is adjusted with the Output Voltage... command (Setup menu) shown in Figure 3-13.

A potential that will cause the reaction to proceed (and the period for which it will be applied) is set using the Stimulator command. In the example shown in Figure 3-13, after a period of 10 s at the base potential of +0.5 V, two 30 s pulses of -0.70 V are to be applied each followed by a 30 s return to the base potential.

The experiment is usually first done on a blank solution containing only electrolyte, followed by a sample solution containing the substrate. The data is collected on separate pages in Scope and the 'blank' data subtracted with the Set Background command. You can then copy and paste the scan to a spreadsheet so that the differences can be plotted against  $1/\sqrt{t}$  in a Cottrell graph.

For further details refer to the *Scope Software Manual*.

**Figure 3-13**  
Scope controls for multiple step chronoamperometry.



# Chronocoulometry

Chart and Scope software have the ability to integrate an incoming signal both online (that is, in real time as you are collecting data), or offline (that is after the experiment is completed). Since the total charge transferred (the total number of coulombs, or electrons, transferred) is equal to the integrated current, the settings used for chronoamperometry, [page 21](#), can also be used for chronocoulometry.

With Chart software it also necessary to set up an unused channel (usually Channel 3) to be the integral of the current signal channel (usually Channel 1).

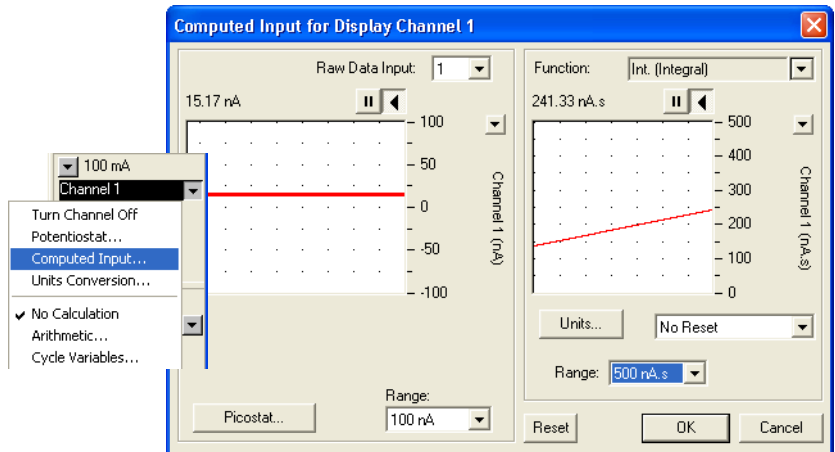
With an online function you need to actually be recording data to obtain the integral. To configure Channel 3 you need to choose the Computed Input command from the Channel Function pop-up menu, which opens the Computed Input dialog box ([Figure 3-14](#) on a Windows computer, and [Figure 3-15](#) on a Macintosh). For more information refer to the Computed Input section in the *Chart Software Manual*.

Post-acquisition integration of a signal is also possible with the Chart Integral channel calculation, which is accessed via the Integral command in the Channel Function pop-up menu — further details are in the *Chart Software Manual*. This is particularly useful when you want to recalculate the integral from the original current signal. The real time methods will give good results only if the appropriate sensitivity range has been pre-selected — and it is not always possible to determine this beforehand. A good strategy is often to use the real time integral function to get an idea of what is happening during an experiment and the use post-acquisition integration to prepare data for a final report.

With Scope software the current signal can be integrated by using the Computed Functions... command, [Figure 3-16](#). The Integrate item is chosen from the Function menu. This is actually a post-acquisition function — you can always cancel it afterwards to look at the underlying current data. Refer to the *Scope Software Manual* for more information.

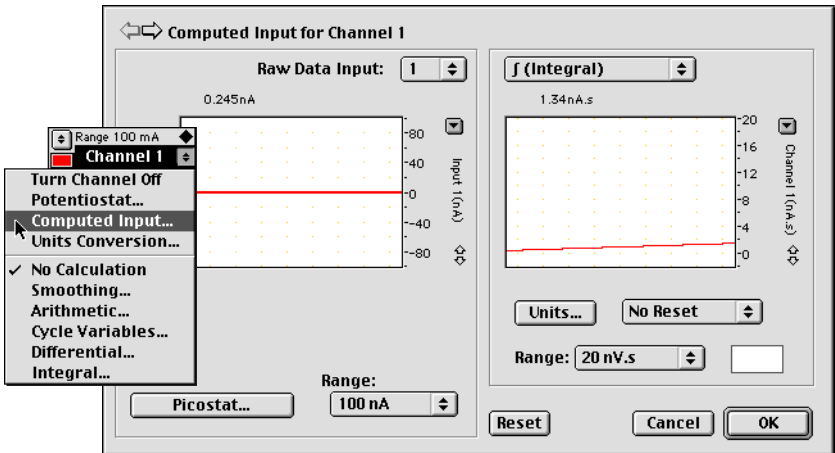
**Figure 3-14**

Online integration of the current signal using Chart Computed Input integration (Windows).



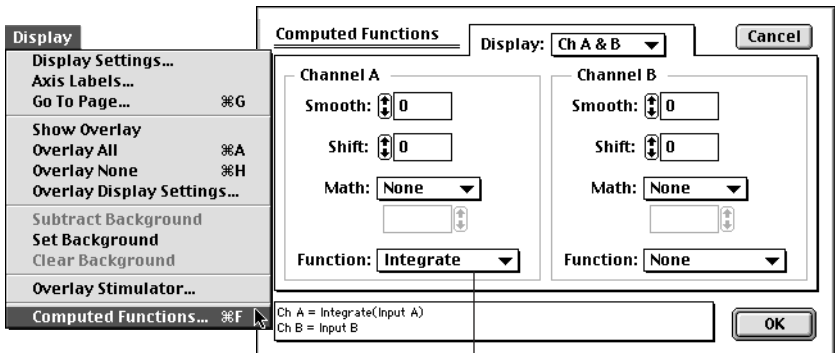
**Figure 3-15**

Online integration of the current signal using Chart Computed Input integration (Macintosh).



**Figure 3-16**

Integration of the current signal using Scope Computed Functions.



Select Integrate in the Function menu

# A

## A P P E N D I X A

# Troubleshooting

This appendix describes some problems that may arise. There is also help in your *e-corder Manual* and *Software Manuals*, located on the *eDAQ Software Installer CD*.

In many cases, a problem can be fixed by turning the e-corder and computer off, checking that all cables are firmly attached, and starting again. If you cannot correct the problem, then please contact your eDAQ representative.

### ***The Adaptor On-line indicator fails to light when the software is opened.***

- Check that the e-corder is connected to the mains power and that it is turned on.
- Check that Adaptor is properly connected to the e-corder and that the cables are not loose. Especially check that the 'E in', 'I Out' and 'E Out' cables are correctly connected, see [page 7](#).
- If possible, change cables and try again. If a second cable works and you find that the first cable is faulty, you should contact eDAQ for a replacement.
- If you are using EChem software, check that the 'E In' cable is connected to the e-corder output and the 'I Out' and 'E Out' cables are connected to e-corder Input 1 and 2, see [page 7](#).
- Try using the Adaptor with another e-corder (if available). If the Online indicator fails to light on the second e-corder, the Adaptor or its cables may be faulty — contact eDAQ. If it works correctly on a

second e-corder then the first e-corder may be faulty — contact eDAQ.

***The 'Bank Elektronik' controls cannot be accessed in Chart, Scope or EChem software (that is only the standard 'Input Amplifier' dialog box of the e-corder can be accessed)***

- Check that the Adaptor Online indicator is illuminated after the software has been opened. If not, then proceed as in the previous problem.
- Check that you have connected the 'E In', 'I Out' and 'E Out' cables correctly to the appropriate e-corder connectors.

***The Potentiostat Overload indicator is on (a potential overload has occurred).***

- Check that the electrode cables are firmly attached to the electrodes.
- Check that the surfaces of the electrodes have not become fouled.

***The current signal goes off scale.***

- Increase the full scale current range setting.
- Make sure each electrode is connected to the correct potentiostat lead.
- Make sure the electrodes and their lead wire contacts are not touching each other.
- Decrease the surface area of your working electrode.
- Decrease the concentration of the background electrolyte.
- Decrease the concentration of the substrate.
- Check that a potential overload (see previous section) has not occurred.

***The results show peaks or steps at the inverse polarity I expect to see (i.e. a peak may be occurring at 0.5 V when it should be at -0.5 V).***

- Check that you are connected to the correct Output (+ or -) of the e-corder. See [Connecting the Adaptor, page 8](#).

- If you are using Chart or Scope, software check that the Invert box is checked or not, as appropriate, for the potential recording channel.

***The results show peaks (or steps) at different potentials than you expect.***

- See previous section.
- Make sure the reference electrode is connected to the correct potentiostat lead wire. Connection of the reference electrode to either the working or auxiliary leads can cause rapid electrode deterioration requiring electrode replacement.
- Check the condition of the reference electrode. All reference electrodes age. For example an Ag/AgCl electrode will age with exposure to light. Eventually the white/light grey coating of AgCl on the central silver wire will be replaced by a brown/black coating of silver oxide. As this process takes place the apparent positions of your voltammetric peaks will appear to shift to different potentials. Most Ag/AgCl electrodes can be quickly regenerated by placing the silver wire in 1 M hydrochloric acid solution for a minute or two (any silver oxide is quickly reconverted to AgCl), although you may wish to remove the old coating completely and re-chloridize the silver wire to ensure a good coating of fresh AgCl. In either case replace the newly coated wire back into the electrode casing with freshly prepared KCl solution. Aged calomel (Hg/Hg<sub>2</sub>Cl<sub>2</sub>) electrodes should usually be replaced. Reference electrodes using a gelled electrolyte will deteriorate if the gel dries out.

***The results show peaks in the negative current direction when you want them in positive direction.***

- Check that the Invert box is checked or not, as appropriate, for the current recording channel. See [Inverting the Signal](#), page 15.

***The results show peaks (or steps) at different potentials than you expect.***

- See previous section.
- Make sure the reference electrode is connected to the correct potentiostat lead wire!
- Check the condition of the reference electrode. All reference electrodes age. For example an Ag/AgCl electrode will age with

exposure to light. Eventually the white/light grey coating of AgCl on the central silver wire will be replaced by a brown/black coating of silver oxide. As this process takes place the apparent positions of your voltammetric peaks will appear to shift to different potentials. Most Ag/AgCl electrodes can be quickly regenerated by placing the silver wire in 1 M hydrochloric acid solution for a minute or two (any silver oxide is quickly reconverted to AgCl) — although you may wish to remove the old coating completely and re-chloridize the silver wire to ensure a good coating of fresh AgCl. Replace the newly coated wire back into the electrode housing and refill with freshly prepared KCl solution. Aged calomel (Hg/Hg<sub>2</sub>Cl<sub>2</sub>) electrodes should usually be replaced. Reference electrodes using a gelled electrolyte will deteriorate if the gel dries out.

### ***The results are contaminated by 'noise'.***

- Most often the noise encountered in electrochemical experiments is a manifestation of mains hum (50 or 60 Hz interference) from power lines and mains-powered equipment. Note that, depending on the sampling rate, aliasing can make such interference appear as a low frequency oscillation superimposed on your data. Ensure common grounding by attaching the e-corder and computer to the same power board which is then attached to a three pin (grounded) power socket. If consistent with the bandwidth required by your experiment, use a 10 Hz (or lower) filter of to record your results. Get an electrical technician to check that the grounding of the power socket itself is in good condition. Try the Mains Filter setting (Chart and Scope software) which is very effective at removing main induced artifacts
- Check electrode connections. Poor electrical contact to the electrodes will act as a source of noise. Particularly check the electrode cables: cables age, especially at the connectors where they are subject to mechanical wear.
- Ensure that the reaction vessel (and electrodes) are positioned as far as possible from power leads, computers, monitors and other mains-powered equipment. Ideally the reaction vessel should be enclosed by a Faraday cage. A Faraday cage (or other electrical shielding) is almost always required when measuring small currents.
- Random spikes occurring during your experiment are more likely to be due to nearby electrical equipment switching on and off. Equipment with high current drain such as electric motors in vacuum



pumps, refrigerators, or air-conditioner compressors should be particularly suspect. Try turning such equipment off during your experiment to identify the source of the problem. You may have to change your location (try a power socket on another circuit in the same room, or even move rooms completely).

Vertical line

# B

## A P P E N D I X B

# Specifications

### Control Ports

Analog signals:	Three female BNC, for E In, I Out and E Out.
I <sup>2</sup> C input and output:	Male and female DB-9 pin connectors. Provides control and power to the Adaptor.
Potentiostat Interface:	Female DB-25 pin connector. Provides digital I/O control over the potentiostat.
Power requirements:	±17 V DC +8 V DC 25 mA typical 2 W

### Physical Configuration

Dimensions (h × w × d):	54 × 105 × 167 mm 2.1 × 4.1 × 6.6 inches
Weight:	0.42 kg (0.93 lb)
Operating conditions:	0 – 35°C 0 – 90% humidity (non-condensing)

*eDAQ reserves the right to alter these specifications at any time.*





# Index

## A

- amperometry 17
- Applied Current  
Potentiostat/Galvanostat 16
- Applied Potential  
Potentiostat 15

## B

- back panel  
Potentiostat 7
- BANK Potentiostat  
compatible models 1

## C

- C4D Amp 2
- checking procedure 3
- chronoamperometry 21
  - analysis 25
  - Chart
    - Macintosh 22
    - Windows computers 21
  - multiple step 23, 26, 30
  - Scope 28
- chronocoulometry 31
- Cottrell equation 25, 30
- curve fitting 26
- cyclic voltammetry 17, 19

## D

- differential pulse voltammetry 17

## E

- E In connector 7
- E Out connector 7
- EChem software 17
- eDAQ Amps 2
  - C4D Amp 2
  - GP Amp 2
  - Picostat 2
  - Potentiostat 2
  - Quad pH Amp 2
  - Quad Tc Amp 2
  - QuadStat 2
- Er mode 11

## F

- fast cyclic voltammetry 18, 19
- fast linear sweep voltammetry 18
- front panel 6

## G

- Galvanostat mode 11, 18
- GP Amp 2

## I

I Out connector 7  
I<sup>2</sup>C connector 7  
input amplifier 10  
Interface Control Window 11

## L

linear scan techniques 18  
linear sweep voltammetry 17

## M

Mains Filter 36  
mains hum 36  
Model selection 11  
Modes of operation  
    Potentiostat, Galvanostat, -Er 11  
multiple step chronoamperometry 23, 26, 30

## N

noise 36  
normal pulse voltammetry 17

## O

Online indicator 6

## P

Picostat 2  
polarity 9  
potentiostat  
    Model selection 11  
Potentiostat (EA161) 2  
Potentiostat Interface port 8  
Potentiostat mode 11  
problems 33  
pulsed amperometry 17

## Q

Quad pH Amp 2

Quad Tc Amp 2

QuadStat 2

## R

Real cell  
    Potentiostat 15  
reference electrode  
    aging and regeneration 35

## S

Scan indicator 6  
square wave voltammetry 17  
Standby cell  
    Potentiostat 15

## T

Timed Events 24

## V

voltammetry  
    cyclic 17  
    differential pulse 17  
    fast cyclic 18, 19  
    fast linear sweep 18  
    linear sweep 17  
    normal pulse 17  
    square wave 17



# License & Warranty

## Trademarks

e-corder and PowerChrom are registered trademarks of eDAQ Pty Ltd. All eDAQ software, hardware, and documentation is protected by copyright. eDAQ retains the exclusive ownership of the trademarks and registered trademarks represented by its company name, logo, and product names.

## Responsibilities

You and any others using any eDAQ product agree to use it in a sensible manner for purposes for which it is suited, and agree to take responsibility for their actions and the results of their actions.

If problems arise with an eDAQ product, eDAQ will make all reasonable efforts to fix them. This service may incur a charge, depending on the nature of the problems, and is subject to the other conditions in this Agreement.

## Hardware Warranty

eDAQ Pty Ltd warrants eDAQ hardware, to be free of defects in material and workmanship for one year from the date of purchase. eDAQ Pty Ltd will repair or replace defective equipment as appropriate.

To obtain a warranty repair/replacement you must first notify us before return of the instrument and we will issue you with a RAN (return authorization

number). You must ship the defective product at your expense. We will pay return shipping. The product should be packed safely (preferably in its original packaging) and have the RAN on the shipping label. Returns sent without a RAN may be refused delivery.

This warranty does not cover hardware that has:

- been modified by the user in any way;
- been subjected to unusual physical, electrical, or environmental stress. This includes damage due to faulty power sockets, inadequate earthing, or power spikes or surges;
- been damaged because of incorrect wiring to ancillary equipment, or because of substandard, connectors or cables; or
- had the original identification marks removed or altered.

## Software License

You have the non-exclusive right to use supplied eDAQ software (Chart, Scope etc). Your employees or students, for example, are entitled to use it, provided they adhere to this agreement. Each separate purchase of the eDAQ software licenses it to be used on two computers at any given time (on one computer for data acquisition with a e-corder hardware unit, and on a second computer for the analysis of existing data files). Although multiple copies of a program may exist on several

computers, more than two copies must not be used simultaneously. Departmental/company licences are available if you wish to run more than two copies simultaneously.

## Technical Support

Please register your unit to receive technical support. Technical assistance is available via email. Please describe the problem with as much detail as possible. Include a small example data file, if appropriate. Please also state:

- the model and serial number of your e-corder unit and Potentiostat Adaptor.
- the type of computer and operating system being used (for example Windows XP, or Macintosh OS 10.2)
- the software version you are using (for example Chart v5.1.2)

We endeavor to answer all your questions, but in some cases, for example where the problem relates to the other equipment that you are using, a nominal fee may be charged.

## Jurisdiction

eDAQ Pty Ltd is bound by the laws of New South Wales in Australia, and any proceedings shall be heard by the Supreme Court of New South Wales in Australia.

## Disclaimer

eDAQ reserves the right to alter specifications of hardware and software without notice.

No liability can be accepted for consequential damages resulting from use of eDAQ products.