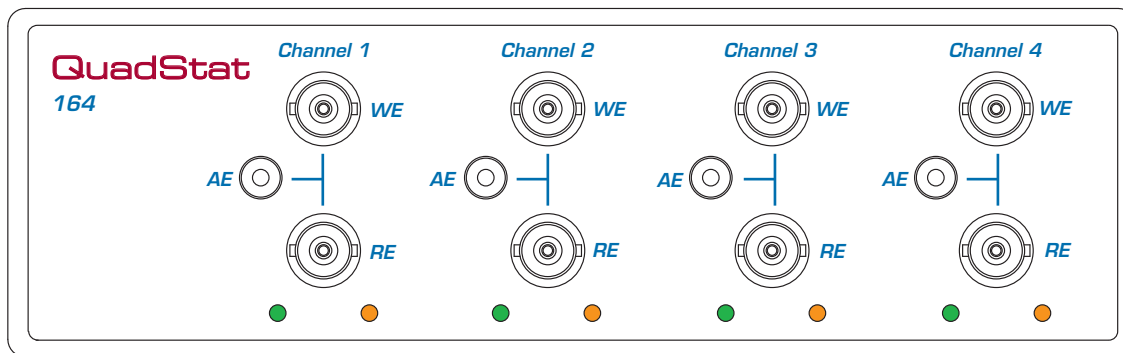
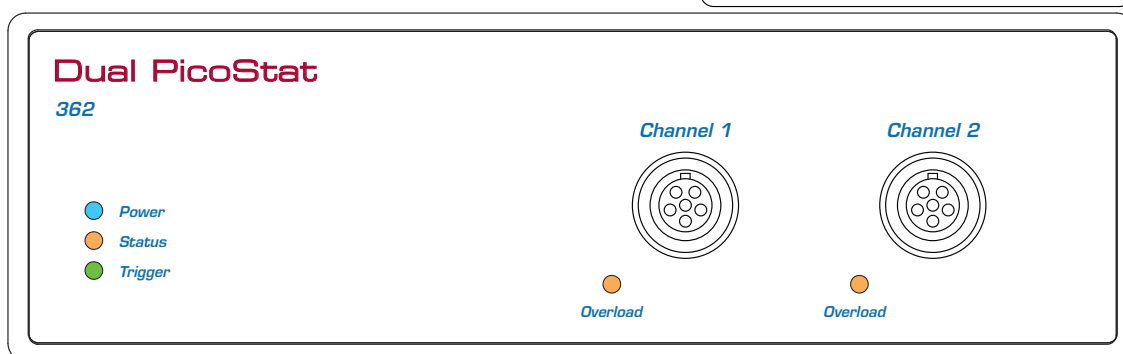
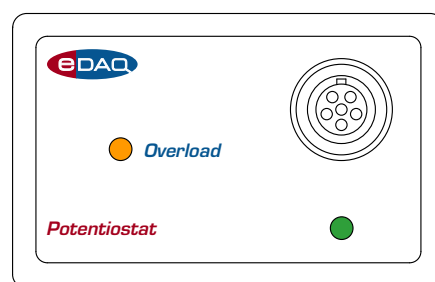


# *Potentiostat, Dual PicoStat & QuadStat*



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.

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Potentiostat (EA163)  
Dual Picostat (EA362)  
QuadStat (EA164)

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# Overview

**NOTE**

*This manual is for the EA163 Potentiostat, EA362 Dual Picostat, and EA164 QuadStat. If you have an older model EA160 or EA161 Potentiostat, or EA162 Picostat then please ask us to send you the appropriate manual.*

There are three eDAQ modular potentiostats:

- Potentiostat (EA163), [Chapter 2](#). Single channel, three electrode potentiostat/galvanostat with gain ranges of 20 nA to 100 mA;
- Dual Picostat (EA362), [Chapter 3](#). Two channel, three electrode, high sensitivity potentiostat with gain ranges of 2 pA to 10  $\mu$ A; and
- QuadStat (EA164), [Chapter 4](#). Four channel, three electrode potentiostat with gain ranges of 200 pA to 10 mA.

They are designed for use with an e-corder<sup>®</sup> system.

Some of the uses of the Potentiostat, Picostat, and QuadStat, are mentioned in [Chapter 5](#), and also in the *EChem Software Manual* which describes the use of the optional EChem software.

# How to Use this Manual

This manual describes how to set up and begin using your Potentiostat ([Chapter 2](#)), Dual Picostat ([Chapter 3](#)), or QuadStat ([Chapter 4](#)). Their use with Chart and Scope software is also described ([Chapter 5](#)).

The appendices provide technical and trouble shooting information.

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

## eDAQ Modular Potentiostats

The Potentiostat, Dual Picostat and QuadStat are modular units and must be used with an e-corder data recording system.

They can performing various voltammetric and amperometric experiments under full software control and are automatically recognised by the Chart, Scope or EChem software which control their gain range, signal filtering, and other settings.

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

## Checking the unit

Before you begin working with your Potentiostat, Dual Picostat, or QuadStat 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 placed as a pdf file on your computer hard disk when you install the software.

# 2

## C H A P T E R   T W O

# The Potentiostat

This chapter describes how to connect and use your model EA163 Potentiostat. If you have an older model EA160 or EA161 Potentiostat please refer to the documentation that came with your unit or contact eDAQ at [support@edaq.com](mailto:support@edaq.com) to obtain the correct document.

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

**NEW FEATURES:** If you have used the older EA161 Potentiostat then you will notice it is very similar to the new EA163 Potentiostat which has higher bandwidth (100 kHz) and so is more suitable for electrochemical impedance spectroscopy (EIS).

# The Front Panel

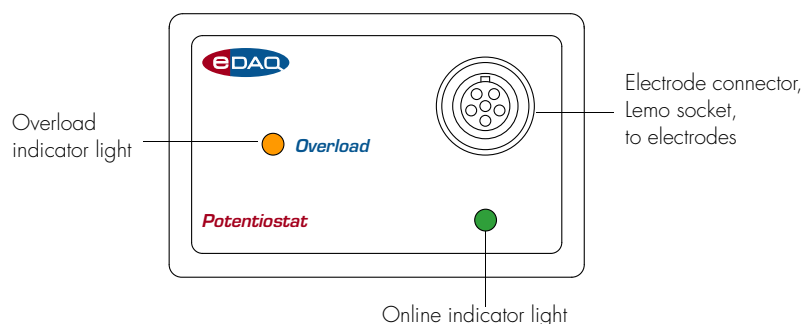
The front panel of the Potentiostat is shown in [Figure 2-1](#).

## The Electrode Connector

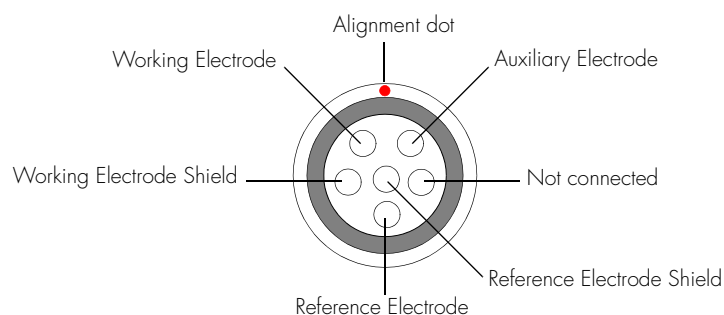
The electrode connector of the Potentiostat provides connection pins for the Working, Auxiliary and Reference electrode lead wires. The connector also provides connections for the lead shields which protect the signals in the cable wiring from electrical interference (noise pickup).

The pin assignments of the Potentiostat electrode connector are shown in [Figure 2-2](#). The Auxiliary and Reference electrode leads have

**Figure 2-1**  
The Potentiostat front panel.



**Figure 2-2**  
The Potentiostat electrode connector as seen when looking at the front panel.





**Table 2-1**  
Color-coding on the leads of the electrode cable.

Color	Electrode
Yellow	Reference
Green	Working
Red	Auxiliary

*To ensure good grip, the electrode cable alligator clips use a spring made from a good quality steel (stainless steel is unsuitable for strong springs). Avoid wetting of the alligator clips, especially with electrolyte solutions which can hasten corrosion. If the alligator clips are wetted then immediately disconnect from the Potentiostat, rinse the clips with a little deionized water from a wash bottle, to remove the electrolyte, and immediately dry by patting with paper tissue. The whole cable must then be allowed to dry thoroughly (several hours at least) before reuse.*

*Never immerse any part of the electrode cable in water, or other liquid!*

coaxial shields which are maintained at the respective electrode potential.

## Electrode Cable

The Potentiostat is supplied with an electrode cable comprising three leads, with each lead terminated by an alligator clip. The Reference and Working electrode leads are shielded to protect the signals from external interference. The alligator clips allow connection to a wide variety of electrodes, and the leads are color-coded to indicate the type of electrode to which they should be attached (Table 2-1).

For normal three-electrode potentiostat, page 13, or galvanostat, page 14, use, the reference electrode must never be connected to either the auxiliary (red) or working (green) leads, otherwise the current that would be passed through the electrode could effectively destroy it as a reference potential source.

When two-electrode potentiostat, or galvanostat, operation is required the auxiliary and reference leads (red and yellow) should be attached to the single 'counter electrode'. The green lead is attached to the working electrode.

When using in **ZRA** (zero resistance ammeter) mode, connect the working (green) and auxiliary (red) leads to the two electrodes (or circuit test points) across which to measure the current, page 14. The reference lead (yellow) can be connected to a reference electrode (or circuit test point) to measure the potential difference to the auxiliary (and working) leads.

When using **High Z** (high impedance) mode, connect the working (green) lead to one electrode and the reference lead (yellow) lead to a reference electrode to measure the potential difference between the leads, page 14. On the EA163 Potentiostat the auxiliary electrode lead

is internally disconnected, so that an open circuit potential can be monitored. On an EA161 Potentiostat the auxiliary lead (red) must be disconnected for an open circuit potential measurement — if connected to a third electrode (or test point) then a ZRA current signal will be provided at E Out, [Figure 2-3](#).

## The Online Indicator

Located at the bottom right of the front panel is the Online indicator., [Figure 2-1](#). When lit, it indicates that the software (such as EChem, Chart or Scope) has located and initialised the Potentiostat. If the light does not go on when the software is run, check that the Potentiostat is properly connected. If there is still a problem, please refer to [Appendix B, page 85](#).

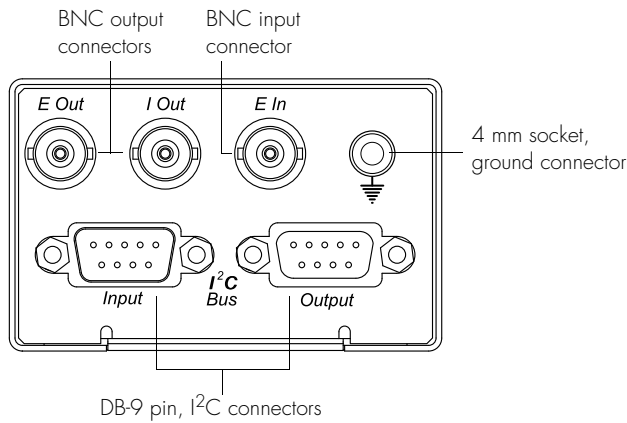
## The Overload Indicator

Located on the left-hand side of the front panel is the Overload indicator, [Figure 2-1](#). When lit, this indicates that the Potentiostat is (or has gone) out of compliance, which usually occurs because of an open circuit or excessive resistance in the electrochemical cell. Higher resistances can be often be encountered when electrodes are fouled by the products of electrolysis reactions. The Potentiostat tries to compensate by increasing the compliance potential (that is, the potential between the auxiliary and working electrodes). If the compliance voltage exceeds specification (about 11 V) potential control of the cell is lost and drifting, or oscillation, of the signal can be seen. Any data collected during this period is unreliable and should be discarded.

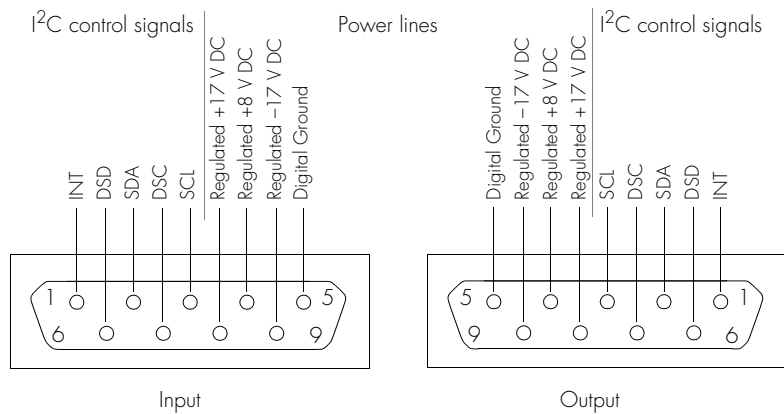
The Overload indicator will light as soon as there is an overload and will stay on until the recording has stopped.

If the indicator light comes on repeatedly, and your connections are good, then try bringing your electrodes closer together, and/or increasing electrolyte concentration, and/or modifying your experimental conditions to avoid fouling of the electrodes. Redesigning your electrochemical cell may be necessary. Normally cells are designed to keep the reference and working electrodes very close together, however, when a potential overload occurs, you also need to consider the distance between the auxiliary and working electrodes.

**Figure 2-3**  
The Potentiostat back panel.



**Figure 2-4**  
The pin assignments for the I<sup>2</sup>C DB-9 connectors.



Note that a potential overload is quite different from a current overload condition. A current overload is caused when the current signal exceeds the full scale limits of the range setting of the current channel, and is usually caused by a low resistance between the electrodes.

## The Back Panel

The back panel of the Potentiostat is shown in [Figure 2-3](#).

## E Out, I Out and E In Connectors

The Potentiostat back panel has three BNC connectors labelled E Out, I Out, and E In. The E In is connected to the Output of the e-corder, usually Output + is used. Reverse the polarity of the Potentiostat by using e-corder Output –.

The Potentiostat provides two signals: the current signal (I Out) indicating the current flow between the working and auxiliary electrodes, and the potential signal (E Out) indicating the potential difference between the working and reference electrodes. Note that the E Out signal is inverted with respect to the applied potential.

For most situations I Out is connected to e-corder input channel 1, and E Out to e-corder input channel 2. 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 Potentiostat to other e-corder input channels.

### **WARNING!**

*The I<sup>2</sup>C connectors are for the power and control of eDAQ Modular Potentiostats, page 2, and should not be used for connection to any other device.*

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

The Potentiostat back panel, [Figure 2–3](#), has two DB-9 pin 'I<sup>2</sup>C bus' connectors labelled Input and Output. The Input connector provides power to the Potentiostat and carries the various control signals (for gain range and filter selection) to and from the e-corder. A cable is provided with the Potentiostat for this purpose. The pin assignments are shown in [Figure 2–4](#).

The Output connector can be used for the attachment of other eDAQ Amps.

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

## Grounding Connector

The Potentiostat back panel, [Figure 2–3](#), has a 4 mm grounding socket. This enables connection of a Faraday cage (with the green grounding cable included with the Potentiostat) the use of which can greatly diminish electrical noise. The Potentiostat is supplied with a green

colored ground cable terminated with a 4 mm pin (attaches to Potentiostat back panel) and an alligator clip (for attachment to Faraday cage). If your Faraday cage is already earthed by its own ground connection then you should not use this cable (otherwise a second pathway to earth would exist which could result in a 'ground loop' and increased signal interference! You can try grounding the Faraday cage via its own connection to earth, or via the Potentiostat ground cable — but *not by both* methods simultaneously.

The construction of the Faraday cage can range from a simple cardboard box covered with aluminium foil, in which the electrochemical cell is located, to a more sophisticated copper mesh enclosure or sheet-metal box. But in all cases, it is essential that the Faraday cage be electrically grounded to act as an effective shield against electrical interference.

The Potentiostat itself is grounded via its connection to the e-corder unit which is in turn earthed via the three pin mains power connector. It is of course important that the power socket that you are using is well earthed.

## Connecting the Potentiostat

Your Potentiostat will have been supplied with an I<sup>2</sup>C cable (DB-9 pin connectors at either end), and three cables with BNC connectors at either end.

**Table 2-2**  
Potentiostat to e-corder connections as shown in Figure 2-5 and Figure 2-6.

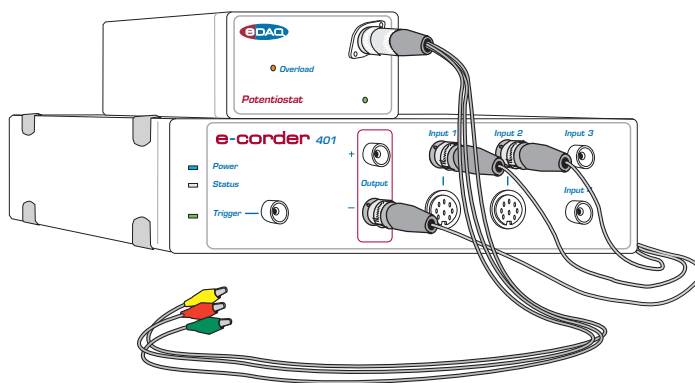
<i>Potentiostat rear panel</i>	<i>e-corder front panel</i>
I Out	Input 1
E Out	Input 2
E In	Output +

**Table 2-3**  
Potentiostat to e-corder connections, reverse polarity.

<i>Potentiostat rear panel</i>	<i>e-corder front panel</i>
I Out	Input 1
E Out	Input 2
E In	Output –

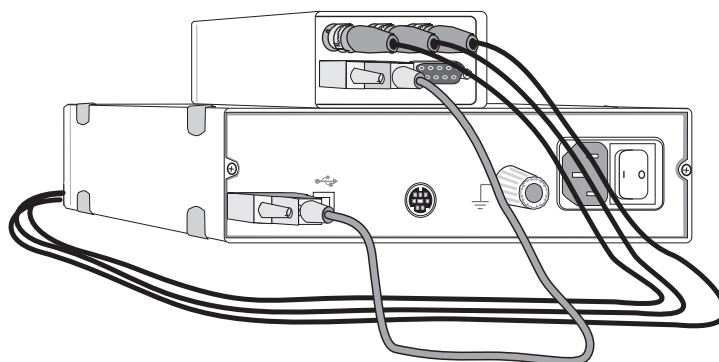
**Figure 2-5**

The Potentiostat shown connected to an e-corder, front view, as described in [Table 2-2](#).



**Figure 2-6**

The Potentiostat shown connected to an e-corder, back view.



First make sure that the e-corder is turned off. Then connect the I<sup>2</sup>C cable to the I<sup>2</sup>C connector on the back panel of the e-corder, and the other the other end to the I<sup>2</sup>C Input connector on the back panel of the Potentiostat. Use the three BNC cables to connect the back panel of the Potentiostat to the front panel of the e-corder as shown 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 2-5](#) and [Figure 2-6](#).

To operate the Potentiostat with the reverse polarity make the connections as shown in [Table 2-3](#). 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 Potentiostat uses two e-corder input channels during normal operation. 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 Input Channel 1, and the potential signal (E Out) to Input Channel 2 of the e-corder. 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* which are installed as pdf files in the eDAQ Documentation folder on your computer hard disk.

## 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 Potentiostat as described above, you are ready to begin.

When the e-corder is turned on, and Chart software started, the Potentiostat Online indicator (green) should light.

From the Channel 1 Function pop-up menu, select the 'Potentiostat' command, which opens the Potentiostat Control window, [Figure 2-7](#).

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 'Potentiostat' then the software has not

recognised the Potentiostat. Exit the software, check all your connections and try again).

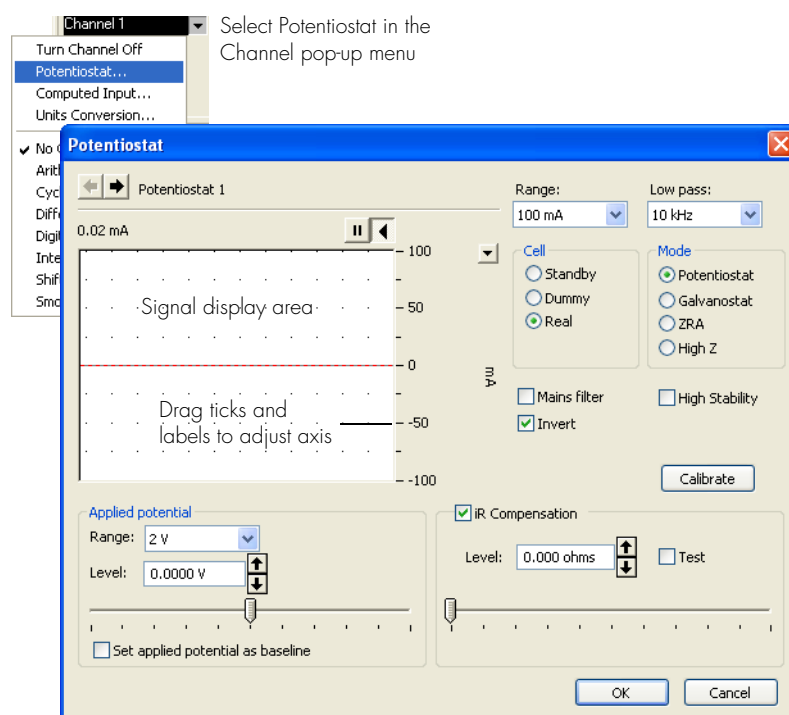
By default, the control window opens with the Potentiostat in Standby mode, that is with the reference and working electrodes isolated so that no current will flow through your electrodes. To connect to the Potentiostat lead wires you must select Real mode. When you click Cancel or OK the Potentiostat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

Now select Dummy mode operation. You will need to adjust the gain range to 20  $\mu\text{A}$  to accommodate your signal amplitude. You can now adjust the applied potential with the slider bar, or by entering the exact potential with the A-button. The resulting current signal should obey Ohm's law:

$$I = E/R$$

so that an applied potential of 1 V should produce a current of 10  $\mu\text{A}$ , while other potential settings should produce corresponding currents.

**Figure 2-7**  
Potentiostat controls with  
Chart software.



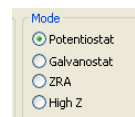


# Potentiostat Control Window

With Chart software, the Potentiostat Control window, [Figure 2-7](#), is accessed from the Potentiostat command in the Channel Function pop-up menu. It control the various current ranges and filtering options for the Potentiostat.

With Scope software, the corresponding controls are shown in [Figure 2-8](#).

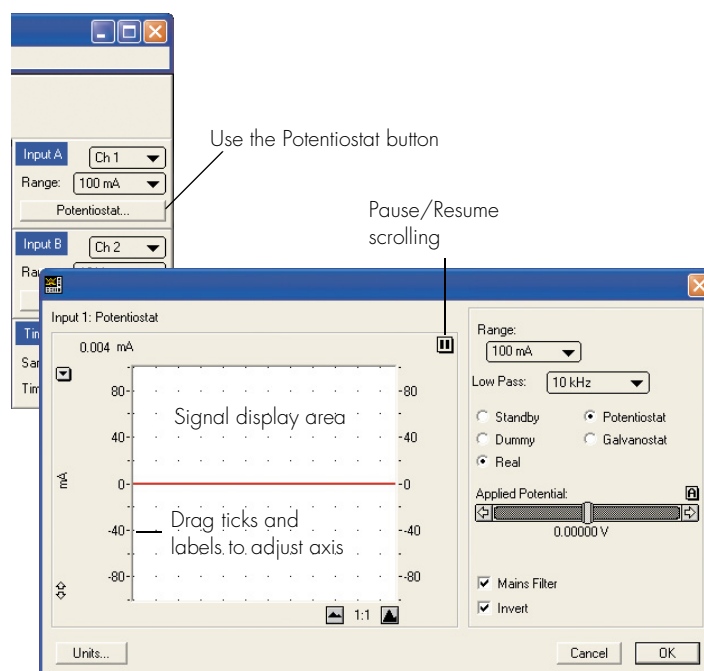
## Modes of Operation



The EA161 Potentiostat can be operated in several different modes by selecting the appropriate radio button:

- **Potentiostat** (Chart, Scope or EChem software), described below. For three-electrode use connect the working (green), reference and auxiliary (red) leads appropriate electrodes (or circuit test points) The current signal is provided at I Out, [Figure 2-3](#). The potential signal is provided at E Out. When two-electrode potentiostat operation is required the auxiliary and reference leads (red and yellow) should be attached to the single 'counter electrode'.

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



**NOTE.** When using early models of EA161 Potentiostat (serial numbers 161-001 to 161-022) as a ZRA you should connect the working electrode lead, and the cable of the Grounding Connector (page 8), to the two electrodes (or circuit test points) across which to measure the current.

- **Galvanostat** (Chart and Scope software), [page 67](#). Connect the electrodes as described for potentiostat operation, above. Note especially that the potential signal is now provided at I Out, [Figure 2-3](#). The current signal is provided at E Out.

- **ZRA**, zero resistance ammeter, (Chart and Scope software). Connect the working (green) and auxiliary (red) leads to the two electrodes (or circuit test points) across which to measure the current, the current signal is provided at I Out. The auxiliary (red) lead is earth return. The reference lead (yellow) can be connected to a reference electrode (or circuit test point) to measure the potential difference to the auxiliary (and working) leads. The high impedance potential signal (if used) is supplied at E Out, [Figure 2-3](#).


- **High Z**, high impedance voltmeter (Chart and Scope software). Connect the working (green) lead to one electrode and the reference lead (yellow) lead to a reference electrode to measure the potential difference between the leads. The high impedance potential signal is delivered at I Out. The auxiliary lead (red) can be connected to a third electrode (or test point) to provide a ZRA current signal at E Out, [Figure 2-3](#).

Please make sure the electrode lead wires are connected appropriately to your experiment, before operating in any of these modes. In particular, incorrect placement of leads may damage the reference electrode, if one is being used.

## Signal Display

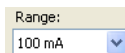
The current signal is previewed in the scrolling display area. Note that the signal is not being recorded to hard disk at this stage, and that when the window is closed the signal trace will be lost.

By using the Dummy or Real modes you can investigate the effect of the [Applied Potential](#), [page 19](#), on the current signal.

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

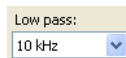
You can shift or stretch the vertical Amplitude axis to make the best use of the available display area — similar to the amplitude axis in the main Chart, Scope or EChem window.

## Setting the Range

A small rectangular box with a light beige background. It contains the text 'Range:' in a small font, followed by a dropdown menu. The dropdown menu is currently set to '100 mA' and has a small blue arrow pointing downwards on its right side.

Use the Range pop-up menu to select the input current range (channel sensitivity). The Potentiostat has ranges of 100 mA to 2 nA, while 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

A small rectangular box with a light beige background. It contains the text 'Low pass:' in a small font, followed by a dropdown menu. The dropdown menu is currently set to '10 kHz' and has a small blue arrow pointing downwards on its right side.

The Potentiostat incorporates four low-pass filters at 10 kHz, 1 kHz, 100 Hz and 10 Hz for removal of high frequency signals ('noise').

In addition the e-corder provides filter settings at 1, 2, 5, 20, 50, 200, 500, and 2000 Hz.

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

With Chart and Scope 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. 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 few seconds to 'learn' the pattern of the interference so that you will need to record for longer than this for it to take full effect. The mains filter can be employed even for experiments in which there are sudden potential jumps.

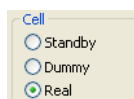
## Inverting the Signal

A small rectangular box with a light beige background. It contains a checked checkbox (a small square with a checkmark inside) followed by the text '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



The Potentiostat can be in one of three cell modes, controlled by the Cell radio buttons:

- **Standby:** If Standby mode is selected the electrode lead wires are disconnected, and the internal dummy cell is connected. The external (real) cell is not connected until the Potentiostat Control window is closed and the Chart, Scope or EChem Start button is clicked. This mode is used if you do not wish to alter the state of the external cell until the method is actually performed. The Applied Potential slider bar control is disabled in this mode.
- **Dummy:** the Potentiostat is connected to the internal 1 M $\Omega$  dummy cell. You can then use the Applied Potential slider control to vary the voltage applied to the dummy cell. The Potentiostat will remain connected to the dummy cell even when the Potentiostat Control window is closed and Chart, Scope or EChem is recording. This is useful for testing the Potentiostat.
- **Real:** the external electrodes are connected to the Potentiostat. The Applied Potential slider control, [Figure 2–7](#) and [Figure 2–8](#), can be used to set the potential 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 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.

## High Stability Operation

☐ High Stability

If the **High Stability** box is ticked then extra capacitance is introduced into the Potentiostat control loop. This stabilizes the Potentiostat in situations where oscillation is encountered (for example where large surface area electrodes are being used in highly resistive solutions).

Do NOT use High Stability, when in Potentiostat mode, unless you first encounter stability problems. High Stability decreases the bandwidth of the Potentiostat control loop. Thus High Stability mode should never be used when fast sweep rates ( $> 1 \text{ V/s}$ ), or when short term pulses ( $< 0.1 \text{ s}$ ), are employed as it will produce a noticeable phase lag between the desired and actual applied potential. Also High Stability operation should not be used to try to correct for oscillations introduced by excessive iR compensation.

High Stability operation can be used routinely when performing fixed potential experiments with amperometric sensors where the response time of the sensor is relatively slow ( $> 0.01 \text{ s}$ ).

It is likely you will need to use High Stability mode when in Galvanostat mode, especially with highly resistive loads.

Note that High Stability operation is not required for either ZRA or High Z operational modes, because the Potentiostat control loop is disabled.

## Current Signal Zero Point Calibration

The Calibrate button is available in Potentiostat and ZRA operating modes, [page 13](#). When the Calibrate button is clicked it corrects for any internal offset error on the current signal. This is only required for very accurate determination of signal values. Current accuracy will be improved from about  $\pm 1\%$  of full scale of range to better than  $\pm 0.2\%$ . If you do not need this accuracy then you do not need to use the Calibrate button!

For best results allow about 10 minutes after opening powering up the e-corder before using the Calibrate function. This allows the unit to warm up — ambient temperature variation of more than a few degrees during an experiment may require periodic recalibration to maintain maximum accuracy.

When calibrating in ZRA mode you must first remove the Electrode connector from the Potentiostat.

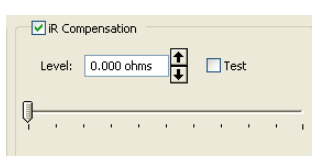
When in Potentiostat mode clicking the Calibrate button will zero the current signal, using the Dummy cell, including any signal due to any

small offset from the e-corder Output at E In. Thus it should be considered a 'relative zero'.

Recalibration is required after you:


- select a different range for the Potentiostat applied potential, [page 19](#);
- turn iR Compensation on or off, [page 18](#).
- change from Potentiostat to ZRA operating mode;
- change from ZRA to Potentiostat operating mode. However, to get true absolute current measurements in Potentiostat mode (independent of small offsets at E In) first calibrate in ZRA mode and then switch to Potentiostat mode without recalibrating.

Note that, in all cases, the Calibrate button does NOT remove background current signals due to actual electron flow in the real cell.



## iR Compensation

iR compensation is available only when in Potentiostat mode. Positive feedback compensation is used.

When the iR Compensation panel is on, then the degree of iR Compensation can be adjusted using the slider bar. First adjust the applied potential to a value where no Faradaic process occurs, use the  buttons for fine control. Now use the slider bar to gradually increase the amount of compensation until the current signal goes into oscillation, then decrease the compensation until stability is restored. For very fine control of iR Compensation use the **Test** checkbox. This applies a small perturbation (1 Hz, 10 mV amplitude square wave) to the electrode. The iR Compensation is adjusted until an appropriate amount of ringing is seen on the potential signal.

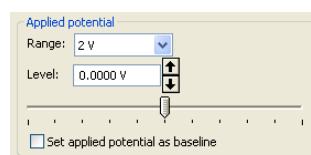
The maximum amount of iR compensation available depends on the selected gain range, [page 93](#).

Note that iR Compensation is set at the particular applied potential you have chosen. If you then proceed to do an experiment involving a potential sweep, the amount of compensation required for complete

compensation will vary during the sweep, and it is possible that the potentiostat will go into oscillation at some point. To avoid this happening it is usual to always slightly undercompensate, that is, to find the point of ideal compensation and then to reduce the setting slightly. The amount of 'undercompensation' is usually determined by trial and error for a particular experiment.

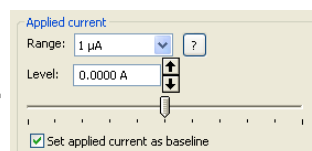
Before using iR Compensation you should always consider other methods of reducing the uncompensated resistance. For example, could the reference electrode be more closely positioned to the working electrode (perhaps by redesigning your reaction chamber), or could the background electrolyte concentration be increased? Also check to ensure that the reference electrode is not clogged or dried out. It is always best to minimize cell resistance within the reaction chamber rather than trying to overcome the problem later with the potentiostat.

## Applied Potential




The Applied Potential controls are enabled when either the Dummy or Real cell is selected. It allows you to adjust the voltage applied to either the dummy cell or external electrodes, depending on the mode selected.

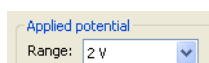
## Applied Current



The Applied Current controls appear when in Galvanostat mode (replacing the Applied Potential controls), see [Figure 5–7, page 68](#). The controls are enabled when either the Dummy or Real cell is selected. Use them to adjust the current applied, [Figure 5–9, page 69](#).

The  button advises on the correct values to be entered into Units Conversion of the current signal channel, see [Figure 5–8, page 68](#).

## Range



When using Chart software this control limits the range over which the applied potential can be set. Smaller ranges offer finer control with the slider bar of the applied potential, [Figure 2–7](#). In Galvanostat mode

this control changes so as to limit the range over which the applied current can be set, [Figure 5–7, page 68](#).

## Remember Potential ☐ Set applied potential as baseline

Check the ☐ Set applied potential as baseline box to remember the value of the applied potential when the Potentiostat control window, [Figure 2–7, page 12](#), is closed. (The potential value is transferred to the Stimulator baseline control, [Figure 5–2, page 63](#)).

# Maintenance

The Potentiostat will not require maintenance during daily operation. However, you should periodically check the instrument for optimum results by switching to potentiostat mode (with iR Compensation off) and applying a known potential,  $E$ , to the Dummy Cell and checking that the resulting current signal value,  $I$ , is in accordance with Ohm's law:

$$I = E/R$$

where  $R$  is the resistance, and is 100 kohm (EA163) or 1 Mohm (EA161) for the dummy cell. Thus a signal of 10  $\mu$ A (EA163) or 1  $\mu$ A (EA161) should be obtained when a potential of 1 V is applied. Try several different potentials and make sure an appropriate current signal is observed in each case.

If this test produces the expected results then your Potentiostat is likely to be functioning correctly. Next use the Potentiostat in Real Cell mode to check the electrode cables by attaching them to a resistor (usually a resistor of  $10^3$ – $10^8$  ohm is ideal) with the working electrode lead on one side of the resistor and the auxiliary and reference leads connected to the other. If the current signal does not obey Ohm's law, then it is likely that the electrode leads have become damaged.

However, if both the Dummy Cell and Real Cell tests produce the expected results, but you are still experiencing difficulties with your experiments, then check the electrodes (reference electrodes, in particular, tend to become clogged or dry out with age), and the design and condition of the reaction vessel, and any salt bridges that you are using.



# 3

## C H A P T E R   T H R E E

# The Dual Picostat

This chapter describes how to connect and use your Dual Picostat (EA362).

The Dual Picostat is designed to function as a:

- potentiostat (Chart, Scope, or EChem software)
- bipotentiostat (Chart software)
- four electrode potentiostat (Chart, Scope or EChem software)
- ZRA, zero resistance ammeter, (Chart or Scope software), or
- high impedance voltmeter (Chart or Scope software).

The mode of operation is under software control.

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

**IMPORTANT:** The EA362 Dual Picostat supersedes the EA162 Picostat, and it has many new features. If you are using an EA162 Picostat please refer to its manual (which can be obtained from our web site at <http://www.edaq.com/support.php> )

# The Front Panel

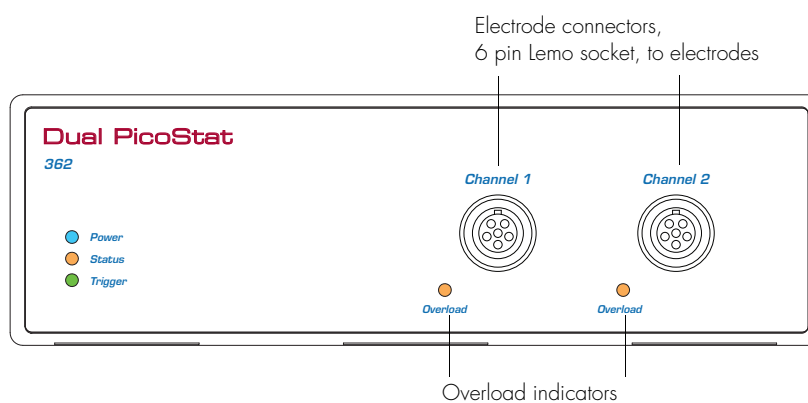
The front panel of the Dual Picostat is shown in [Figure 3-1](#).

## Electrode Connectors

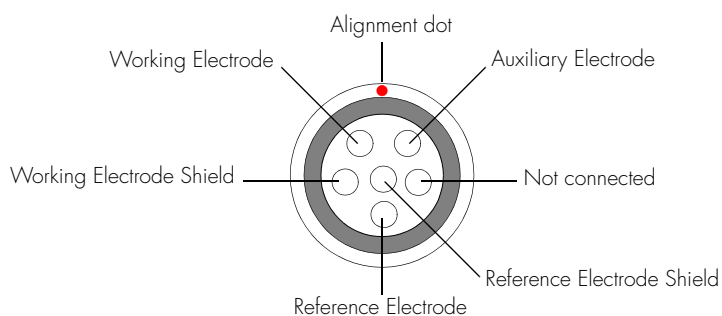
The electrode connectors of the Dual Picostat accept the cables that go to the Working, Auxiliary and Reference electrodes. These connectors also provide connections for the lead wire shields which protect the signals in the cable wiring from electrical interference (noise pickup).

The pin assignments of the Dual Picostat electrode connectors are shown in [Figure 3-2](#). The Working and Reference electrode leads have coaxial shields which are maintained at the respective electrode potentials to minimise lead capacitance.

**Figure 3-1**  
The Dual Picostat front panel.



**Figure 3-2**  
A Dual Picostat electrode connector as seen when looking at the front panel.



**Table 3–1**  
Color-coding on the leads of the electrode cables.

Color	Electrode
Yellow	Reference
Green	Working
Red	Auxiliary

## Electrode Cable

The Dual Picostat is supplied with two three-lead electrode cables, with each lead terminated by an alligator clip. The Reference and Working electrode leads are shielded to protect the signals from external interference. The alligator clips allow connection to a wide variety of electrodes. The leads are color-coded to indicate the type of electrode to which they should be attached (Table 3–1).

For normal three-electrode use, the reference electrode must never be connected to either the auxiliary or working leads, otherwise the current that would be passed through the reference electrode could effectively destroy it as a reference potential source.

If two-electrode operation is required the auxiliary and reference electrode leads (red and yellow) can be attached to the single ‘counter electrode’. The green electrode lead is attached to the working electrode.

When attaching the cable to the Dual Picostat make sure that the red dot on the cable connector is aligned with the top of the Dual Picostat electrode connector, Figure 3–2. Insert the cable connector and push gently until it locks into position. To remove the cable pull the knurled connector body gently until it disengages. Do NOT twist the connector, or pull on the cable! The cables can be left in position when not in use which will extend cable life-time by minimising ‘wear and tear’.

## The Power Indicator



Located at the left of the front panel is the Power indicator, Figure 3–1 which will illuminate when the unit is powered up.

## The Status Indicator



When software (such as EChem, Chart or Scope) has located and initialised the Dual Picostat the Status Indicator will turn on. If the light does not go on when the software is run, check that the Dual Picostat is properly connected. If there is still a problem, please refer to Appendix B [Troubleshooting](#), page 85.

## The Overload Indicator



Located below and slightly to the left of the electrode cable connectors, the Overload indicators, [Figure 3-1](#) will light continuously when an 'out-of-compliance' situation occurs, usually because of an open circuit condition (such as an unconnected or faulty electrode), or the resistance is too high in the electrochemical cell. Higher resistances can be often be encountered when electrodes are fouled by the products of electrolysis reactions. The Dual Picostat tries to compensate by increasing the compliance potential (that is, the potential between the auxiliary and working electrodes). If the compliance voltage exceeds specification, about 12 V, potential control of the sample is lost and drifting, or oscillation, of the signal can be seen. Any data collected during this period is unreliable and should be discarded.

The Overload indicator will remain lit, and an alarm beep will sound once an overload has occurred, even if the overload condition subsequently goes away — it will be reset once the scan has finished.

If the indicator comes on repeatedly, and your connections are good, then try bringing your electrodes closer together, and/or increasing electrolyte concentration, and/or modifying your experimental conditions to avoid fouling of the electrodes. Redesigning your electrochemical cell may be necessary to minimise inter-electrode resistances. Normally electrochemical cells are designed to keep the reference and working electrodes very close together, however, when a potential overload occurs, you also need to consider the distance between the auxiliary and working electrodes.

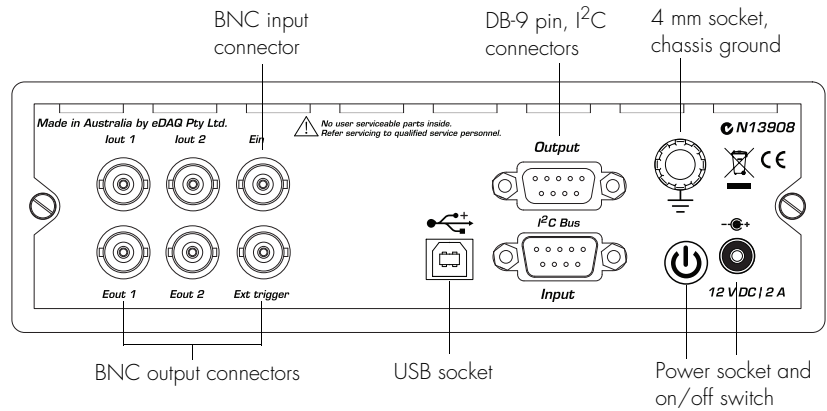
NOTE: A potential overload is quite different from a current overload condition. A current overload is caused when the current signal exceeds the full scale limits of the sensitivity setting of the current channel. This is due to a low resistance between the auxiliary and

working electrodes. The Overload indicator will flash if the current signal exceeds its range.

## The Back Panel

The back panel of the Dual Picostat is shown in [Figure 3–3](#).

**Figure 3–3**  
The Dual Picostat back panel.



## Power Connector, On/Off

The Dual Picostat requires 12 V DC power from a mains adaptor (supplied). Press the Power On/Off button for about a second to turn the unit on and off. Because it uses DC power the Dual Picostat can be used inside Faraday cages if required.

## E Out, I Out and E In Connectors

The Dual Picostat back panel has six BNC connectors labelled E out 1 and 2, I out 1 and 2, and E in and Ext Trigger. The E in is connected to the Output of the e-corder, usually Output + is used.

The Dual Picostat provides up to four analog signals: the potential signals (E out 1 and 2); and the current signals (I out 1 and 2).

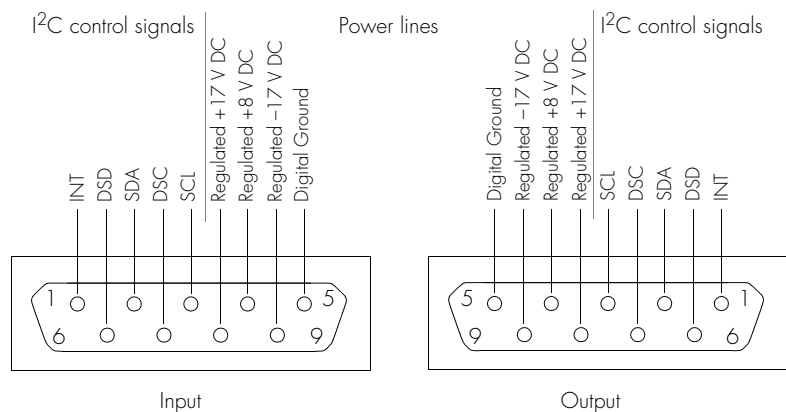
See the section [Connecting the Dual Picostat](#), [page 27](#), for connection details.

**Figure 3–4**

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

**WARNING!**

The I<sup>2</sup>C connectors are for the control of eDAQ Modular Potentiostats, page 2, and should not be used for connection to any other device.



## USB socket

The USB socket is for future development and at the time writing it is unimplemented. Check our web site periodically for the release of new features.

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

The Dual Picostat back panel, Figure 3–3, has two DB-9 pin 'I<sup>2</sup>C bus' connectors labelled Input and Output. The Input connector carries the various control signals (for gain range and filter selection) to and from the e-corder. A cable is provided with the Dual Picostat for this purpose. The pin assignments are shown in Figure 3–4.

The Output connector can be used for the attachment of a second Dual Picostat or other suitable eDAQ Amp. More information about the I<sup>2</sup>C connector can be found in your *e-corder Manual*.

## Grounding Connector

The Dual Picostat back panel, Figure 3–3, has a 4 mm grounding socket. This enables connection of a Faraday cage (with the green grounding cable included with the Dual Picostat) the use of which can greatly diminish electrical noise. The construction of the Faraday cage can range from a simple cardboard box covered with aluminium foil, in which the electrochemical cell is located, to a more sophisticated copper mesh enclosure or sheet-metal box.

In all cases, it is essential that the Faraday cage be electrically grounded to act as an effective shield against electrical interference. The case of the Dual Picostat is grounded via its connection to the e-corder unit which is in turn earthed via the three pin mains power connector. It is of course important that the power socket that you are using is well earthed. The purpose of this ground cable to the Faraday cage is to provide an easy means of grounding the cage — please note that it is not for grounding the Dual Picostat.

If your Faraday cage is already earthed by its own ground connection then you should not use the ground cable to the Dual Picostat! Use of the cable in this instance will provide a second pathway to earth which could result in a ground loop which can actually increase signal interference! Thus you can try grounding the Faraday cage via its own connection to earth, *or* via the Dual Picostat ground cable — but *not by both* methods simultaneously.

## Connecting the Dual Picostat

The Dual Picostat will have been supplied with an I<sup>2</sup>C cable (DB-9 pin connectors at either end), and two cables each with three BNC connectors at either end.

First make sure that the e-corder is turned off. Then connect the I<sup>2</sup>C cable to the I<sup>2</sup>C connector on the back panel of the e-corder, and the other the other end to the I<sup>2</sup>C Input connector on the back panel of the Dual Picostat. Use the BNC cables to connect the back panel of the Dual Picostat to the front panel of the e-corder as in [Table 3-2](#).

With these connections, when you use the software to set a more positive voltage at 'E in', a more oxidising (anodic) potential will be applied at the working electrode. Such an arrangement is shown in [Figure 3-5](#) and [Figure 3-6](#).

To operate the Dual Picostat with the reverse polarity change the connection at e-corder Output + to e-corder Output -. Now when you set a more positive voltage in the software, a more negative potential is sent to E in, and a more reducing (cathodic) potential will be applied at the working electrode.

**Table 3–2**

Dual Picostat to e-corder BNC connections for single channel or 4-electrode mode operation.

<i>Dual Picostat rear panel</i>	<i>e-corder front panel</i>
Iout 1	Input 1
Eout 1	Input 2
Ein	Output +

**Table 3–3**

Dual Picostat to e-corder BNC connections, for bipotentiostat or dual channel operation.

<i>Dual Picostat rear panel</i>	<i>e-corder front panel</i>
Iout 1	Input 1
Eout 1	Input 2
E In	Output +
Iout 2	Input 3
Eout 2	Input 4

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

The Dual Picostat typically uses two, three, or four e-corder input channels during normal operation, depending on its configuration:

- when used as a single channel potentiostat, or when used as a four-electrode potentiostat, there are two signals (current and potential). Connect Iout 1 (current signal) to e-corder Input 1, and Eout 1 (potential signal) to e-corder Input 2, [Table 3–2](#).
- when using the Dual Picostat as two potentiostats with two separate samples, there are two current signals (Iout 1 and Iout 2) and two potential signals (Eout 1 and Eout 2). Connect the Picostat as shown in [Table 3–3](#).
- when used as a bipotentiostat, there are two current signals (Iout 1 and Iout 2) and one potential signal (Eout 1). Connect the Picostat as shown in [Table 3–3](#).

Note that it is also possible when using Chart software to connect the Dual Picostat to other e-corder input channels but in these cases the discussion that follows would change accordingly.



**Figure 3-5**  
The Dual Picostat shown  
connected to an e-corder,  
front view.



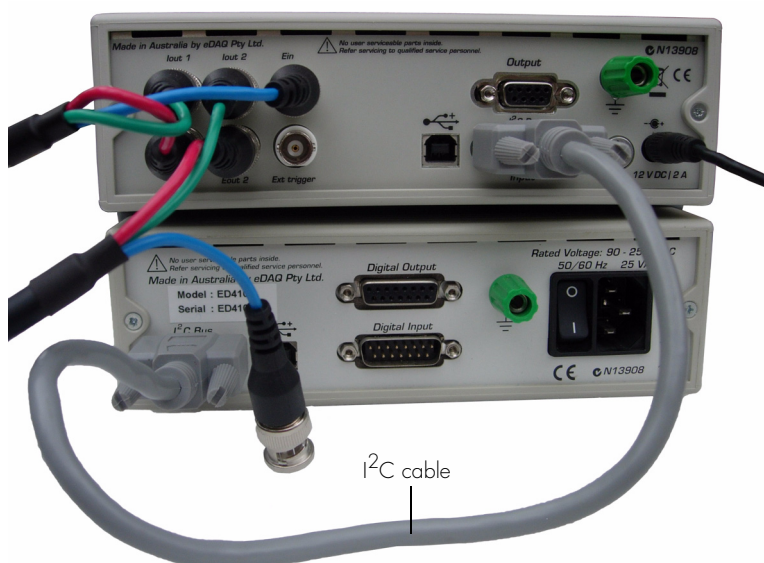
Omit this cable for  
single channel or  
4-electrode operation

**Figure 3-6**  
The Dual Picostat shown  
connected to an e-corder,  
back view.



Single channel or  
4-electrode operation

Dual channel or  
Bipotentiostat operation



I<sup>2</sup>C cable

With Chart software, Channel 2 (and Channel 4) are used to display the applied potential signals. The settings for these channels are controlled using the standard Input Amplifier dialog box, described in the *Chart Software Manual* which is installed as pdf files in the eDAQ Documentation folder on your computer hard disk.

When using EChem software, always connect e-corder Input 1 to the current signal (Picostat Iout), and e-corder Input 2 to the potential signal (the Picostat Eout 1).

## First Use

After you have installed the software, connected the e-corder and computer as described in the booklet that is supplied with the e-corder system, and connected the Dual Picostat as described above, you are ready to begin.

With the e-corder and Dual Picostat turned on, start the Chart software, the Dual Picostat Status indicator (green) should light. Then attach the 10 Mohm test resistor (supplied with the Dual Picostat) to the electrode leads so that the Working electrode lead is connected to one end of the resistor, and Reference and Auxiliary electrode leads to the other) and perform the test described in [Maintenance, page 37](#).

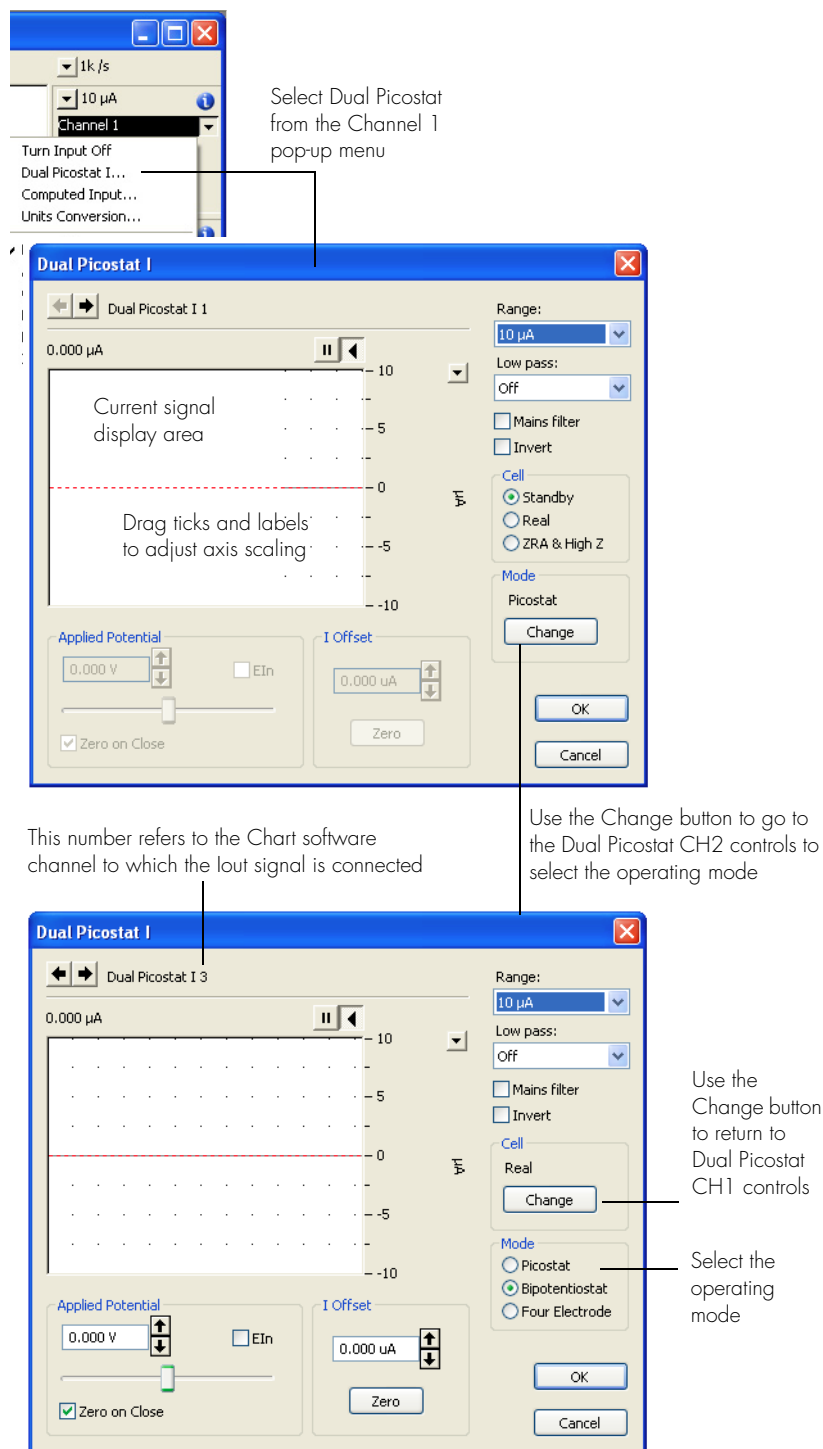
## Dual Picostat Control Window

With Chart software, the Dual Picostat Control window is accessed from the Dual Picostat command in the Channel Function pop-up menu. [Figure 3-7](#) shows the control window which sets the various current ranges and filtering options. With EChem software, the corresponding controls are shown in [Figure 3-8](#).

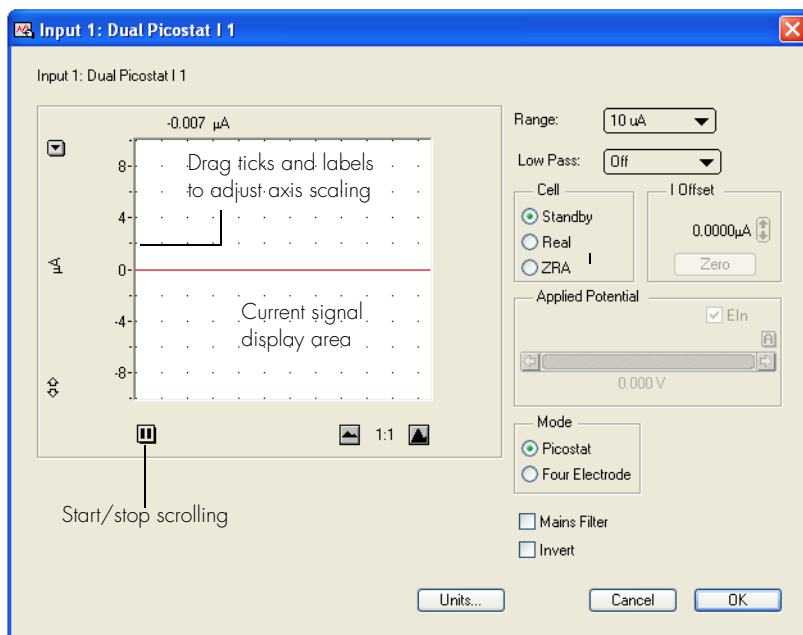
From the Chart software Channel 1 pop-up menu, select the 'Dual Picostat' command, which accesses the control window, [Figure 3-7](#).

The Dual Picostat control window allows you to preview the current signal without actually recording the signal to the computer hard disk. (If the menu says 'Input Amplifier' instead of 'Dual Picostat' then the software has not recognised the Dual Picostat. Exit the software, check all your connections and try again).

**Figure 3–7**  
Accessing the Dual  
Picostat controls with  
Chart software.



**Figure 3–8**  
Accessing the Dual  
Picostat controls with  
EChem software.



By default, the control window opens with the Dual Picostat in Standby mode, that is, with the electrodes isolated so that no current will flow through your sample. To connect to the electrodes you must select Real Cell mode.

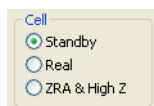
You will need to adjust the gain range to 200 nA to accommodate your signal amplitude, and to select the 10 Hz low-pass filter (and/or Mains Filter) to minimise high frequency noise on the signal — especially if you are working outside a Faraday cage.

You can now adjust the applied potential with the slider bar, or by entering the exact potential with text entry. The resulting current signal should obey Ohm's law:

$$I = E/R$$

so that with a 10 Mohm test resistor,  $R$ , in place, an applied potential,  $E$ , of 1 V should produce a current,  $I$ , of 100 nA, while other potential settings should produce corresponding currents.

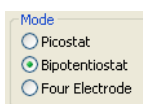
## Cell Control



The Dual Picostat can be in one of three cell modes, controlled by the Cell radio buttons:

- **Standby:** If Standby cell mode is selected the auxiliary and reference electrodes are isolated by an internal relay which effectively means that all the electrodes are at a 'floating' potential and that no current will be passed through your experimental solution. The electrodes will not be connected until the Picostat control dialog is closed and the Chart, Scope or EChem Start button is clicked to begin a scan. The Applied Potential control is disabled in this mode.
- **Real:** In Real cell mode the electrodes will be active and the Applied Potential slider control can be used to adjust the potential. When you close the control dialog (using EChem software) the Picostat will revert to Standby mode until the Start button is clicked to begin a scan. If you are using Chart software, the Picostat will remain in Real mode when the dialog is closed — this allows you to start and stop recording data while the electrodes remain active, which allows periodic recording of the signal from amperometric biosensors or *in vivo* electrodes without disturbing the environment around the electrodes.
- **ZRA & High Z:** In this cell mode the Dual Picostat current channel acts as a zero resistance ammeter (ZRA) while the associated potential channel acts as a high impedance voltmeter (High Z). The ZRA measures the current flowing between the working and auxiliary electrode lead wires, while the high impedance voltmeter monitors the potential between the working and reference electrode lead positions. Note that:
  - ZRA & High Z cell mode is only accessible when in Picostat operating mode;
  - it is not possible to adjust the potential while using this mode;
  - Dual Picostat CH2 can act as a second ZRA and high impedance voltmeter.

## Operating Mode Control




The Dual Picostat can perform as several potentiostat types, Picostat (normal three electrode potentiostat), bipotentiostat, or 4-electrode potentiostat:

- **Picostat:** Typically use only CH1 if you want to use the instrument as a single channel potentiostat, which produces a current signal at lout 1, and a potential signal at Eout 1. However both Dual Picostat channels are active in this mode, and if CH2 is also used the unit can be used with two separate samples (just as if two separate potentiostats were being employed). In this case there are two current signals (at lout 1 and lout 2), and two potential signals (at Eout 1 and Eout 2).
- **Bipotentiostat:** This mode employs two working, one (or two) reference and one auxiliary electrode. Both CH1 and CH2 of the Dual Picostat must be used. Always use the reference and auxiliary electrode of CH1. This mode is useful for *in vivo* neurotransmitter monitoring where it is required to measure dopamine release at two sites in a rodent brain. Note that there are two current signals (at lout 1 and lout 2) and two potential signals (at Eout 1 and Eout 2). If using just one reference electrode in the sample then connect the CH2 reference lead wire to the CH 2 auxiliary lead wire, otherwise the CH2 auxiliary (red) lead wire is left unconnected.
- **Four electrode:** Typically, the working electrode from CH1 is positioned on one side of a membrane, or interface, while the auxiliary and reference electrodes from CH1 are positioned on the other side. The reference electrode from second Dual Picostat channel, CH2, is positioned on the same side as the CH1 working electrode. Normally the distance between the two reference electrodes is kept to a minimum. The potential between the two reference electrodes can be controlled while the current flowing between the working and auxiliary electrodes of CH1 is monitored. This type of four electrode potentiostat is often used for ITES (Interface between Two immiscible Electrolyte Solutions) studies. It is also sometimes referred to as a 'four electrode voltage clamp' and used by electrophysiologists for studies of epithelial membrane tissue in Ussing chambers. Note that there is only one current signal (at lout 1), and one potential signal (at Eout 1). Also note that the working and auxiliary lead wires of CH2 are left unconnected.

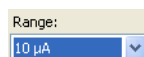
## Signal Display

The current signal is previewed scrolling across the 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 is lost.

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

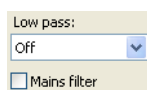
You can shift or stretch the vertical Amplitude axis to make the best use of the available display area. All changes to axis scaling are reflected in the Chart and Scope main window, and vice versa.

## Setting the Range

A small rectangular pop-up menu with a light beige background. It has a label 'Range:' at the top. Below the label is a blue dropdown menu showing '10 µA' with a small downward arrow on the right.

The Range pop-up menu lets you select the input current range or sensitivity. The Dual Picostat has ranges of 2 pA to 10 µA (if you require a system to monitor larger currents, use the eDAQ Potentiostat which has ranges up to 100 mA). 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 data will be truncated.

## Filtering

A small rectangular pop-up menu with a light beige background. It has a label 'Low pass:' at the top. Below the label is a blue dropdown menu showing 'Off' with a small downward arrow on the right. Below the dropdown is a checkbox labeled 'Mains filter' which is currently unchecked.

The Off setting gives the full bandwidth of the Dual Picostat which can be up to 16 kHz (but which may also be limited by the characteristics of your electrodes and sample solution). The Dual Picostat has an internal 10 Hz low-pass filter that can be turned on for removal of high frequency signals ('noise'). Other low pass filter settings are provided by the e-corder unit.

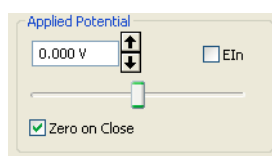
The 10 Hz filter setting is highly effective for the removal of mains hum (50 or 60 Hz interference) and, as a general rule, should be employed whenever possible. However, it should not be used for either pulse amperometric or voltammetric experiments where the pulses are shorter than 100 ms, or for voltammetric experiments where fast scan rates (greater than 100 mV/s) are used, or for other signals which are likely to exhibit fast rise or fall times — otherwise excessive smoothing of the signal may occur.

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

## Inverting the Signal ☐ Invert

The Invert checkbox allows you to invert the incoming current signal. It provides a simple way to redefine the direction (up or down) of an anodic (or cathodic) current signal. This control *does not* affect the actual direction of electron flow at the electrodes.

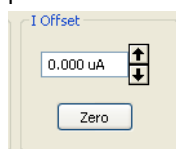
## Applied Potential



The applied potential slider control is only enabled in Real mode. It allows you to adjust a constant voltage (up to  $\pm 2.5$  V) applied to the electrodes. To change the value drag the slider bar to an appropriate potential, or use the text entry controls to enter an exact value. A more positive value will cause a more positive (more anodic) potential to be applied at the working electrode. If 'Zero on Close' is selected, the Applied Potential is set to zero when the window is closed.



If ☐ EIn, 'E in', is selected then any potential signal (up to  $\pm 10$  V) provided at the back panel E in connector, [Figure 3-3](#), will be added to that set with the Applied Potential control. For voltammetric experiments with EChem software make sure that E in is selected, and that the Applied Potential is set to zero.

## I Offset

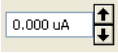


The I Offset control can be used to 'zero' a background current signal so that small peaks or transient signals can be more accurately determined in the presence of a large baseline current signal. While the



baseline current is being monitored click the  button. It may take a few seconds to zero the signal. Normally this will suffice to accurately zero the signal, and you can then choose a more sensitive current range setting to observe your signals. Use the  buttons to manually adjust the amount of offset if required (use Ctrl-click for fine adjustment).

Each Dual Picostat channel can have a different amount of offset applied to its current signal.

The amount of current offset is reported in the text box,  in which you can also enter a value directly.

If you need to zero the current signals after recording has started, then you can use the Zero All Inputs command in the Chart software Setup menu, [Figure 3–9](#).

**Figure 3–9**  
The Chart software Setup menu.



## Maintenance

The Dual Picostat will not require maintenance during daily operation. However, you should periodically check it for optimum results.

First set up the Dual Picostat and e-corder as outlined earlier in this Chapter. Connect the electrode cables connected, and attached to the 10 Mohm test resistor, as described in the section on [First Use](#), [page 30](#).

Open the Picostat Control window, [Figure 3–7](#) or [Figure 3–8](#). Adjust the gain range to 200 nA to accommodate your signal amplitude, and select the 10 Hz low-pass filter (and/or Mains Filter) to minimise high frequency noise on the signal — especially if you are working outside a Faraday cage.

You can now adjust the applied potential with the slider bar, or by entering the exact potential with text entry. The resulting current signal should obey Ohm's law:

$$I = E/R$$

so that with a 10 Mohm test resistor,  $R$ , in place, an applied potential,  $E$ , of 1 V should produce a current,  $I$ , of 100 nA, while other potential settings should produce corresponding currents.

If the current signal does not obey Ohm's law, then first recheck your connections of the Dual Picostat to the e-corder, [page 27](#). Then check with the second electrode cable. If the problem persists then it is possible that the Dual Picostat itself needs repair. If one electrode cable works well, but the second does not then it is likely you have a faulty cable, replacements can be obtained from your eDAQ supplier.

If these tests indicate that the Dual Picostat is working correctly, but you are still experiencing difficulties with your experiments, then you should check the electrodes you are using, the connections to them, and the design and condition of the reaction vessel, and any salt bridges that you are using.

# 4

## CHAPTER FOUR

# The QuadStat

This chapter describes how to connect and use your QuadStat.

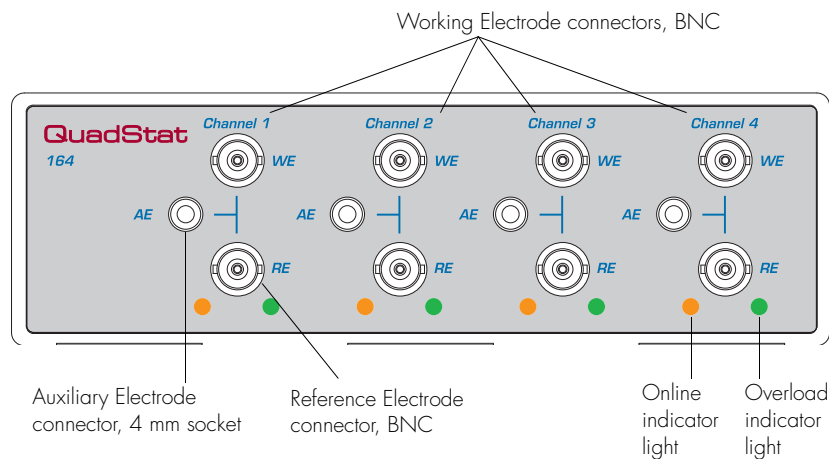
The QuadStat (EA164) is a four-channel, three-electrode potentiostat with gain ranges of 200 pA to 10 mA per channel). It can also be used as a: bipotentiostat (two working electrodes with a common reference and common auxiliary electrode), multiple working electrode potentiostat (three or four working electrodes with a common reference and auxiliary electrode); or as four-channel zero resistance ammeter (ZRA).

It is ideal for monitoring multiple amperometric sensors such as those used for: dissolved oxygen electrodes, nitric oxide electrodes, or enzymatic biosensors. It can also be used for experimentation with multiple small scale fuel cells (e.g. microbial fuel cells), small solar panels, and mini-batteries or other small power sources.

Note that when used with EChem software only a single QuadStat channel can be used. With Scope software one or two channels can be used. With Chart software, one to four QuadStat channels can be used.

**IMPORTANT:** This manual refers to QuadStat units, with serial numbers of 164-113 and later). Earlier QuadStat units did not have a ZRA operating mode, did not have all the gain range settings of the present version, and had a 1 Mohm dummy cell (instead of the present 100 kohm dummy cell).

**Figure 4-1**  
The QuadStat front panel



## The Front Panel

The front panel of the QuadStat is shown in [Figure 4-1](#).

### Electrode Connectors

The front panel of the QuadStat provides connections for the Working WE), Auxiliary (AE), and Reference (RE) electrodes. BNC connectors are used for the WE and RE leads. The shields of these connectors are driven to the same potential as the electrode.

The connector for the AE lead is a socket for a 4 mm pin.

### Electrode Cables

The QuadStat is supplied with appropriate electrode cables, with each lead terminated by an alligator clip which allows connection to a wide variety of electrodes. The leads are color-coded to indicate the type of electrode to which they should be attached ([Table 4-1](#)). The RE and WE leads are shielded to protect the signals from external interference. The shields are driven to the same potential as the electrode to minimize lead capacitance.

**Table 4-1**

Color-coding on the leads of the electrode cables.

<i>Color</i>	<i>Electrode</i>
Yellow	Reference
Green	Working
Red	Auxiliary

If two-electrode operation is required the auxiliary and reference electrode leads (red and yellow) can be attached to the single 'counter electrode'.

## The Online Indicators

Along the lower edge of the QuadStat front panel are a series Online indicators, [Figure 4-1](#). When lit, they indicate that the software (such as EChem, Chart or Scope) has located and initialised that QuadStat channel. If the light does not go on when the software is run, check that the QuadStat is properly connected. If there is still a problem, please refer to Appendix B [Troubleshooting, page 85](#).

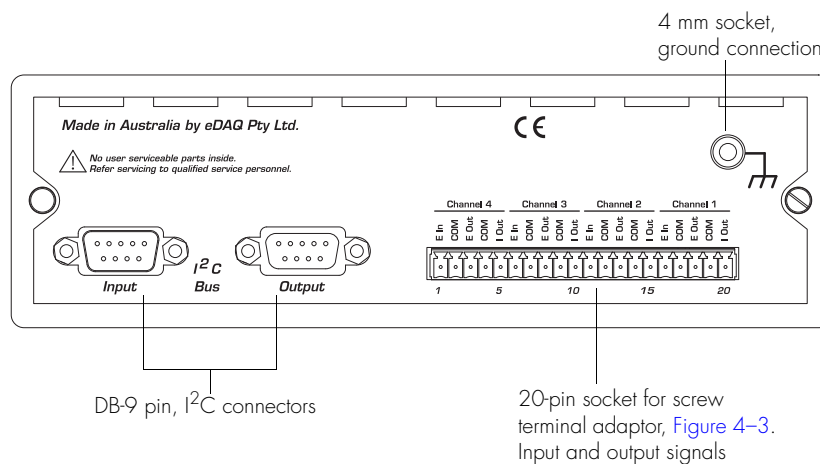
## The Overload Indicators

Also along the lower edge of the front panel are the Overload indicators, [Figure 4-1](#). When lit, these indicate either that the QuadStat has overloaded, which usually occurs because it has gone out of compliance because of an open circuit (such as an unconnected AE or faulty electrode), or the resistance is too high in the electrochemical cell. High resistances can be often be encountered when electrodes are fouled by the products of electrolysis reactions. The QuadStat tries to compensate by increasing the compliance potential (that is, the potential of the auxiliary electrode). If the compliance voltage exceeds specification, about 11 V, potential control of the cell is lost and drifting, or oscillation, of the signal can be seen. Any data collected during this period is unreliable and should be discarded.

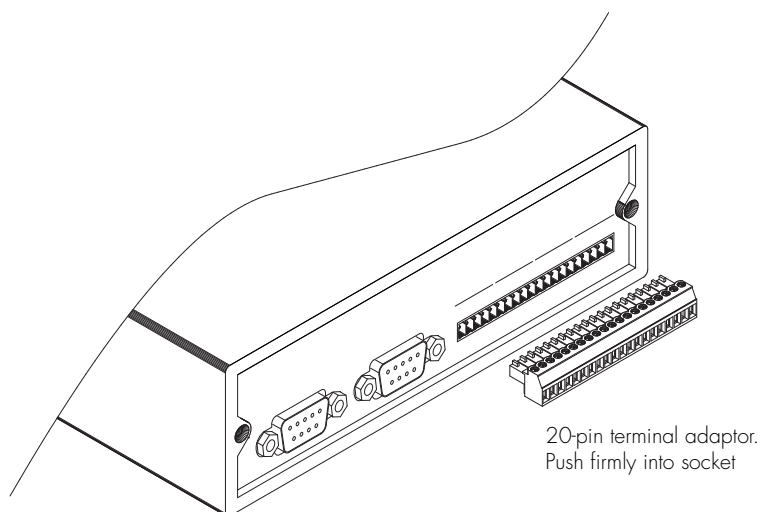
The QuadStat Overload indicators will remain lit once an overload has occurred — they will be reset once the scan has finished.

If an overload indicator comes on repeatedly, and your connections are good, then try bringing your electrodes closer together, and/or increasing electrolyte concentration, and/or modifying your experimental conditions to avoid fouling of the electrodes. Redesigning your electrochemical cell may be necessary. Normally electrochemical cells are designed to keep the reference and working electrodes very close together, however, when a potential overload occurs, you also

**Figure 4-2**  
The QuadStat back panel.



**Figure 4-3**  
The 20-pin screw terminal adaptor.



need to consider the distance between the auxiliary and working electrodes.

NOTE 1: The overload light may come on when a QuadStat channel is being used for a second, third, or fourth working electrode, and the corresponding reference and auxiliary electrode connectors are not being used, see [Using a Common Reference and Auxiliary, page 49](#). This does NOT indicate faulty operation.

NOTE 2: A potential overload is quite different from a current overload condition. A current overload is caused when the current signal exceeds the full scale limits of the sensitivity setting of the current channel. This is, in turn, due to a low resistance between the electrodes. In some circumstances a current overload can also cause the QuadStat overload indicators to light.

## The Back Panel

The back panel of the QuadStat is shown in [Figure 4-2](#).

### E Out, I Out and E In Connectors

The QuadStat is supplied with a 20 pin screw terminal adaptor, [Figure 4-3](#), which plugs into the 20 pin socket on the back panel. The pin positions are labelled I Out, E Out, E In, and COM, for each QuadStat channel (Channels 1 – 4).

The COM (common) pins are provided for connection to signal ground (black wires of the supplied coaxial cables, [Figure 4-5](#)). You can use any COM pin for the ground connection of any I Out, E Out, or E In signal.

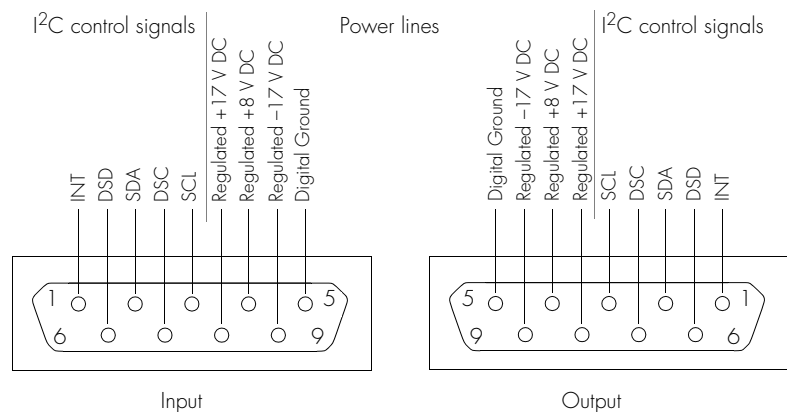
### I<sup>2</sup>C Connectors

The QuadStat back panel, [Figure 4-2](#), has two DB-9 pin 'I<sup>2</sup>C bus' connectors labelled Input and Output. The Input connector provides power to the QuadStat and carries the various control signals (for gain range and filter selection) to and from the e-corder connection. A cable is provided with the QuadStat for this purpose. The pin assignments are shown in [Figure 4-4](#).

**Figure 4–4**  
The I<sup>2</sup>C connectors.

**WARNING!**

The I<sup>2</sup>C connectors are for the power and control of **eDAQ Modular Potentiostats**, page 2, and should not be used for connection to any other device.



The Output connector can be used for the attachment of another QuadStat, or other eDAQ Amp.

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

## Grounding Connector

The QuadStat back panel, [Figure 4–2](#), has a 4 mm grounding socket. This enables connection of a Faraday cage (with the green grounding cable included with the QuadStat) the use of which can greatly diminish electrical noise.

The construction of the Faraday cage can range from a simple cardboard box covered with aluminium foil, in which the electrochemical cell is located, to a more sophisticated copper mesh enclosure or sheet-metal box. In all cases, it is essential that the Faraday cage be electrically grounded to act as an effective shield against electrical interference.

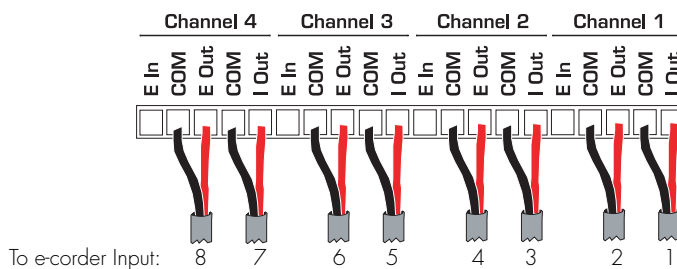
The QuadStat itself is grounded via its connection to the e-corder unit which is in turn earthed via the three pin mains power connector. It is also important that the power socket that you are using is well earthed.

You can try grounding the Faraday cage via its own connection to earth, or via the QuadStat ground cable — but *not by both* methods simultaneously. The purpose of this ground cable to the Faraday cage is to provide an easy means of grounding the cage — please note that it



**Figure 4-5**

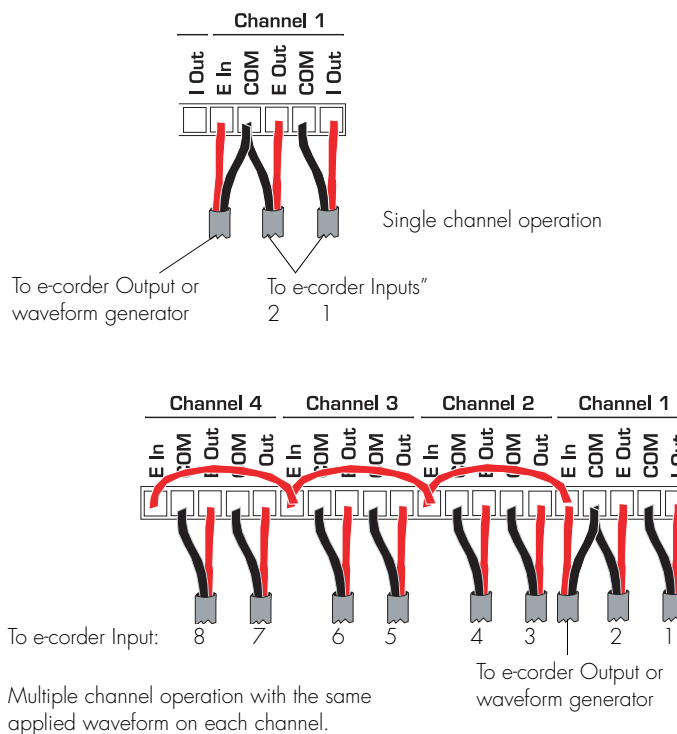
Signal connections from the terminal adaptor. Black colored wires are connected to COM pins.



For single channel operation use only Channel 1.  
For bipotentiostat operation use only Channel 1 and Channel 2.

**Figure 4-6**

Using the external inputs of the QuadStat. Note connection to E In is only required for potentials of more than  $\pm 2.5$  V, or for pulsed or ramped waveforms.



is not for grounding the QuadStat itself. If your Faraday cage is already earthed by its own ground connection then you should not use the QuadStat ground cable! Use of the QuadStat cable in this instance will provide a second pathway to earth which could result in a 'ground loop' which can actually increase signal interference!

The grounding connector is equivalent to the COM pins of the 20 pin terminal socket, [Figure 4-2](#).

## Connecting the QuadStat

Your QuadStat will have been supplied with an I<sup>2</sup>C cable (DB-9 pin connectors at either end), and nine cables with BNC connectors at one end and bare wires at the other.

First make sure that the e-corder is turned off. Then connect the I<sup>2</sup>C cable to the I<sup>2</sup>C connector on the back panel of the e-corder, and the other the other end to the I<sup>2</sup>C Input connector on the back panel of the QuadStat.

The QuadStat provides two signals per channel: the potential signal (E Out) indicating the potential difference between the working and reference electrodes; and the current signal (I Out) indicating the current flow between the working and auxiliary electrodes. Use the BNC cables, with bare wires at one end, to connect the terminal strip ([Figure 4-3](#)) as described in [Figure 4-2](#) and [Figure 4-5](#) or [Figure 4-6](#). Note there are several possibilities depending on how many of the QuadStat channels you wish to use and whether you require the use of the e-corder Output, or an external waveform generator.

For experiments where the working electrode potential is held constant (between  $\pm 2.5$  V) the E In pin positions are unused, [Figure 4-5](#). If the electrode potential required is greater than  $\pm 2.5$  V, or is to be pulsed or ramped during the experiment, then the E In input must be connected to a suitable external signal, such as from the Output of the e-corder, or a waveform generator, [Figure 4-6](#). If an e-corder is used, then normally

**Table 4-2**

QuadStat to e-corder BNC connections. See [Figure 4-7](#) for actual appearance.

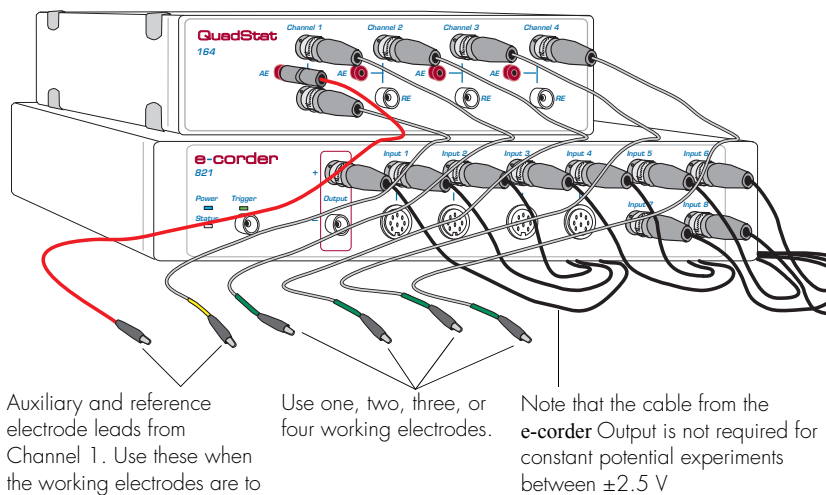
<i>QuadStat rear panel</i>	<i>e-corder front panel</i>
I Out (Ch 1, Ch 2, Ch 3, Ch 4)	Input 1, 3, 5, 7
E Out (Ch 1, Ch 2, Ch 3, Ch 4)*	Input 2, 4, 6, 8
E In †	Output +‡

\* It is not always necessary to monitor E Out depending on your experimental requirements.

† Connections to E In are only required if using an external waveform to control the applied potential.

‡ Use Output – to send a signal of reverse polarity to the QuadStat.

**Figure 4-7**  
The QuadStat shown connected to an e-corder, front view, using the connections described in [Table 4-2](#).



See also [Figure 4-8](#)

connect to Output +. 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 4-7](#).

If you need to reverse the polarity of the QuadStat, use 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.

By linking the E In positions with short wires you can control the potentials of all electrode potentials simultaneously, [Figure 4-6](#).

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

Each QuadStat channel uses two e-corder input channels during normal operation (for recording of the current and potential signals). The remainder of this chapter assumes that you have connected the current signal of QuadStat channel 1 to e-corder Input 1 and the potential signal to e-corder Input 2, and other channels as shown in [Table 4-2](#) and [Figure 4-5](#). (It is also possible, when using Chart or Scope

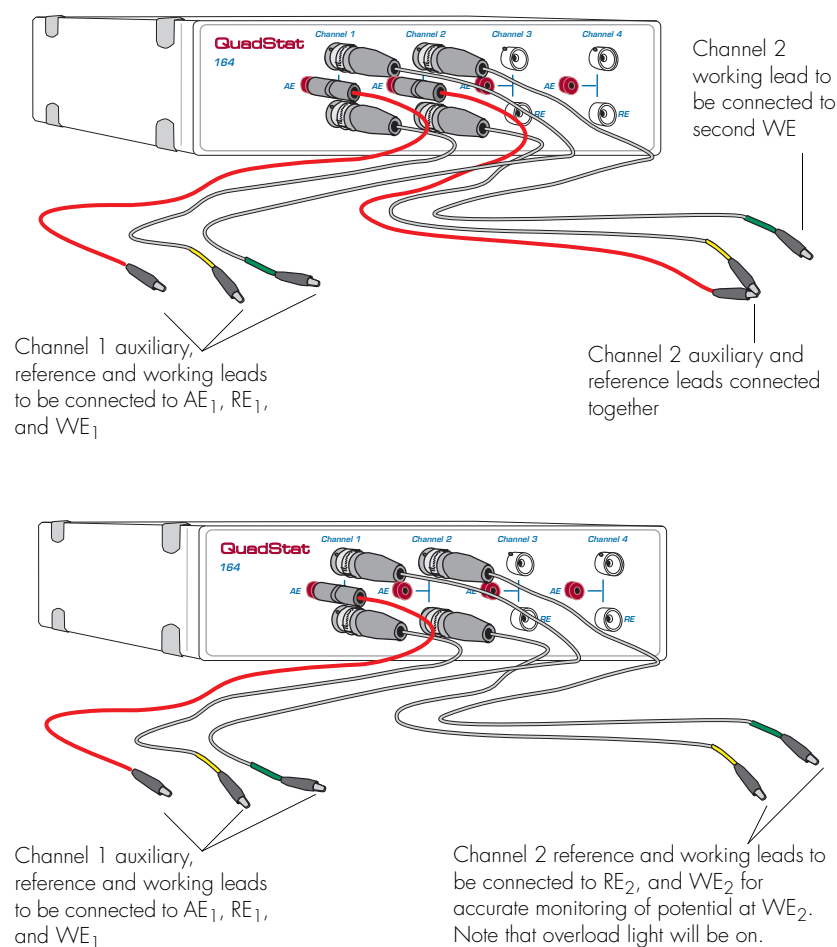
software, to connect the QuadStat to other e-corder input channels in which case the description that follows would change accordingly).

When using EChem software, e-corder Input 1 is always set to be the current signal (the I channel), and e-corder Input 2 is automatically set to be the potential signal (the E channel). Thus when using EChem software connect the QuadStat Channel 1 current signal (I Out) to e-corder Input1, and the QuadStat Channel 1 potential signal (E Out) to e-corder Input 2. Other QuadStat channels remain unconnected.

To record the applied potential signals (E Out) of a QuadStat with Chart and Scope software, first configure the settings of the standard

**Figure 4-8**

These alternative configurations are equivalent to a bipotentiostat.



Input Amplifier dialog box, described in the *Chart and Scope Software Manuals*.

## Using a Common Reference and Auxiliary

Often the QuadStat will be used with a single reference electrode, RE, and a single auxiliary electrode, AE<sub>1</sub>, (on QuadStat channel 1) with multiple working electrodes (WE<sub>1</sub>, WE<sub>2</sub>, WE<sub>3</sub>, WE<sub>4</sub>) on some, or all, of the other QuadStat channels. To monitor the applied potential signal for Channels 2–4 connect the unused AE and RE sockets together. You can use the extra AE and RE lead wires supplied with the QuadStat, connecting the alligator clips to one another, for this purpose, see [Figure 4–8](#).

If the unused AE and RE sockets on Channels 2, 3, or 4 on the front panel of QuadStat remain unconnected (as shown in [Figure 4–7](#)) then the QuadStat will still function correctly. However the corresponding potential signals at E Out<sub>2, 3, 4</sub> are not accurately monitored, and the overload lights on these channels will come on — but this does NOT indicate a fault condition. Only the overload light of Channel 1 (where the reference and auxiliary electrodes are connected) is now able to correctly indicate an overload.

When you use a common reference and auxiliary electrode with multiple working electrodes, AND use an external waveform at E In on Channel 1, this waveform can be effectively applied to all working working electrodes by linking the E In connectors together (as shown [Figure 4–6](#)). For example if a triangular waveform is applied at QuadStat Channel 1 then all the QuadStat channels will perform cyclic voltammetry. The triangular potential waveform can be offset at each separate working electrode by using the Applied Potential control, [page 55](#).

Finally, when you use a common reference and auxiliary electrode with multiple working electrodes, make sure that all the QuadStat channels are set to the same cell mode (Standby, Dummy or Real), [page 54](#). Recording signals with some channels set on Dummy, while others are on Real/Standby can cause incorrect current signals.

## Using Multiple References

When using the QuadStat with a single auxiliary electrode,  $AE_1$ , (on QuadStat channel 1) and multiple working electrodes ( $WE_1$ ,  $WE_2$ ,  $WE_3$ ,  $WE_4$ ) it is possible to use a separate reference electrode ( $RE_1$ ,  $RE_2$ ,  $RE_3$ ,  $RE_4$ ) ideally placed near the corresponding WE.

This allows the potential at each of  $WE_1$ ,  $WE_2$ ,  $WE_3$ , and  $WE_4$  to be accurately monitored. In the previous section, where the AE and RE connectors were linked together, the requested applied potential is being monitored. However when each RE is nearby to its corresponding WE, the actual potential at each WE is being measured. This configuration is especially recommended where the WE's are not equidistant to the AE and  $RE_1$ . Remember that QuadStat channels on which an AE is not connected will show an overload light — but in this case there is no fault condition.

## Using Multiple References and Auxiliaries

The QuadStat can also be used to conduct experiments in different reaction vessels. In this case you should use a set of three electrodes (working, reference, auxiliary) in each reaction vessel. Each QuadStat channel behaves as a separate potentiostat. Independent signals can be applied at each QuadStat channel E in connector, if desired, so that different experiments can be run in each reaction vessel.

Please note that the use of multiple auxiliary electrodes in the same reaction vessel will almost certainly lead to unpredictable effects and is not recommended!

## First Use

After you have installed the software, connected the e-corder and computer, and connected the QuadStat as described above, you are ready to begin.

When the e-corder is turned on, and Chart software started, the QuadStat Online indicators (green), [Figure 4–1 on page 40](#), should light for every channel connected.

From the Chart software Channel 1 pop-up menu, select the 'QuadStat' command, (also on Chart software Channels 3, 5, 7 if all four working electrodes are being used) which accesses the QuadStat control window, [Figure 4-9](#), or [Figure 4-10](#).

The QuadStat control window allows you to preview the current signal without actually recording the signal to the computer hard disk. (If the menu says 'Input Amplifier' instead of 'QuadStat' then the software has not recognised the QuadStat. Exit the software, check all your connections and try again).

By default, the control window opens with the QuadStat in Standby mode, that is with the reference and working electrodes isolated so that no current will flow through your electrodes. To connect to the QuadStat electrode lead wires you must select Real mode. When you click Cancel or OK the QuadStat will revert to Standby mode until recording is started. For now, select the Dummy cell mode which connects an internal 100 kohm resistor between the electrodes.

You will need to adjust the gain range to 50  $\mu\text{A}$  to accommodate your signal amplitude. If the signal is noisy select the 10 Hz low-pass filter (and/or Mains Filter).

You can now adjust the applied potential with the slider bar, or by entering the exact potential with text entry. The resulting current signal should obey Ohm's law:

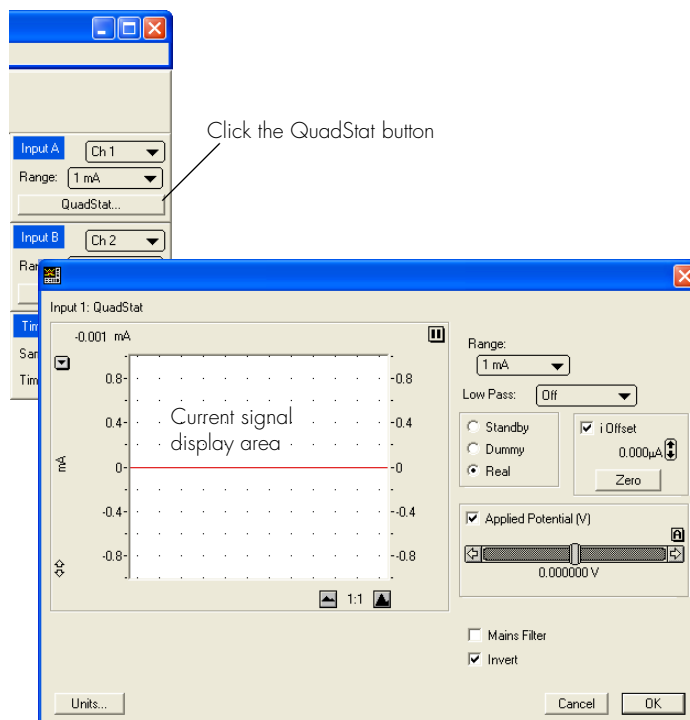
$$I = E/R$$

so that with a 100 kohm test resistance, R, an applied potential, E, of 1 V should produce a current, I, of 10  $\mu\text{A}$ , while other potential settings should produce corresponding currents. If this is so, then your QuadStat is working correctly and you can proceed to your experiment.

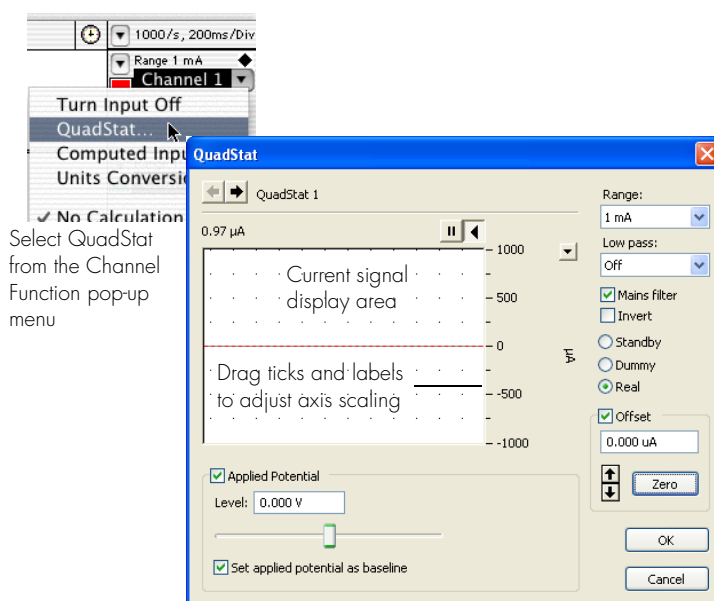
## QuadStat Control Window

With Chart software, the QuadStat Control window is accessed from the QuadStat command in the Channel Function pop-up menu. [Figure 4-9](#) shows the control window which contains the various current ranges and filtering options.

**Figure 4–9**  
Accessing the QuadStat controls with Chart software.



**Figure 4–10**  
Accessing the QuadStat controls with Scope software.






With Scope software, the corresponding controls are shown in [Figure 4-10](#).

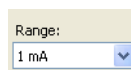
## Signal Display

The current signal is previewed scrolling across the 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 is lost.

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

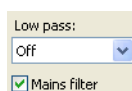
You can shift or stretch the vertical Amplitude axis to make the best use of the available display area. All changes to axis scaling are reflected in the Chart and Scope main window, and vice versa.

## Setting the Range



The Range pop-up menu lets you select the input current range or sensitivity. The QuadStat has ranges of 20 pA to 10 mA. 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 data will be truncated and therefore lost.

## Filtering



The QuadStat has low-pass filter settings (10 kHz to 1 Hz) for removal of high frequency signals ('noise'). The Off setting gives the full bandwidth of the QuadStat which can be up to 16 kHz (but which may also be limited by the characteristics of your electrodes and sample solution). The more sensitive gain ranges will have a smaller maximum bandwidth.

The 10 Hz (and smaller) filter settings, are highly effective for the removal of mains hum (50 or 60 Hz interference) and, as a general rule, should be employed whenever possible. However, low pass filters should be used with care when performing pulse amperometric or voltammetric experiments, or for other signals which are likely to exhibit fast rise or fall times. For example, if you are using the 10 Hz filter, then applied potential pulses should be longer than 100 ms, and scan rates

less than about 100 mV/s — otherwise excessive smoothing of the resulting current signal may occur.

When you use the Chart and Scope software there is a Mains Filter checkbox. When 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. Note that the mains filter is NOT a simple notch filter, and it can remove 50 or 60 Hz interference even if it is not a pure sinusoidal waveform. However, the mains filter does take about a second to 'learn' the pattern of the interference so that you will need to record for longer than this for it to take full effect. The mains filter can even be employed for experiments in which there are sudden potential jumps.

## Inverting the Signal

The Invert checkbox allows you to invert the incoming current signal. It provides a simple way to redefine the directions (up or down) of an anodic (or cathodic) current signal. This control *does not* affect the direction of current flow at the electrodes.

## Cell Control

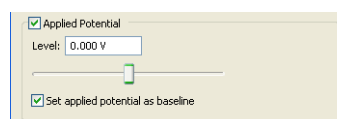
The QuadStat can be in one of three operating modes, controlled by the Cell radio buttons:

- **Standby:** If Standby mode is selected the auxiliary and reference electrodes are isolated by an internal relay which effectively means that all the electrodes are at a 'floating' potential and that no current will be passed through your experimental solution. The electrodes will not be connected until the QuadStat control dialog is closed and the Chart, Scope or EChem Start button is clicked to begin a scan. The Applied Potential control is disabled in this mode.
- **Dummy:** When Dummy mode is selected the QuadStat channel is connected to the internal 100 kohm dummy cell. You can then use the Applied Potential slider control to vary the voltage applied to the dummy cell. The QuadStat will remain connected to the dummy cell even when the QuadStat Control window is closed and Chart, Scope or EChem is recording. This is useful for testing the QuadStat.
- **Real:** In Real mode the electrodes will be active and the Applied Potential slider control can be used to adjust the potential. When

you close the control dialog (using EChem software) the QuadStat will revert to Standby mode until the Start button is clicked to begin a scan. If you are using Chart or Scope software, the QuadStat will remain in Real mode when the dialog is closed — this allows you to start and stop recording data while the electrodes remain active, which allows periodic recording of the signal from amperometric biosensors or *in vivo* electrodes without disturbing the environment around the electrodes.

Please note that when using multiple working electrodes in the same reaction vessel, with a single reference and single auxiliary electrode, make sure that all the QuadStat channels are set to the same value (Standby, Dummy or Real). Running an experiment with some channels set on Dummy, while others are on Real/Standby can cause incorrect current signals.

## Applied Potential

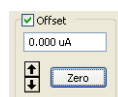


The Applied Potential control offsets the voltage (up to  $\pm 2.5$  V) applied to either the dummy cell or external working electrodes. This potential,  $E_{off}$ , is remembered by the QuadStat and will be applied when you start a scan if the ☒ Set applied potential as baseline checkbox is ticked. Note that each QuadStat channel may have a different value for  $E_{off}$ .


This potential value will be summed with any external input waveform,  $E_{in}$ , from the E In connection on the terminal strip connector on the QuadStat back panel. The total potential,  $E_{total} = E_{off} + E_{in}$ , must be less than  $\pm 10$  V.

If you wish to use the QuadStat exclusively with an external voltage input (for example with EChem software) then make sure the ☒ Applied Potential checkbox is NOT ticked. This will ensure that  $E_{off}$  is zero.

## Zero Offset



The current Offset control can be used to 'zero' a background current signal so that small peaks or transient signals can be more accurately determined in the presence of a large baseline current signal. First tick the ☒ Offset checkbox. Then, while the baseline current is being monitored click the  button. It may take a few seconds to zero

the signal. Normally this will suffice to accurately zero the signal, and you can then choose a more sensitive current range setting to observe your signals. Use the  buttons to manually adjust the amount of offset if required (use Ctrl-click for fine adjustment).

Each QuadStat channel can have a different amount of offset applied to its current signal. The maximum amount of offset available is dependent on the selected gain range (see QuadStat specifications for [Current Measurement and Control, page 96](#)).

The amount of current offset is reported in the text box, .

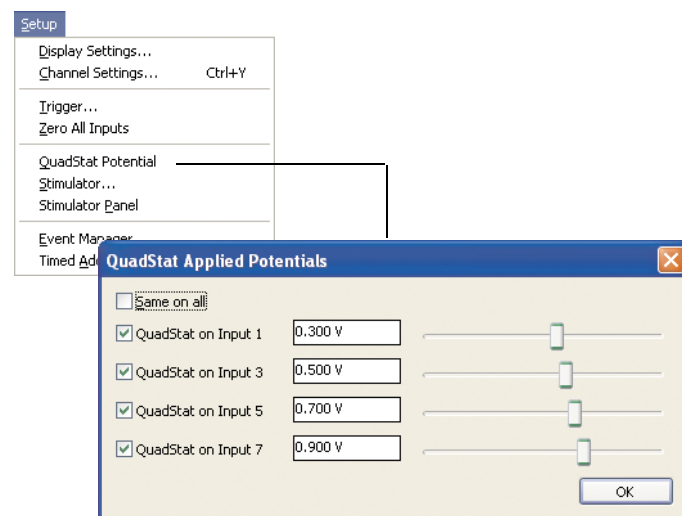
If you need to zero the current signals after recording has started, then you can use the Zero All Inputs command in the Chart software Setup menu, [Figure 4-11](#).

## QuadStat Potential Window

When using the QuadStat with Chart software it is possible to alter the applied potentials during recording.

Access the QuadStat Applied Potentials window, [Figure 4-11](#), from the Chart Setup menu. Working electrode potentials can then be adjusted independently on each connected QuadStat channel. This is equivalent to adjusting the potentials using the Applied Potential controls,

**Figure 4-11**  
The QuadStat Applied Potential controls with Chart software.



page 55, in the QuadStat Control Window, [Figure 4–9 on page 52](#), except that adjustments can be made while recording is in progress.

It is also possible to automate the QuadStat Potential controls with the Macro feature of Chart so that the working electrode potentials can be altered at predetermined times. Please consult the *Chart Software Manual*, installed in the eDAQ Documentation folder on your hard disk, for more information about the use of Macros.

## Maintenance

Your QuadStat will not require maintenance during daily operation. However, you should periodically check the QuadStat for optimum results by applying a known potential,  $E$ , to the Dummy Cell. Open the QuadStat Control window, [Figure 4–9](#) or [Figure 4–10](#), and go to Dummy Cell mode. The current signal,  $I$ , should obey Ohm's law:

$$I = E/R$$

where  $R$  is the resistance, and is  $10^5$  ohm for the dummy cell, and  $E$  is the applied potential. Thus a signal of  $10\text{ }\mu\text{A}$  should be obtained when a potential of  $1\text{ V}$  is applied. Try several different potentials and make sure an appropriate current signal is observed. Repeat this procedure on the different QuadStat channels. If this test produces the expected results then your QuadStat is likely to be functioning correctly.

Also periodically repeat this procedure with the electrode cables connected, and attached to a test resistor, as described in the section on [First Use, page 50](#). If the current signal does not obey Ohm's law, then first recheck your connections of the QuadStat to the e-corder, [page 46](#). If the problem persists then it is possible that the electrode leads or the QuadStat itself has become damaged.

If these tests indicate that the QuadStat is working correctly, but you are still experiencing difficulties with your experiments, then you should now check the electrodes you are using, the connections to them, and the design and condition of the reaction vessel, and any salt bridges that you are using.



# 5

## C H A P T E R F I V E

# 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 as pdf files in the eDAQ Documentation folder on your computer hard disk.

Also discussed is the use of the Potentiostat when configured as a galvanostat.

Additional experiments such as current-sampled staircase linear sweep, differential pulse, normal pulse, square wave and 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

The Potentiostat, Picostat, and Quadstat apply a potential difference across a pair of working and reference electrodes whilst monitoring the current flow between the working and auxiliary electrodes.

## NOTE

*The QuadStat also has internal potential adjustment of  $\pm 2$  V. When the e-corder output is connected to a QuadStat channel 'E in', this value is summed with the value set by the Chart or Scope Stimulator controls, to a maximum of  $\pm 10$  V.*

This potential difference is determined by a 'command voltage' which is sent from the e-corder output to the 'E In' connector of the, Dual Picostat, [Figure 3–3, on page 25](#), Potentiostat, [Figure 2–4, on page 7](#) or QuadStat, [Figure 4–2, on page 42](#). The QuadStat and Dual Picostat can also generate a constant command voltage internally. Chart and Scope software control the e-corder output via the 'Stimulator' controls in their Setup menus, as do the Techniques in the EChem software. For a full description of these controls, and the waveforms that can be produced, you should consult the *Chart Software, Scope Software and Manuals*.

You can use your e-corder and Potentiostat, Picostat, or QuadStat with Chart and Scope software to perform the following experiments:

- Linear Scan techniques, [page 61](#) — use Scope software with the Potentiostat to provide a potential ramp (up to 500 V/s) and to subtract charging current contributions. The bandwidth of the Picostat and QuadStat are sufficient for scan rates up to about 10 V/s
- Chronoamperometry [page 62](#) & [page 64](#), Amperometry, Constant Potential Electrolysis, [page 70](#), — monitor the current signal at fixed potentials
- Chronocoulometry [page 65](#) — monitor and integrate the current signal at a fixed potentials
- Chronopotentiometry [page 67](#), Constant Current Electrolysis [page 71](#) — monitor the potential signal when the Potentiostat is used as a galvanostat to maintain a constant current at the working electrode. Note that the Picostat and QuadStat cannot be used as galvanostats
- Monitoring of amperometric sensors [page 72](#), including dissolved oxygen and nitric oxide electrodes.
- zero resistance ammeter, or high impedance voltmeter, with the Potentiostat, [page 13](#).



# Linear Scan Techniques

Linear sweep or cyclic voltammetry are usually best performed with EChem software, or with the Chart software and an EA175 Waveform Generator.

## Fast Cyclic (or Linear Sweep) Voltammetry

EChem software can perform Fast Cyclic Voltammetry (FCV) up to about 400 V/s but by this point you are reaching the limits of the bandwidth of the e-corder itself and your results may show some distortion. Also note that at very high gains the bandwidth of the potentiostat you are using is lessened and can also distort the voltammogram at fast sweep rates. For experiments involving fast sweep rates use the full bandwidth of the potentiostat (that is, if possible, do not use the low-pass filters) or the response of the current signal may be modified by the low-pass filter time response characteristics.

When performing FCV, a large background charging current is often recorded. This can be many times larger than the signal you are 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 EChem '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 sweep settings) as the substrate solution to be effective. See the *EChem 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 65](#)).

Single, double and even multi-step chronoamperometry, can be performed with Chart 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 or Dual Picostat, or QuadStat (that is, if possible, do not use the low-pass filters) or the response of the current signal may be modified by the low-pass filter time response characteristics.

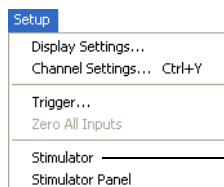
If you are using a QuadStat, then most constant potential experiments between  $\pm 2.5$  V are done by adjusting the internal QuadStat [Applied Potential, page 55](#). If you require applied potentials greater than  $\pm 2.5$  V, or pulsed waveforms, then you can use the Chart Stimulator controls as described below.

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 Picostat or Potentiostat, or QuadStat, 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 5-1](#).

To adjust the command voltage you will first need to select the range by adjusting the Output Range control in the Stimulator controls [Figure 5-2](#). 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:

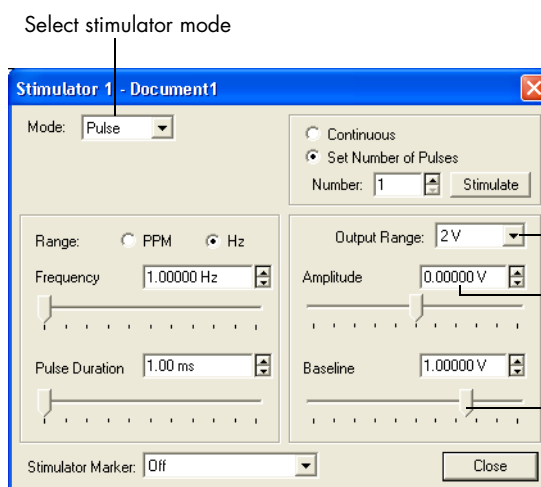
1. set the **Stimulator** to Pulse mode;
2. set the **Pulse Amplitude** to zero volts;
3. set the **Baseline** control to the desired voltage;
4. adjust the current input range to an appropriate value, [page 15](#) and [page 35](#);

**Figure 5-1**  
Chart Setup menu



Use the Stimulator command to access the Stimulator (applied potential waveform) controls, [Figure 5-2](#) & [Figure 5-3](#)

**Figure 5-2**  
Chart Stimulator  
(waveform output)  
controls

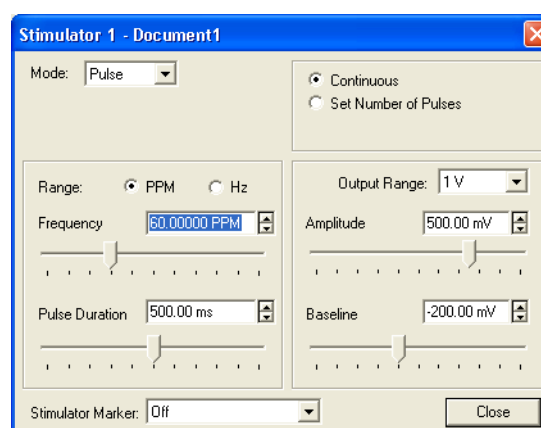


Select output range

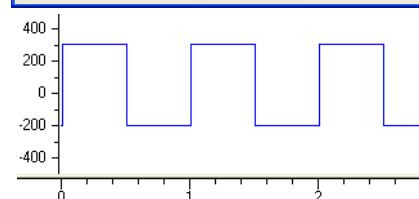
Enter exact values as text.  
Use zero amplitude for  
constant output

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

**Figure 5-3**  
Stimulator controls for  
multiple step  
chronoamperometry



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

5. 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
6. 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  $\mu$ 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 5–3](#). Note that the pulse amplitudes are added to any value set by the Baseline control.

For long term voltage steps 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.

## 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 or Picostat (that is, do not use the low-pass filters) or the response of the current signal may be dominated by the low-pass filter time response characteristics. The base potential is adjusted with the Output Voltage... command (Setup menu) shown in [Figure 5–4](#).

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 5–4](#), after a period of 10  $\mu$ 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.

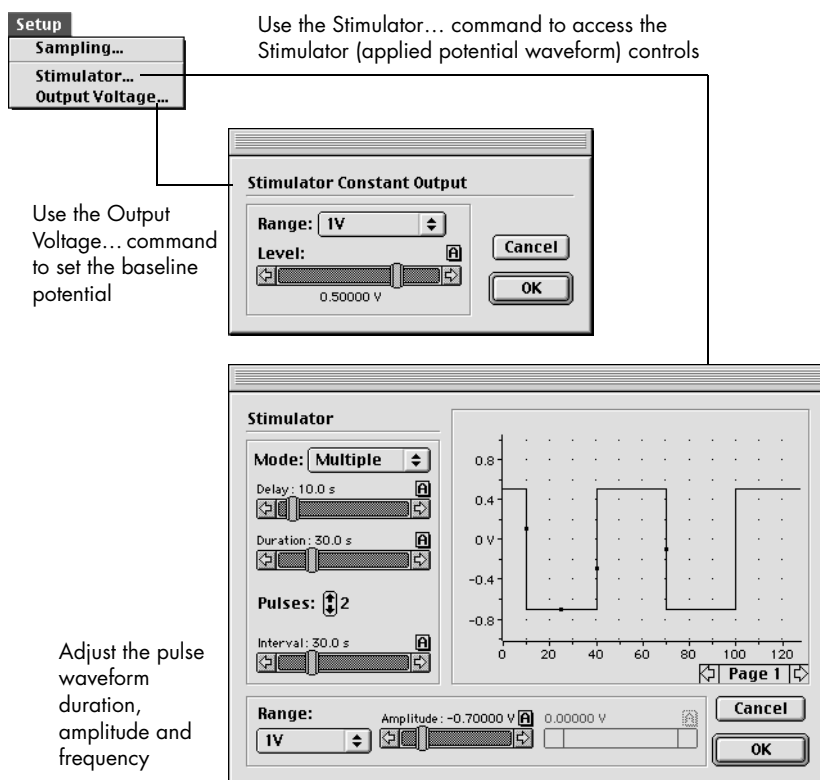
The experiment is usually first done on a blank solution containing only electrolyte, followed by a sample solution containing the substrate. The data is collected on separate pages in Scope and the 'blank' data subtracted with the Set Background command. You can then copy and paste the scan to a spreadsheet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

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

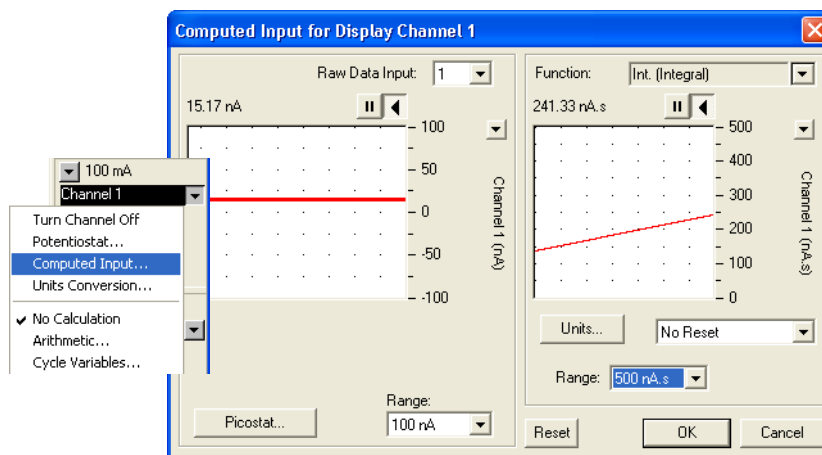
## Chronocoulometry

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 62](#), can also be used for chronocoulometry.

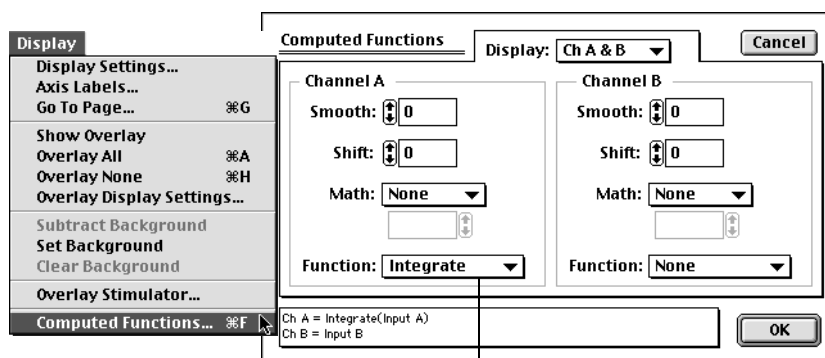
**Figure 5-4**  
Scope controls for multiple step chronoamperometry.



**Figure 5-5**  
Online integration of the current signal using Chart Computed Input integration



**Figure 5-6**  
Integration of the current signal using Scope Computed Functions.



Select Integrate in the Function menu

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 5-5](#). 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 5–6](#). 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.

## Chronopotentiometry

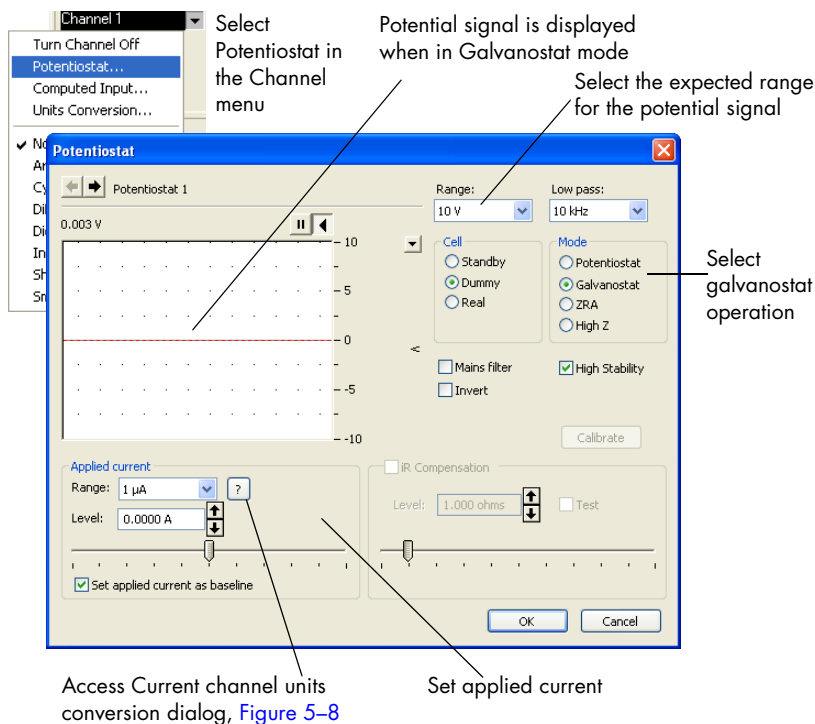
Chronopotentiometry requires that a constant current be maintained between the working and auxiliary (counter) electrodes. The potential at the working electrode is monitored. For many systems the potential will remain approximately constant until the electroactive species is consumed, after which there will be a sudden change in the potential.

For this type of experiment it will be necessary to run the Potentiostat in Galvanostat mode with Chart or Scope software. For correct operation make sure that the 'CH 1 (I)' cable of the Potentiostat is connected to Input 1 of the e-corder, and the 'CH2 (E)' cable of the Potentiostat is connected to Input 2.

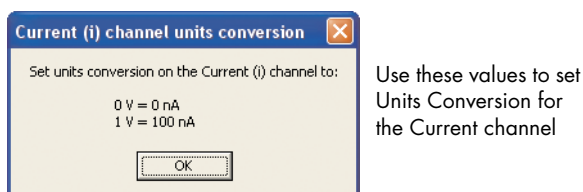
Note that galvanostat mode is not available with the Picostat or QuadStat.

When using the Potentiostat as a galvanostat, the applied current can be set within ranges up to 100 mA. Select the smallest range setting consistent with your desired current to ensure maximum accuracy. For example, if a current of 750  $\mu\text{A}$  is required then a range setting of 1 mA (1000  $\mu\text{A}$ ) should be used, and then exact current value adjusted accordingly.

**Figure 5–7**  
Setting up the  
Potentiostat as a  
Galvanostat.



**Figure 5–8**  
The Current channel units  
conversion dialog box.



The current values used should ensure that the resulting potentials do not exceed  $\pm 10$  V (the maximum limit of the Potentiostat) — highly resistive loads can easily produce large potentials, even with small currents.

Remember that when setting zero, or very small, currents there is always a small amount of offset (error) in the system. If you are trying to measure the potential of a system under zero current conditions then it would generally be more accurate to use a zero current potentiometer (or pH meter) than a galvanostat, or use the Potentiostat in High Z mode, [page 14](#).



## Chart software

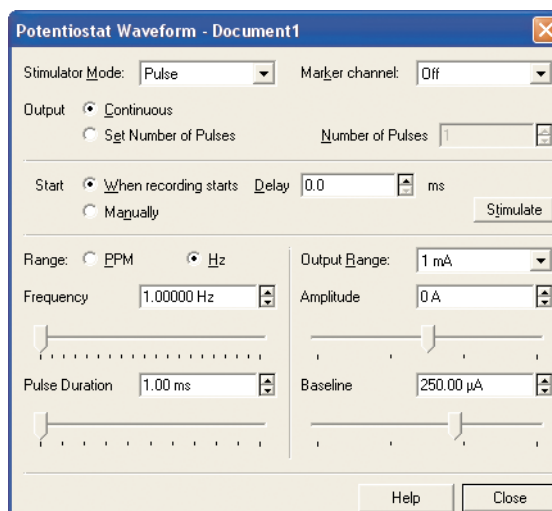
To switch to the Galvanostat mode of operation, choose the Potentiostat command in the Channel Function pop-up menu to open the control window, and turn on the Galvanostat and Dummy radio buttons, [Figure 5–7](#).

When in Galvanostat mode, the current and potential signals will be reversed from normal (potentiostatic) operation. That is, the 'I Out' cable will be carrying the potential signal (which will now appear on Channel 1) and the 'E Out' cable the current signal (which will now appear on Channel 2). You will need to configure the Units Conversion of Channel 2 so as to ensure that the current signal is recorded in the correct current units, [Figure 5–8](#).

When the Chart Stimulator command (Setup menu) is selected it accesses the Stimulator dialog box which, when using galvanostat mode, allows a baseline and various current waveforms to be configured, [Figure 5–9](#).

Always select an appropriate current range for your system. While it is possible to set an applied current of up to 100 mA, the Potentiostat/Galvanostat cannot supply a potential much greater than  $\pm 10$  V. Even relatively small applied currents, with a highly resistive load, may require potentials in excess of this. If in doubt, start with a small test current and observe the resulting potential.

**Figure 5–9**  
The Chart Stimulator dialog box when the Potentiostat is in Galvanostat mode



When in Galvanostat mode the Stimulator controls are used to adjust the applied current. Compare with Potentiostat mode where the Stimulator is used to adjust applied potential, [Figure 5–3](#).

With highly resistive loads, using a large range setting with a small (close to zero) current can produce unexpected large potential signals because of small offsets in the current control circuitry. If you require very small currents always use a small range setting as well, this will ensure a minimum offset error. For example, to apply a current of only 25 nA use the 100 nA range setting for best results.

## Controlled Potential Electrolysis

This technique is essentially the same as [Chronoamperometry with Chart, page 62](#), and is also known as amperometry. A constant potential is applied at the working electrode which is sufficient to oxidize or reduce the substrate. As the electrolysis continues the substrate is eventually completely consumed and the current will decrease to a residual value. Both the potential and the current flow can be monitored with Chart software in the same way as for chronoamperometry. The integrated current (that is, the total charge transferred) can also be determined by integrating the current signal (see [Chronocoulometry, page 65](#)) and this can be used to determine the extent of reaction.

If the solution is being stirred during the procedure then eventually all the substrate can be oxidized, or reduced, to a new material and the technique may be referred to as electrosynthesis rather than chronoamperometry.

If the reaction causes the formation of a polymer film on the electrode (for example pyrrole to polypyrrole, or aniline to polyaniline) then the technique may be referred to as electropolymerisation.

To effect the electrolysis of a substrate a potential should be chosen which is at least 50 – 200 mV more oxidising (or reducing) than the half-wave potential in order to ensure complete reaction. It is usually important to separate the auxiliary (counter) electrode from the main body of the electrochemical cell by means of a salt bridge. This is to prevent the reduction products formed in the region of the auxiliary electrode reacting with the products of oxidation at the working electrode (or, if reducing the substrate, to prevent the products of oxidation forming around the auxiliary electrode from reacting with the reduction products at the working electrode). The working electrode is usually constructed to have relatively large surface area so that a

greater amount of material can be electrolyzed in a small time — thus a plate or gauze electrode is usually chosen, although reticulated vitreous carbon (RVC) which has an open pore foam structure, is also a popular choice.

The conversion of large amounts of substrate (> 100 mg) in reasonable times requires relatively high currents to be passed through the cell. Thus the Potentiostat (maximum current 100 mA) is usually more suitable than the Picostat (< 100 nA) or QuadStat (< 1 mA/channel) for electrosynthesis. The production of organic compounds in highly resistive non-aqueous solvents often requires the addition of large amounts of electrolyte to sustain the current flow (and which can lead to difficulties in later separation of the product). For studies using water, or other protic solvent, the addition of a small quantity of acid (or base) can greatly increase the conductivity of the solution, without having to add large amounts of electrolyte.

## Controlled Current Electrolysis

These techniques are essentially the same as [Chronopotentiometry, page 67](#), and require the use of a galvanostat. The Potentiostat, when operated in Galvanostat mode, is suitable for experiments where currents less than 100 mA and potentials less than 10 V are anticipated. (Please note that the Picostat, or QuadStat, cannot be operated as a galvanostat). A constant current is applied between the working and auxiliary electrodes and the potential at the working electrode is monitored as the substrate is oxidized or reduced. As the electrolysis continues the substrate is eventually completely consumed and the potential will alter as the galvanostat tries to maintain a constant current. Both the potential and the current flow can be monitored with Chart software. The integrated current (that is, the total charge transferred) can also be determined which can be used to determine the extent of reaction.

Electrosynthesis and electropolymerization reactions are often more quickly carried out under constant current conditions than under constant potential conditions, but there is a greater risk of side reactions occurring as the potential changes. Ideally there should be no competing redox reactions at nearby potentials.

A sudden change in the potential usually indicates when the substrate is fully consumed.

It is important to limit the current so that the potential stays within the compliance voltage (10 V) of the Potentiostat/Galvanostat, at least until the reaction of interest is complete. Electropolymerization reactions can be problematic as a non-conducting polymer film adhering to the working electrode will increase its electrical resistance causing the Potentiostat to eventually go out of compliance and experience a potential overload.

## Amperometric Sensors

Amperometric sensors require a potentiostat of appropriate gain range. You will also need to determine whether the sensor is of a two or three electrode configuration. The Potentiostat (up to 100 nA), Picostat (up to 100 nA), and QuadStat (up to 1 mA) can be used in three electrode mode (working, reference and auxiliary electrode), or in two electrode mode (working and counter electrodes) by connecting the reference and auxiliary leads of the electrode cable together. You will also need to determine the correct 'polarising voltage' to use with your sensor. The 'polarising voltage' is the potential at which the Potentiostat, Picostat, or QuadStat, applies at the working electrode.

Note that the QuadStat can be employed with up to four sensors, each at a different potential, all using the same reference and auxiliary electrode. The QuadStat also has the ability to zero (or offset) a background current signal which can be of use when trying to amplify small peaks on a large background signal, see [Zero Offset, page 55](#).

Normally Chart software (rather than Scope) is the better choice of recording software for these types of sensor. The Units Conversion feature of Chart can be used to perform two point calibration of the signal in the units of your choice, which is satisfactory for most uses — see the *Chart Software Manual* for more information.

If multiple point, or non—linear, calibration is required then the Chart Multiple Point Calibration extension can be used with up to twelve calibration points.

If you are performing a flow injection analysis, or similar experiment, then the Chart Flow Analysis extension can be used to determine peak areas, prepare a calibration graph, and apply the calibration results to determine the amount of material in the unknown sample peaks. PowerChrom software can also be used with the QuadStat and e-corder hardware as a electrochemical detector for HPLC experiments.

You can download the extensions and accompanying documentation from the eDAQ web site at [www.eDAQ.com](http://www.eDAQ.com).

If the experiment requires a differential pulse methodology then the MultiPulse Amperometry techniques of the EChem software can be used for various pulse amperometric experiments. See the *EChem Software Manual* for more details.

## Biosensors

Biosensors are often amperometric sensors and so can be used with the Picostat, QuadStat, or Potentiostat which can then be referred to as 'biosensor meters'. The first step is to establish the polarising voltage and current measurement requirements of your sensor.

The QuadStat also has the ability to zero (or offset) a background current signal which can be of use when trying to amplify small peaks on a large background signal, see [Zero Offset, page 55](#).

Many biosensors provide a linear signal response over a range of concentrations but deviations occur over wider concentration ranges. The Chart software Multiple Point Calibration extension can be used to extend the useful range over which a biosensor can be used by compensating for a non-linear signal response.

If your biosensor only develops small currents (typically less than a microampere) it may be suitable for use with a two electrode system (working and counter electrode). In this case a low cost eDAQ Biosensor isoPod (EA352 or EPU352) might be a suitable alternative to a three electrode potentiostat.

## Microdialysis Sensor

The Sycopel™ Microdialysis Biosensor is available in several configurations for both in vivo or in vitro detection of analytes including

glutamate, glucose, glycerol, ascorbate and acetylcholine. The Picostat and QuadStat have suitable current ranges for use with this sensor, and Chart software would normally be used to record data.

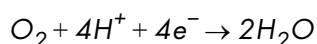
The QuadStat also has the ability to zero (or offset) a background current signal which can be of use when trying to amplify small peaks on a large background signal, see [Zero Offset, page 55](#).

The Chart software Multiple Point Calibration extension can be used to extend the useful range of metabolite concentrations over which this sensor can be used by compensating for non-linear signal response.

## Dissolved Oxygen (dO<sub>2</sub>) Sensors

Usually a polarographic (Clark) oxygen sensor electrode and meter are purchased from the same manufacturer to ensure matching sensitivity. The Potentiostat, QuadStat, Picostat, dO<sub>2</sub> isoPod, and Biosenor isoPod, can be used as alternatives to standard meters as they have adjustable sensitivity and can be matched to a wide range of current signal requirements. In addition you can use them to alter the polarising voltage to 'fine tune' your sensor.

Most polarographic oxygen electrodes comprise a gold or platinum cathodic working electrode (to which the working electrode lead is attached) and a silver anodic counter electrode (to which the reference and auxiliary electrode leads are attached). Usually a reducing polarising voltage (applied potential) of between -0.7 to -0.8 V is required at the working electrode to effect the reaction:



The exact potential can be varied to suit the sample being measured and to minimise side reactions. A smaller potential reduces sensitivity but is usually more selective for oxygen.

Most oxygen sensors exhibit a slow response time and will take at least several seconds to stabilise. Thus you would normally employ a 1 – 10 Hz filter setting on the Potentiostat, QuadStat, Picostat or isoPod, in order to minimise any electrical noise. The Mains Filter, where available, setting can also be employed to further reduce mains hum. Sampling rates of about 1 /s are usually optimal.

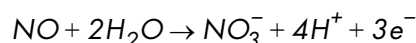
The QuadStat and isoPods have the ability to zero (or offset) a background current signal which can be of use when trying to amplify small peaks on a large background signal, see [Zero Offset, page 55](#).

If you intend to monitor oxygen concentrations over a wide range then you can calibrate the electrode at several concentrations and use the Chart software Multiple Point Calibration extension to compensate for a non-linear electrode response. You can download the extension from [www.edaq.com](http://www.edaq.com).

## Nitric Oxide (NO) Sensors

Most polarographic nitric oxide sensors can be used with the Picostat, QuadStat, or Nitric Oxide isoPod which are suitable for detecting the small currents expected from the low concentrations of nitric oxide found in natural biological systems.

Most commercial nitric oxide sensors comprise a carbon fibre anodic working electrode (to which the working electrode lead is attached) and a cathodic counter electrode (to which the reference and auxiliary electrode leads are attached). Usually an oxidising polarising voltage (applied potential) of up to +1.0 V is required at the working electrode, to effect the reaction:



The exact potential can be varied to suit the type of electrode (many electrodes have special catalytic surface coatings), the sample being measured, and to minimise side reactions. At potentials smaller than +0.7 V, the current signal is usually dependent on the polarising voltage, but between +0.7 and +1.0 V the current signal tends to plateau as the reaction is limited by the rate at which nitric oxide can diffuse to the anode surface. At potentials greater than +1.0 V the hydrolysis of water gradually becomes the dominant source of the current signal.

Most nitric oxide sensors exhibit a slow response time and will take at least several seconds to stabilise. Thus you would normally employ a 1 – 10 Hz filter setting in order to minimize any electrical noise. The Mains Filter setting, where available, can also be employed to further reduce mains hum. Sampling rates of about 1 /s are usually optimal.

It is recommended in most cases to calibrate the electrode at several nitric oxide concentrations covering your range of interest. Use the Chart software Multiple Point Calibration extension which can be downloaded from the eDAQ web site at [www.edaq.com](http://www.edaq.com).

The QuadStat and isoPod have the ability to zero (or offset) a background current signal which can be of use when trying to amplify small peaks on a large background signal, see [Zero Offset, page 55](#).



# A

## A P P E N D I X A

# Technical Aspects

This appendix describes various technical aspects of potentiostat construction — however, you do not need to understand this material to use the Potentiostat, Dual Picostat or QuadStat. Please note that this information is not intended as a service manual and that user modification of the equipment voids your rights under warranty.

The Potentiostat, Dual Picostat, QuadStat and other eDAQ Amps have been designed to be used with an e-corder system. All internal functions (gain ranges, filters, real/standby/dummy cell selection) of the Potentiostat, Picostat or QuadStat are controlled from the e-corder by sending information on a special communications connection called the 'I<sup>2</sup>C bus' which also supplies DC power.

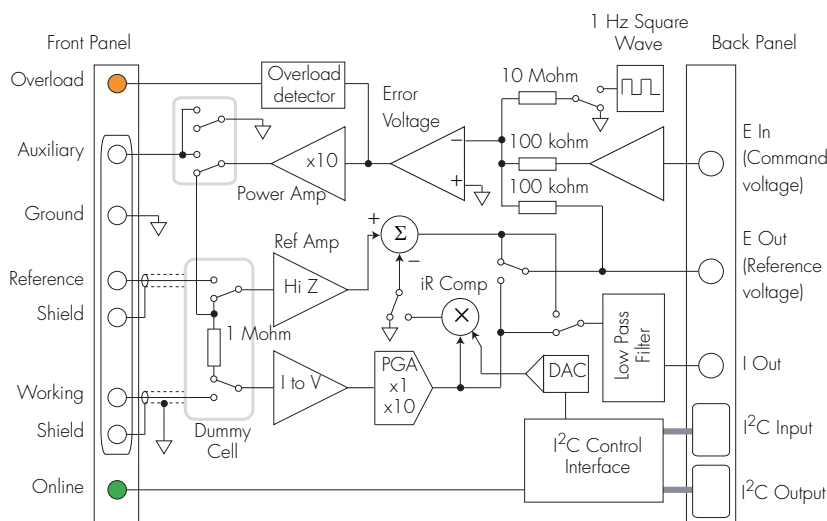
## Potentiostat

The Potentiostat is designed to function as a:

- potentiostat (Chart, Scope, or EChem software)
- galvanostat (Chart or Scope software)
- ZRA, zero resistance ammeter, (Chart or Scope software) or
- high impedance voltmeter (Chart or Scope software).

The mode of operation is under software control. A block diagram of the Potentiostat circuitry is shown in [Figure A-1](#).

**Figure A-1**  
Block diagram of the  
Potentiostat.



The Potentiostat is fitted with an internal 1 Mohm dummy cell for use in potentiostat or galvanostat mode. The dummy cell is physically connected whenever the 'Real cell' mode is not being used.

The Potentiostat has gain ranges at decade intervals from 1 nA/V to 10 mA/V, provided by an I to V convertor and PGA. The maximum current output signal (I Out) is 10 V at any gain setting. Secondary amplification of the current signal is done by the e-corder. The gain and secondary amplification are set by the single 'range' menu in the software which offers a combined total of 21 current range settings from  $\pm 20$  nA to  $\pm 100$  mA in 1:2:5 steps, [page 92](#).

The Potentiostat uses an error amplifier to compare the difference in potential between the reference electrode and the excitation potential set using the analog output of the e-corder. The output of this amplifier provides an error signal the feed back circuit tries to zero.

The coaxial shields of the reference and working electrode lead wires (which carry a high impedance signal) are maintained at the same potential as the lead wire itself to minimise electrical interference, and reduce lead capacitance.

The Potentiostat is powered by regulated +17 V, -17 V and +8 V DC lines from the e-corder, see [Figure 2-3 on page 7](#). The use of DC power allows its use inside Faraday cages.

# Dual Picostat

The Dual Picostat is designed to function as a:

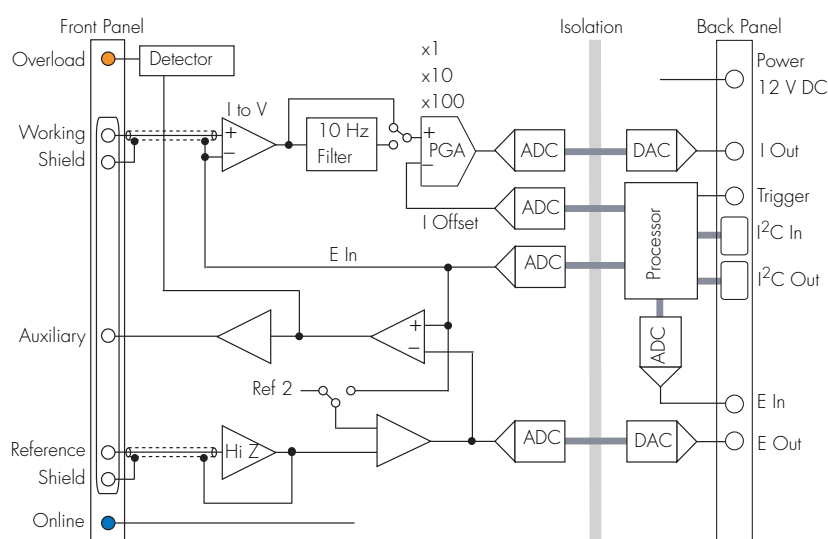
- potentiostat (Chart, Scope, or EChem software)
- bipotentiostat (Chart software)
- four electrode potentiostat (Chart, Scope or EChem software)
- ZRA, zero resistance ammeter, (Chart or Scope software) or
- high impedance voltmeter (Chart or Scope software).

The mode of operation is under software control. A block diagram of its construction is shown in [Figure A-2](#).

An error voltage is generated from the difference between the voltage on the Reference electrode and the excitation (command) voltage set by the e-corder unit. This difference or error voltage drives a high gain power amplifier (VCVS – voltage controlled voltage source) the output of which is applied to the Auxiliary electrode in such a way as to minimise the error voltage.

The Dual Picostat, and the electrochemical cell to which it is connected, forms a composite feedback control system in which the value of the

**Figure A-2**  
Block diagram of the  
Dual Picostat. Only  
Channel 1 is shown.



Auxiliary potential is controlled in such a way as to make the Reference voltage equal to the desired Command voltage. The current flow in the Working electrode is the quantity being measured.

When the Dual Picostat is in Standby mode the Working and Reference electrodes are disconnected by an internal relay. This prevents unwanted current flowing through your electrochemical cell.

An overload condition is indicated when the error voltage exceeds a small non-zero value. This indicates that the system could not establish or sustain a balanced state. The Overload light will be reset when scanning is stopped or when the Picostat controls are next accessed.

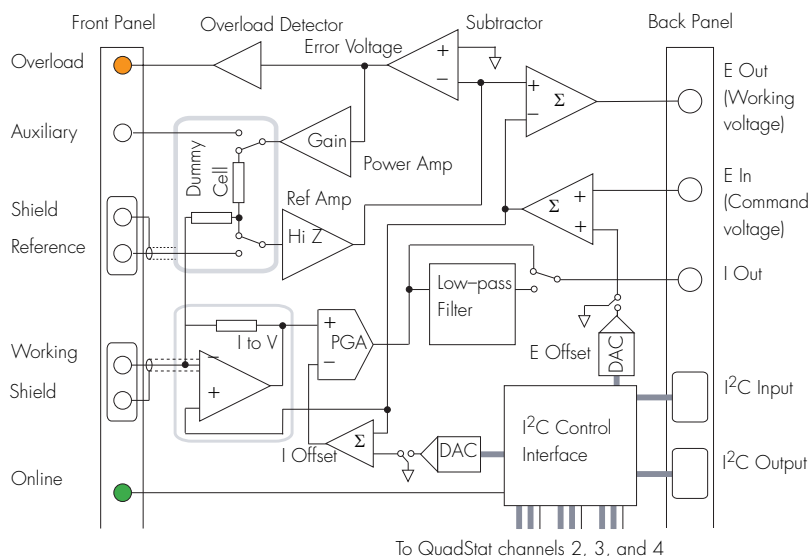
The reference voltage is measured by a high input impedance amplifier which provides a low impedance output to the e-corder unit.

The voltage at the working electrode is effectively held at a virtual ground potential (zero volts) by a voltage-to-current convertor, the output of which reads directly in current units and is also applied to the e-corder unit. The Dual Picostat has three gain ranges of 100 pA/V, 1 nA/V, and 10 nA/V. The maximum current output signal (I Out) is 10 V at any gain setting. Secondary amplification of the current signal is done by the e-corder. The gain and secondary amplification are set by the single 'range' menu in the software which offers a combined total of eleven current range settings from  $\pm 10$  pA to  $\pm 100$  nA in 1:2:5 steps.

The coaxial shields of the reference and working electrode lead wires (which carry a high impedance signal) are maintained at the same potential as the lead wire itself to minimise electrical interference, and reduce lead capacitance.

The Dual Picostat is powered by a +12 V DC source which allows its use inside Faraday cages.

**Figure A-3**  
Block diagram of the  
QuadStat, Channel 1.



## QuadStat

The QuadStat comprises four subunits which function in potentiostat mode only — that is the QuadStat cannot be used as a galvanostat. A block diagram of the QuadStat construction is shown in [Figure A-3](#).

Within the QuadStat an 'error voltage' is generated from the difference between the voltage on the Reference electrode and ground (0 V). This error voltage drives a high gain amplifier (VCVS – voltage controlled voltage source) the output of which is applied to the Counter electrode (CE) in such a way as to minimise the error voltage.

The QuadStat, and the electrochemical cell to which it is connected, form a composite feedback control system in which the value of the Counter Electrode (CE) voltage is controlled in such a way as to make the Reference voltage, with respect to the Working electrode, equal to the desired Command voltage. The current flow (I) in the Working Electrode (WE) is the quantity being measured. A DAC provides a means to offset (zero) this current signal so that small current signal variations can be more accurately determined.

When the QuadStat is in Standby mode the CE and RE are disconnected from the cell and connected to an internal dummy cell. This prevents current flowing through the electrochemical cell and allows a simple test of circuit operations.

A steady state, or transient, overload condition is indicated when the error voltage exceeds a small non-zero value. This indicates that the system could not establish or sustain a balanced state. The Overload light will be reset when scanning is stopped or when the QuadStat controls are next accessed.

The RE voltage is measured by a high input impedance amplifier whose output is summed with the WE potential to provide a low impedance output to the e-corder unit proportional to the actual potential difference between the RE and WE.

The WE is held at a virtual potential by a current-to-voltage convertor. The different WE's can be individually adjusted (that is the virtual potential can be adjusted) over a range of  $\pm 2.5$  V with the internal E Offset DAC. In particular, note that this E Offset adjustment is added to any signal applied at E In so that the final potential at the WE is the sum of these two values

The current signal each working electrode is measured by a separate current-to-voltage convertor. The signal can be zeroed by means of an offset current — this is useful in situations where a large 'background current' is present, as might occur in a flow injection analysis experiment, or during in vivo neurotransmitter monitoring.

Internally the QuadStat has gain ranges at decade intervals from 100 pA/V to 1 mA/V for the high current model) provided by an I to V convertor and PGA. The maximum current output signal (I Out) is 10 V at any gain setting. Secondary amplification of the current signal is done by the e-corder. The gain and secondary amplification are set by the single 'range' menu in the software which offers a combined total of twenty one current range settings from  $\pm 200$  pA to  $\pm 10$  mA in 2:5:10 steps.

The coaxial shields of the RE and WE lead wires (which carry a high impedance signal) are maintained at the same potential as the lead wire to minimise electrical interference, and reduce lead capacitance.

The QuadStat is powered is powered by regulated +17 V, -17 V and +8 V DC lines from the e-corder, see [Figure 4-4 on page 44](#). The use of DC power allows its use inside Faraday cages.

Multiple-electrode potentiostat designs, for sensor work, have been reported in "Development of a computer controlled multichannel potentiostat for applications with flowing solution analysis", Tang Fang, Michael McGrath, Dermot Diamond, and Malcolm R. Smyth, *Analytica Chimica Acta*, **305**, 347-358, 1995.





# B

## A P P E N D I X B

# Troubleshooting

This appendix describes some problems that may arise when using the Potentiostat, Dual Picostat or QuadStat. There is also help in your *e-corder Manual* and *Software Manuals*, placed on your computer hard disc by the *eDAQ Software Installer CD*.

In many cases, a problem can be fixed by turning the e-corder and computer off, checking connections, and starting again. Also try performing the maintenance checks on [page 20](#), [page 37](#), and [page 57](#). If you cannot correct the problem, then please contact your eDAQ representative.

### ***The 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 Potentiostat, Dual Picostat, or QuadStat 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 9](#), [page 28](#), and [page 46](#).
- 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 9](#), [page 28](#), and [page 46](#).

- Try using the Potentiostat, Dual Picostat, or QuadStat with another e-corder (if available). If the Online indicator fails to light on the second e-corder, the Potentiostat, Picostat, or QuadStat, 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 'Potentiostat', 'Dual Picostat', or 'QuadStat' 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 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 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 Dual Picostat or QuadStat Overload indicators may come on when the software is started — this is normal. The Overload indicator light should go out when you open the Dual Picostat or QuadStat controls, or start recording.
- See [page 6](#), [page 24](#) or [page 41](#) for more details.

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

- Increase the full scale current range setting.
- Make sure each electrode is connected to the correct lead wire, [page 5](#), [page 23](#), or [page 40](#).
- Make sure the electrodes (and the alligator clips connecting them to the lead wires) 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 Potentiostat, page 9](#), or [Connecting the Dual Picostat, page 27](#), or [Connecting the QuadStat, page 46](#).
- 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 (yellow) lead wire, [page 5](#), [page 23](#) or [page 40](#). Connection of the reference electrode to either the working or auxiliary leads can also 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 on page 15](#), [page 36](#), or [page 54](#).

***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 (yellow) 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, for better results, 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 ( $\text{Hg}/\text{Hg}_2\text{Cl}_2$ ) 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 the 10 Hz (or lower) filter of to record your results, see [page 15](#), [page 35](#), and [page 53](#). 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 alligator clips where they are subject to mechanical wear. Use a multimeter to ensure that

the clips are still in good electrical contact with the correct pins in the input connector of the electrode cable, see [Figure 2–2 on page 4](#), [Figure 3–3 on page 25](#), and [Figure 4–2 on page 42](#). Also check that the shield pins have not shorted to any of the alligator clips.

- 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 (and even the Potentiostat or Picostat) should be enclosed by a Faraday cage. A Faraday cage (or other electrical shielding) is almost always required when measure small currents (which is usual with the Picostat).
- 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).



# C

## A P P E N D I X C

# Specifications

## Potentiostat

### Power Amp

Compliance voltage:	> 10 V
Output current:	±100 mA maximum
Current limit:	±200 mA
Slew rate:	3 V/μs

### Electrometer

Input resistance:	$10^{13}$ ohm
Input bias current:	< 1 pA @ 25°C
Input voltage:	±10 V maximum
Output voltage:	±10 V maximum
Output offset voltage:	±0.8 V maximum
Output offset drift:	±0.8 μV/°C
Gain accuracy:	0.1%

## Current Measurement and Control

Current Range Setting	Potentiostat gain ( $\mu\text{A}/\text{V}$ )
$\pm 100, 50, 20 \text{ mA}$	10 000
$\pm 10, 5, 2 \text{ mA}$	1 000
$\pm 1 \text{ mA}, 500, 200 \mu\text{A}$	100
$\pm 100, 50, 20 \mu\text{A}$	10
$\pm 10, 5, 2 \mu\text{A}$	1
$\pm 1 \mu\text{A}, 500, 200 \text{ nA}$	0.1
$\pm 100, 50, 20 \text{ nA}$	0.01

Maximum current signal: 10 V

Low pass filters: 10 000, 1000, 100, 10 Hz  
3rd order Bessel

e-corder filter settings: 10 kHz to 1 Hz in 10:5:2 steps

Gain accuracy: 0.2% at ranges 1 mA or smaller  
1% at ranges 10 mA or larger

Drift with temperature:  $\pm 0.3 \text{ mV}/^\circ\text{C}$

## Control Loop

Voltage offset error:  $\pm 1 \text{ mV}$

Voltage gain error: 0.1%

Bandwidth (unity loop gain): 100 kHz (EA163) 16 kHz (EA161)  
@  $-90^\circ$  lag  
160 Hz (high stability mode, @  $-90^\circ$  lag)

Ramp follower error:  $\pm 1 \text{ mV}$  @ 100 V/s  
 $\pm 1 \text{ mV}$  @ 1 V/s (high stability mode)



## iR Compensation

Current Range Setting	Compensation Range
$\pm 100, 50, 20$ mA	0 – 10 ohm
$\pm 10, 5, 2$ mA	0 – 100 ohm
$\pm 1$ mA, 500, 200 $\mu$ A	0 – 1 kohm
$\pm 100, 50, 20$ $\mu$ A	0 – 10 kohm
$\pm 10, 5, 2$ $\mu$ A	0 – 100 kohm
$\pm 1$ $\mu$ A, 500, 200 nA	0 – 1 Mohm
$\pm 100, 50, 20$ nA	0 – 10 Mohm

## Control Port

I<sup>2</sup>C input and output: Male and female DB-9 pin connectors.  
Provides control and power to the Potentiostat.

Power requirements:  $\pm 17$  V DC  
+8 V DC  
25 mA typical  
2 W

## Physical Configuration

Dimensions (h  $\times$  w  $\times$  d): 50  $\times$  76  $\times$  260 mm  
1.96  $\times$  3.0  $\times$  10.2 inches

Weight: 0.8 kg (1.8 lb)

Operating conditions: 0 – 35°C  
0 – 90% humidity (non-condensing)

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

# Dual Picostat

## Electrometer & Power Amp

Compliance voltage: > 13 V  
Control voltage: <  $\pm 2.5$  V  
Applied potential: <  $\pm 10$  V (with external input)  
Output current: <  $\pm 10$   $\mu$ A  
Input impedance:  $10^{13}$  ohm || 1 pF  
Input bias current: < 250 fA @ 25°C (60 fA typical)

## Current Measurement and Control

Current Range Setting	Gain (nA/V)	Current Offset	Bandwidth (Hz)
$\pm 10, 5, 2$ $\mu$ A	10 000	$\pm 100$ $\mu$ A	> 5000
$\pm 1$ $\mu$ A, 500, 200 nA	1 000	$\pm 10$ $\mu$ A	> 5000
$\pm 100, 50, 20$ nA	100	$\pm 1$ $\mu$ A	> 5000
$\pm 10, 5, 2$ nA	10	$\pm 100$ nA	> 500
$\pm 1$ nA, 500, 200 pA	1	$\pm 10$ nA	> 500
$\pm 100, 50, 20$ pA	0.1	$\pm 1$ nA	> 500
$\pm 10, 5, 2$ pA	0.01	$\pm 100$ pA	> 500

DC current error: <  $\pm 1\%$  FS on ranges of 10 pA – 1 nA  
<  $\pm 0.5\%$  FS on ranges of 2 nA – 10  $\mu$ A

Filter setting: 10 Hz low-pass. 3rd order Bessel

e-corder filter settings: 2 kHz to 1 Hz in 10:5:2 steps

Drift with temperature: < 20  $\mu$ V/°C

## Control Port

I<sup>2</sup>C input and output: Male and female DB-9 pin connectors.  
Control of the Dual Picostat.

## Power

Requirements: 12 V DC, 2 A  
+8 V DC, ~ 20 mA

## Physical Configuration

Dimensions (h × w × d): 50 × 76 × 260 mm  
1.96 × 3.0 × 10.2 inches

Weight: 0.8 kg (1.8 lb)

Operating conditions: 0 – 35°C  
0 – 90% humidity (non-condensing)

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

# QuadStat

## Current Measurement and Control

Current Range Setting	Gain (nA/V)	Current Offset	Bandwidth (kHz)
$\pm 10, 5, 2, 1 \text{ mA}$ $\pm 500, 200 \text{ }\mu\text{A}$	1 000 000	$\pm 10 \text{ mA}$	>10
$\pm 100, 50, 20 \text{ }\mu\text{A}$	100 000	$\pm 1 \text{ mA}$	>10
$\pm 10, 5, 2 \text{ }\mu\text{A}$	10 000	$\pm 100 \text{ }\mu\text{A}$	>10
$\pm 1 \text{ }\mu\text{A}, 500, 200 \text{ nA}$	1 000	$\pm 10 \text{ }\mu\text{A}$	>10
$\pm 100, 50, 20 \text{ nA}$	100	$\pm 1 \text{ }\mu\text{A}$	>10
$\pm 10, 5, 2 \text{ nA}$	10	$\pm 100 \text{ nA}$	>1
$\pm 1 \text{ nA}, 500, 200 \text{ pA}$	1	$\pm 10 \text{ nA}$	>1
$\pm 100, 50, 20 \text{ pA}$	0.1	$\pm 1 \text{ nA}$	>1

DC current error: <  $\pm 0.5\%$  FS on ranges 200 nA – 10 mA  
<  $\pm 1\%$  FS on ranges 200 pA – 100 nA

Low pass filter: 10 Hz, 3rd order Bessel

e-corder filter settings: 10 kHz to 1 Hz in 10:5:2 steps

Drift with temperature: < 10  $\mu\text{V}/^\circ\text{C}$

## Electrometer & Power Amp

Compliance voltage: > 5 V on 10 mA range  
> 7.5 V on 5 mA range  
> 10 V on other ranges

Maximum control voltage:  $\pm 2.5 \text{ V}$  using internal control  
 $\pm 10 \text{ V}$  using external input

Output current:  $\pm 1 \text{ mA}$  maximum

Input impedance:  $10^{13} \text{ ohm} \parallel 1 \text{ pF}$

Input bias current: < 1 pA @ 25°C

## Control Port

I<sup>2</sup>C input and output: Male and female DB-9 pin connectors.  
Provides control and power.

Power requirements:  $\pm 17$  V DC,  $\sim 20$  mA  
+8 V DC,  $\sim 20$  mA  
 $\sim 0.6$  W quiescent

## Physical Configuration

Dimensions (h  $\times$  w  $\times$  d): 60  $\times$  150  $\times$  200 mm  
2.4  $\times$  5.9  $\times$  7.9 inches

Weight: 1.5 kg (3.3 lb)

Operating conditions: 0 – 35°C  
0 – 90% humidity (non-condensing)

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



# Electrochemical Equations

## Linear Sweep and Cyclic Voltammetry

### The Randles-Sevcik Equation

For a substrate with a reversible redox reaction, at a planar disk electrode, in an unstirred solution, the peak current during linear sweep, or cyclic voltammetry, is given by the Randles–Sevcik equation:

$$i_{pa} = 269n^{3/2}AD^{1/2}Cv^{1/2} \quad \text{for an oxidation (anodic scan)}$$

$$i_{pc} = -269n^{3/2}AD^{1/2}Cv^{1/2} \quad \text{for a reduction (cathodic scan)}$$

The potential where this occurs is given by:

$$E_{pc} = E_{1/2} - \frac{0.0285}{n} \quad E_{pa} = E_{1/2} + \frac{0.0285}{n}$$

where

$i_{pa}$  = current peak during anodic scan (A)

$i_{pc}$  = current peak during cathodic scan (A)

$E_{pa}$  = potential at current peak for anodic scan (V)

$E_{pc}$  = potential at current peak for cathodic scan (V)

$E_{1/2}$  = half potential as determined by cyclic voltammetry (V)

$n$  = the number of electrons transferred to (or from) the substrate molecule

$A$  = area of the exposed surface of the electrode (cm<sup>2</sup>)

$D$  = diffusion coefficient of the substrate molecule ( $\text{cm}^2/\text{s}$ )

$C$  = concentration of the substrate molecule ( $\text{mol/L}$ )

$\nu$  = the scan rate ( $\text{V/s}$ ).

The factor of 269 is a parameter that derives from using a planar electrode at a temperature of 298 K.

Thus a truly reversible reaction can be identified by examining the adherence of the system to the following functions:

$$i_p \propto \sqrt{\nu}$$

$E_p$  independence of the scan rate,  $\nu$

In addition cyclic voltammograms will exhibit:

$$|E_{pa} - E_{pc}| = \frac{57}{n} \text{ mV (at 298 K)}$$

$$\left| \frac{i_{pa}}{i_{pc}} \right| = 1$$

When working in highly resistive solutions, which is often the case when using organic solvents for cyclic voltammetry, the reference electrode should be kept as close as possible to the working electrode to minimise  $iR$  drop. Nonetheless there may still be an appreciable uncompensated resistance which will cause  $|E_{pa} - E_{pc}|$  to become larger than predicted, the peaks to be broader, and the peak currents to be smaller.



# Chronoamperometry

## The Cottrell Equation

When chronoamperometry is performed on an unstirred solution at a planar electrode the faradaic current response is described by the Cottrell equation

$$i = \frac{nFAD^{1/2}C}{1000\pi^{1/2}t^{1/2}}$$

where

$n$  = the number of electrons transferred to (or from) the substrate molecule

$F$  = Faraday's constant, 96485 C/mol

$A$  = area of the exposed surface of the electrode (cm<sup>2</sup>)

$D$  = diffusion coefficient of the substrate molecule (cm<sup>2</sup>/s).

$C$  = concentration of the substrate molecule (mol/L)

$t$  = time (s).

The factor of 1000 is to bring both cm and dm (1 dm<sup>3</sup> = 1000 cm<sup>3</sup> = 1 L) in the equation to common units.

Thus a plot of  $i$  versus  $1/\sqrt{Dt}$  should produce a straight line.

In aqueous solution the coefficient of diffusion usually has a value of between 10<sup>-5</sup> and 10<sup>-6</sup> cm<sup>2</sup>/s.

# Chronocoulometry

## The Integrated Cottrell Equation

The current response equation at a planar electrode for a chronocoulometric experiment is the time integrated form of the Cottrell equation:

$$Q = \int_0^t i \, dt = \frac{2nFAD^{1/2}Ct^{1/2}}{1000\pi^{1/2}} + k$$

where

$Q$  = the number coulombs transferred

$n$  = the number of electrons transferred to (or from) the substrate molecule

$A$  = area of the exposed surface of the electrode ( $\text{cm}^2$ )

$D$  = diffusion coefficient of the substrate molecule ( $\text{cm}^2/\text{s}$ ).

$C$  = concentration of the substrate molecule ( $\text{mol/L}$ )

$t$  = time (s)

$k$  = a constant.

Thus a plot of  $Q$  versus  $t^{1/2}$  should produce a straight line with an intercept of  $k$ , which in turn can be defined as:

$$k = nFA\Gamma + Q_{dl}$$

where

$\Gamma$  = surface concentration of adsorbed substrate ( $\text{mol}/\text{cm}^2$ )

$Q_{dl}$  = double layer charging.

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