Electrochemical Processor Model 1000

Operating Manual

Manufactured by:
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Warranty

We guarantee the manufacture of the instrument and parts against faults for a period of twelve months from the invoice date.

If a fault should occur within this period then we undertake to either:

- Supply free of charge replacement parts for you to fit to the instrument.
- Upon return of the item at your expense, repair or replace (at our discretion) the instrument free of charge and return it to you at our expense.

1. Getting Started

Thank you for purchasing Rank Brothers equipment. Please ensure that you have read and understood this operating manual before use. You should safely store this manual for future reference.

1.1 Do Not

- Do not plug into your local mains supply until you have checked that your local supply voltage matches that stated on the label at the rear of the instrument (adjacent to the mains inlet connector).
- Do not change the fuse or remove any covers with the mains lead connected to the unit.

1.2 Do

- Do ensure that if the moulded plug is removed from the mains lead it is disposed of safely.
- Do read and understand this manual before use.

1.3 Connection to your Mains Supply

IMPORTANT: This unit must be earthed to ensure operator safety. The mains inlet lead may have a moulded plug fitted that is not suitable for connection to your local supply. If it is necessary to remove this plug and fit a suitable one, the removed plug must be safely disposed. The removed plug would present a serious shock hazard if plugged into a suitable supply with the bare wires exposed.

The wires of the mains inlet lead are coloured as follows:

GREEN and YELLOW	EARTH
BLUE	NEUTRAL
BROWN	LIVE

As the colours of the wires in the mains lead may not correspond with the coloured markings identifying the connections in your plug, proceed as follows:

- The GREEN and YELLOW coloured wire must be connected to the terminal in the plug marked with the letter E or marked with the earth symbol or coloured GREEN or coloured GREEN and YELLOW.
- The BLUE wire must be connected to the terminal marked N or coloured BLACK.
- The BROWN wire must be connected to the terminal marked L or coloured RED.

Before connecting the unit to your mains supply ensure that your supply voltage matches that on the label at the rear of the instrument adjacent to the inlet connector.

For operator safety only the correct fuse must be used. Before changing a fuse, switch off the mains supply and disconnect the mains inlet lead from the instrument. The correct fuse is as follows:

220/240 V	T160mA
100/110 V	T160mA

The unit contains no user serviceable parts. The cover should only be removed by competent personnel (after first switching off the power supply and disconnecting the mains inlet lead).

For any servicing or repairs, the unit should be returned to the manufacturer with a covering letter. Please ensure the unit is carefully packaged to avoid damage during shipment.

2. The Galvanostat Controls

The front panel controls are split into three separate groups. Each group will be explained individually although some of the features are very similar between the potentiostat and galvanostat circuit. The galvanostat operates as a constant current device maintaining constant current between the working and counter electrodes whilst enabling the voltage of the working electrode to be monitored with respect to the reference electrode (in three electrode mode). The controls are situated on the left-hand side of the front panel and have blue backgrounds to distinguish them from the potentiostat controls.

Note the galvanostat circuit is completely separate from the potentiostat and can thus be operated simultaneously to, and independently from, the potentiostat circuit.

2.1 Plating Voltage Socket

This is a BNC socket and gives a buffered output of the plating voltage, which can be monitored continuously via a voltmeter or chart recorder.

2.2 Cell Output Socket

This is a BNC socket and gives a buffered output of the potential of the working electrode with respect to the reference electrode, allowing continuous monitoring of this potential. Note the output from this socket can be from −10 V to +10 V approximately, even though the system monitor is only able to measure −3 V to +3 V.

2.3 Plating Current Range Switch

This switch allows the appropriate plating current range to be selected, with four positions giving 1, 10, 100, and 1000 microamps per volt plating voltage (e.g. with the

range switch on 10 and the plating voltage 1.32 volts the plating current is: *10 × 1.32 = 132 microamps*).

2.4 Plating Voltage Potentiometer

The potentiometer knob is a ten-turn type and is lockable in any position by the black lever on the outside (move anti-clockwise to unlock, clockwise to lock). The plating voltage is adjustable between −3 and +3 volts with 000 on the dial giving −3 volts and 1000 giving +3 volts. The plating voltage can be monitored at the Plating Voltage socket at the bottom of the galvanostat section or by selecting *Plating Voltage* on the system monitor switch and reading from the meter. The sign of the plating voltage indicates the direction of flow of current between the working and counter electrodes with a positive plating voltage indicating positive currents (i.e. the working electrode is the anode) and a negative plating voltage indicating negative currents (i.e. the working electrode is the cathode).

2.5 Cell Selector Switch

This switch enables the user to select the type of cell being used; it has three positions:

In the *2 Electrode* position, the counter and reference electrodes are internally shorted together. The cell output monitors the voltage between the working and counter/reference electrodes to be measured for any particular current flowing and this provides a measure of the impedance of the cell.

In the *Test* position a test load of 20 kΩ is insert into the circuit with 10kΩ inserted between the working and reference electrodes and 10 kΩ between the reference and counter electrodes. The cell output, as before, measures the voltage of the working electrode with respect to the reference electrode and hence the voltage across one of the 10 kΩ resistors. The cell output should thus correspond to the cell current multiplied by 10,000 (the 10 kΩ load). For example with the *Plating Current* range switch at 100 microamps the plating voltage will be equal to the cell output voltage. Hence with a plating voltage of 1.6 volts:

$$plating\ current = 1.6 \times 100 = 160\ microamps$$

$$cell\ output\ voltage = cell\ current\ (amps) \times cell\ impedance\ (\Omega)$$

$$cell\ output\ voltage = 0.00016 \times 10000 = 1.6\ volts$$

This enables a quick check to be made on the galvanostat circuit to ensure correct operation. Note the cell output will be of the opposite sign to the plating voltage (e.g. −1.6 volts). If the *Plating Current* range switch is in any other position then the cell output will need to be scaled as appropriate by a factor of tens.

In the *3 Electrode* position a constant current is maintained between the working and counter electrodes and the cell output voltage is again monitoring the reference electrode voltage.

2.6 Cell Socket

This socket is a high-grade 5-pin DIN socket for connection of the cell. The pin connections are as follows:

Pin 1	Counter electrode.
Pin 2	0 V can be connected to screen of screened cable.
Pin 3	Working electrode.
Pin 4	Guard. This output exactly tracks the cell output voltage, when connected to the screen of the cable it provides better noise pickup reduction than connecting the screen to 0 V. Note this connection is an output and must not be connected to 0 V, otherwise damage will occur to the galvanostat circuit.
Pin 5	Reference electrode.

A DIN to 4 mm lead is supplied to allow cells with 4 mm plugs to be easily connected via the 5-pin DIN socket. The lead has four 4 mm colour coded sockets as follows:

Red	Reference electrode.
Green	0 V
Yellow	Working electrode.
Blue	Counter electrode.

Note this lead has no connection to pin 4 (the Guard) of the DIN plug.

3. The Potentiostat Controls

The potentiostat is a constant voltage device. In three electrode mode it controls the voltage between the working and counter electrodes to maintain a constant potential between the working and reference electrodes. The cell output provides a direct measure of the current flowing between the working and counter electrodes. The controls are in the middle of the front panel and have a red background to distinguish them from the galvanostat. The potentiostat has more controls than the galvanostat but similar controls are sited in similar positions to make them easier to use. The individual controls and their functions are described in detail below.

Note the potentiostat circuit is completely separate from the galvanostat and can thus be operated simultaneously to, and independently from, the galvanostat circuit.

3.1 Polarising Voltage Socket

This is a BNC type and allows continuous monitoring of the buffered polarising voltage.

3.2 Cell Output Socket

This is a BNC type and allows continuous monitoring of the buffered cell output voltage. Note this output can vary between −10 V and +10 V approximately although the system monitor can only measure between −3 V to +3 V.

3.3 Cell Current Range Switch

This selects the sensitivity of the cell output and has four ranges of 0.1, 1, 10, and 100 microamps per volt. Thus if the switch is set to 0.1 microamps and the cell output is 2.25, then the current flowing between the counter and working electrodes is:

$$0.1 \times 2.25 = 0.225 \text{ microamps}$$

The least sensitive range position is 100, it is advisable to start with the switch in this position until the current flowing is known and the appropriate range can be selected.

3.4 Polarising Voltage Potentiometer

This control is a lockable ten-turn type that can be locked in any position by sliding the black lever clockwise. Sliding the lever anticlockwise releases the knob for adjustment. The polarising voltage is the voltage maintained between the working and reference electrodes and is measured with respect to the reference electrode (or counter/reference electrode in two electrode mode). For example a polarising voltage of −0.8 V indicates that the reference electrode (counter/reference in two electrode mode) is being held at +0.8 V (the working electrode is held at 0 V by a virtual earth circuit). The polarising voltage is infinitely adjustable between −3 V and +3 V with 000 on the dial giving −3 V and 1000 giving +3 V. The polarising voltage is available at the polarising voltage socket for continuous monitoring.

3.5 Filter Switch

The *Filter* switch allows a low pass filter to be used if required. The filter has a pass band from dc to approximately 10 Hz and is extremely useful for removing unwanted electrical noise from the cell, especially from stirrer and mains pickup when using the higher sensitivity cell current ranges. The filter should not introduce any significant dc offset into the cell output voltage (less than 5 millivolts) but it may be advisable to ensure that the *Filter* switch is in the correct position before adjusting the offset current controls.

3.6 Offset Current Range Switch

This switch enables an appropriate offset current to be selected. The switch has five positions: *Off*, −0.03 to 0.03, −0.3 to 0.3, −3 to 3, −30 to 30 microamps, with the *Off* position disabling the offset current completely.

3.7 Offset Current Adjust Potentiometer

This is a ten-turn type and provides infinite adjustment of the offset current in the range selected by the *Offset Current* range switch. Thus with the range switch set to −0.3 to 0.3 microamps, a reading of 000 gives an offset current of −0.3 microamps and a reading of 1000 gives an offset current of 0.3 microamps.

The offset current is summed with the current flowing between the counter and working electrodes and produces an offset voltage at the cell output socket. It can be used to trim out any residual current in the cell (i.e. to produce a cell output voltage of zero volts in the absence of the gas being monitored). It is also possible to use the offset current to suppress the zero and hence accurately monitor small changes in output current from the cell whilst a large background current is present. The following is an example and will serve to clarify this facility:

THE PROBLEM: We wish to accurately monitor the oxygen levels in a sample between 60% and 65% saturation. Normally the chart recorder is adjusted from 0% to 100% so that fluctuations in the sample occur in a band only 5% of the width of the chart paper.

THE SOLUTION: When calibrated the oxygen electrode has a cell current of 1.56 microamps with air saturated water. Therefore the *Cell Current* switch would normally be set to 1 microamp per volt giving a cell output of approximately 1.56 V. 60% saturation will thus give a cell current of *1.56 × (60 / 100)* or 0.936 microamps. Fill the electrode with a known 60% oxygen saturated sample (this can be made up by mixing oxygen saturated and oxygen purged samples in the appropriate ratios). Switch the *Cell Current* range switch to the 0.1 microamp position and the *Offset Current* range switch to the 3 microamp range and use the *Offset Current Adjust* potentiometer to adjust the cell output to 0 V. A cell output of 0 V now represents 60% saturation and an output of 1 V represents approximately 65% saturation thus the full chart width now represents 60% to 65% saturation allowing the user to zoom in on the area of interest.

3.8 Cell Selector Switch

This switch has three positions as follows:

In the *2 Electrode* position, the counter and reference electrodes are internally shorted together and held at minus the polarising voltage. This mode is required to use our oxygen electrodes.

In the *Test* position, two 1 MΩ resistors are substituted for the cell producing a dummy load. With a polarising voltage of 1 V the cell current will be 1 microamp giving a cell output of 1 V when the *Cell Current* range switch is on 1 microamp. The cell output voltage will therefore be within a few millivolts of the polarising voltage providing the *Cell Current* range switch is set to 1. This provides a useful check to ensure that the potentiostat circuit is operating correctly. Note that with the *Cell Current* range switch in

positions other than 1 microamp the cell output will need to be scaled by factors of ten as appropriate (e.g. with the range switch on 10 microamps the cell output voltage will be equal to the polarising voltage divided by ten).

In the *3 Electrode* position a constant potential (the polarising voltage) is maintained between the working and reference electrodes, while the cell output measures the current flowing between the counter and working electrodes.

3.9 Cell Socket

The socket is a high-grade 5-pin DIN socket for connection to the cell. The pin connections are as follows:

Pin 1	Counter electrode.
Pin 2	0 V can be connected to screen of screened cable.
Pin 3	Working electrode.
Pin 4	0 V as per pin 2.
Pin 5	Reference electrode.

A DIN to 4 mm lead is supplied to allow cells with 4 mm plugs to be easily connected via the 5-pin DIN socket. The lead has four 4 mm colour coded sockets as follows:

Red	Reference electrode.
Green	0 V
Yellow	Working electrode.
Blue	Counter electrode.

4. System Monitor Controls

The system monitor consists of a digital voltmeter coupled to a selector switch plus two warning LEDs (Light Emitting Diodes). The individual components are described in more detail below.

4.1 Digital Voltmeter

The digital voltmeter (DVM) measures voltages between −3 V and +3 V with an accuracy of 0.5% full scale. The meter has five triangular flags which illuminate to indicate which parameter is being measured (e.g. galvanostat cell output). The meter also has a bar graph scaled −3 V to +3 V. The meter will only measure voltages in the range −3 V to +3 V, with measurements outside these ranges causing the whole display to flash indicating an over-range condition. Note no damage will occur to the meter with voltages between −10 V to +10 V. Thus with a chart recorder connected, it may be advantageous to allow the cell output to exceed the meter range giving a greater resolution of measurement.

4.2 Monitor Selection Switch

This switch enables selection of the appropriate voltage to be displayed by the DVM. It is possible to select either, the galvanostat cell output or plating voltage, or potentiostat polarising voltage or cell output. The galvanostat voltages are colour coded blue and the potentiostat voltages are coded red.

4.3 Galvanostat Overload LED

This LED monitors the output voltage of the drive amplifier (connected to the working electrode) and will illuminate if the voltage becomes higher than 9 V or lower than −9 V. The drive amplifier output is only able to swing between −10 V and +10 V and thus the LED illuminates to show an overload on the drive amplifier output. The LED will illuminate for four reasons:

- The cell has too high an impedance to achieve the plating current desired.
- The cell has a fault and is open circuit.
- No cell is connected to the galvanostat.
- A two electrode cell is being used with the cell selector switch in three electrode mode.

4.4 Potentiostat Overload LED

This LED is also connected to the drive amplifier and monitors the voltage in exactly the same way as the galvanostat overload. The LED will illuminate for either of the following three reasons:

- The reference electrode (in three electrode mode) is open circuit.
- A two electrode cell is being used with the cell selector in three electrode mode.
- No cell is connected to the potentiostat.

Note if either of the galvanostat or potentiostat circuits are not being used then setting the appropriate cell selector to *Test* and setting the appropriate current range switch to the least sensitive range (fully anticlockwise) should ensure that the overload LED's are extinguished.

4.5 Power Indicator

This neon is illuminated to show that the unit is connected to your local power and the power switch is on.

5. Using with a Rank Brothers Oxygen Electrode

When using the Electrochemical Processor with a Rank Brothers Oxygen Electrode the following notes may be found helpful.

5.1 Connections

The platinum electrode (connected to the blue wire of the cable) is the working electrode and should be connected to pin 3 of a 5-pin 240 degree DIN plug, and the silver electrode (connected to the red wire of the cable) is the counter electrode and should be connected to pin 1. The screen of the cable should be connected to 0 V, pin 2 or pin 4 to reduce noise pickup.

5.2 Operation

1. Set up the electrode with a new membrane and fresh electrolyte.
2. Plug the electrode into the potentiostat and place the electrode onto a stirring head.
3. Set stirring speed to a suitable speed (about 600 rpm).
4. Switch on the Electrochemical Processor and set the potentiostat controls as follows: *Cell Current* to 100 microamps, *Filter* to *Off*, *Offset Current* to *Off*, and *Cell* selector to *2 Electrode* mode.
5. Adjust the polarising voltage to a suitable value, normally −0.6 V. This can be set using the system monitor to display the current value.
6. Switch the system monitor to potentiostat cell output and monitor the current flowing. If necessary the *Cell Current* range switch can be adjusted to give a more suitable cell output voltage.
7. To trim out any residual current from the cell, purge all the oxygen from the cell. Then if the cell output voltage is not zero select a suitable *Offset Current* range and set the *Offset Current Adjust* potentiometer until the cell output is zero. The calibration can then be carried out with 100% saturated samples.
8. The system is now ready to carry out your tests.
9. If the cell output has too much electrical noise then the filter can be switched in, but for very accurate work it may be advisable to trim out the residual current and carry out the calibration after the filter has been switched in.