

# GAMRY INSTRUMENTS

## Reference 3000™ Potentiostat/Galvanostat/ZRA Operator's Manual



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Please have your instrument model and serial numbers available, as well as any applicable software and firmware revisions.

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## Chapter 1: Safety Considerations

Your Reference 3000 Potentiostat/Galvanostat/ZRA has been supplied in a safe condition. This chapter of the Reference 3000 Operator's Manual contains some information and warnings that you must follow to insure continued safe operation of the Reference 3000.

The safety information in this chapter applies to both the Reference 3000 and the Reference 3000 equipped with its AE Auxiliary Electrometer.

### Inspection

When you receive your Reference 3000 Potentiostat/Galvanostat/ZRA, inspect it for evidence of shipping damage. If you note any damage, please notify Gamry Instruments Inc. and the shipping carrier immediately. Save the shipping container for possible inspection by the carrier.



**Warning:** A Reference 3000 damaged in shipment can be a safety hazard. Do not operate damaged apparatus until a qualified service technician has verified its safety. Tag a damaged Reference 3000 to indicate that it could be a safety hazard.

### Product Safety

The Reference 3000 has been designed, tested and certified to meet the requirements of an international standard, EN 61010, *Safety requirements for **electrical equipment** for measurement, control, and laboratory use*. As defined in this standard, it is a Category II apparatus, with any "hazardous live voltages" protected by "reinforced insulation".

The Reference 3000 contains a limited amount of internal circuitry that operate at "hazardous live" voltages as defined in EN 61010 (the standard mentioned above). "Reinforced insulation" (again defined in EN 61010) is used to reduce the risk of electrical shock because of this "hazardous live" voltage.

The majority of the Reference 3000's circuitry does not contain voltages higher than 42 V DC. As a generalization, input and output voltages in the Reference 3000 are limited to 36 V. This voltage level is considered safe.

The "AC Adapter" supplied with the Reference 3000 is certified under EN 60950. The AC Adapter converts the AC mains voltage to 24 V DC, which is used to power the Reference 3000.

Always use the AC adapter (power brick) supplied with your Reference 3000 to supply DC power to the instrument.



**Caution:** Do not use a DC power source other than the AC adapter model provided with your Reference 3000. Other replacements may void the performance and/or safety characteristics of the Reference 3000.

## AC Mains Connection to the Power Brick

The Reference 3000 does not connect directly to an AC Mains supply. Instead, the mains are connected to the desktop AC adapter (power brick), which outputs 24 V DC, which in turn powers the Reference 3000.



The Reference 3000's AC Adapter is rated for operation from 100 to 240 V AC, 47 to 63 Hz. It should therefore be useful throughout the world.

The Reference 3000 is normally provided with an AC line cord suitable for your location. This AC line cord connects the AC mains to the AC power adapter. If your Reference 3000 has been provided without an AC line cord, or a cord that is not compatible with your local AC mains socket, obtain a line cord certified for use in your country. Contact your local Gamry Representative or e-mail to [techsupport@gamry.com](mailto:techsupport@gamry.com) if you are uncertain what AC line cord to use.

## Grounding in the Reference 3000

The circuitry and the metal case of the Reference 3000 are *not* connected to an earth ground. If they were connected to earth ground, it would compromise the Reference 3000's ability to make measurements in electrochemical cells that contain earth grounded conductors. A few examples of such cells include autoclaves, metallographic stress apparatus, chemical storage tanks, and most large fuel-cell stacks.

Most electrochemical cells are isolated from earth ground, so isolation of the Reference 3000 from earth is not required. In these cases, connection of the Reference 3000 chassis to an earth ground may lower noise seen in electrochemical tests. A **Chassis Ground** binding post on the rear panel of the Reference 3000 makes for easy implementation of this connection. Simply run a wire from this binding post to a suitable source of earth ground. A black 1.2-meter wire is provided with the Reference 3000 to ease this connection.



Sources of earth ground include

- Most metal water pipes,
- The chassis of **most** electronic apparatus (which are generally earth-grounded), and
- The protective ground terminal of an AC mains power plug.

We recommend that you discuss grounding with an electrical or electronics professional prior to making this earth-ground connection.

Note this connection of the Reference 3000 to an earth ground is not a "Protective Earth Ground" as defined in EN 61010. The Reference 3000 is safe in the absence of this connection.

This binding post is not intended for any use other than connecting the Reference 3000 to an earth ground to improve shielding against noise. Connecting this binding post to a hazardous voltage can create a significant safety hazard.



**Warning:** Do not connect the chassis ground binding post to any voltage other than earth ground. An improper connection can create a safety hazard, which could result in personal injury or death.

An earth ground connection can cause problems when testing batteries, fuel cells, or capacitors. Many of these devices can source huge currents, often tens or hundreds of amperes. If the Reference 3000 chassis is earth grounded, and another location in the stack is accidentally (or intentionally) connected to earth ground, a portion of the stack is shorted through the Reference 3000's cell cable. Very large current flows when this occurs. Fuses in the cell cable will open up to prevent damage to the instrument. When this happens, the failed fuses must be replaced before the instrument can be used again. The fuses in the cell cable are not essential for operator safety. A section in Chapter **Error! Reference source not found.** describes the fuses and their replacement in detail.



The fuses in the Reference 3000 cell cable do not protect against a safety hazard. They are needed to prevent damage to the instrument if it is improperly connected.

## Operation with Earth-Grounded Cells and Auxiliary Apparatus

As described above, the Reference 3000 circuitry is isolated from earth ground, allowing it to make measurements on cells that include an earth ground. This ground isolation is often called **floating** operation. Cells with earth ground include many autoclaves, pipelines and storage tanks, and many fuel-cell systems.

Connection of the Reference 3000 to auxiliary apparatus will often earth-ground the Reference 3000, destroying its ability to float and make measurements on earth-grounded cells. Connection of the Monitor BNCs to an oscilloscope is an example where the instrument is earthed.

The **User I/O** connector can be connected to earth-grounded apparatus without earth-grounding the Reference 3000, if the cable connections are done carefully. The metal shell on the Reference 3000 **User I/O** Connector is connected to the instrument's chassis, which is a floating ground. In a system that needs isolation from earth ground, the shield of a **User I/O** cable must not connect the D-connector's metal shell to earth ground. Reference all **User I/O** signals to pin 6 of the D-connector, which is isolated ground on the Reference 3000.



**Caution:** Floating operation of the Reference 3000 can be compromised by improper cables to your I/O Connector. We do not recommend use of standard 15-pin shielded cables with this connector. Custom cables with the shield connected to pin 6 of the D-connector are preferred.



**Warning:** Do not connect the chassis ground binding post to any voltage other than earth ground. An improper connection can create a safety hazard, which could result in personal injury or death.

The Reference 3000 contains surge suppressors that limit the voltage difference between the Reference 3000's chassis ground and earth ground to about 40 V. These surge suppressors are not part of the safety mechanisms in the Reference 3000. Instead they are present to limit the possibility of improper instrument operation or instrument damage caused by electrostatic discharge (static electricity) and other surge events such as lightning.

## Temperature and Ventilation

Your Reference 3000 Potentiostat/Galvanostat/ZRA was designed for indoor use at ambient temperatures between 0°C and 45°C.

The Reference 3000 uses forced air-cooling to keep its electronic components within their recommended operating temperature range. Three fans on the rear panel of the Reference 3000 draw air into the chassis. The air exits from slots located on the sides of the chassis near the front panel.



**Caution:** Do not block the airflow into or out of the Reference 3000 chassis. While the circuitry should shut down before it is damaged by excessive heat, the Reference 3000 enclosure may become uncomfortably hot to the touch if insufficient air flows through the chassis. Running the Reference 3000 without adequate cooling could shorten the time to failure of some of the circuitry.

Be careful when operating the Reference 3000 in an enclosed space (such as an enclosed relay rack or NEMA enclosure). The temperature within the enclosure must not exceed 45°C. You may need to provide ventilation holes or even forced air-cooling for the enclosed space if you determine that there is an excessive temperature rise within the space.

## Defects and Abnormal Stresses

Treat your Reference 3000 as potentially hazardous if any of the following is true of the unit:

- It shows visible damage,
- It does not operate properly,
- It has been stored for a long period of time under unfavorable conditions,
- It has been dropped or subjected to severe transport stress,
- It has been subjected to environmental stress (corrosive atmosphere, fire, etc.).

Do not use your Reference 3000 or any other apparatus if you think it could be hazardous. Have it checked by qualified service personnel.

## Environmental Limits

There are environmental limit conditions on the storage, shipping and operation of this equipment. The Reference 3000 is *not* designed for outdoor use.

### Storage

Ambient Temperature	–40°C to 75°C
Relative Humidity	Maximum 90% non-condensing

### Shipping

Same as storage plus	
Acceleration	Maximum 30 G

### Operation

Ambient Temperature	0°C to 45°C
Relative Humidity	Maximum 90% non-condensing



**Warning:** The Reference 3000 is not designed for operation in conditions where liquid water may enter the chassis, or water vapor may condense within the chassis. Operation of a Reference 3000 that has water within the chassis can create a safety hazard, which could result in personal injury or death.

## Cleaning

Disconnect the Reference 3000 from all power sources prior to cleaning.

Use a cloth *lightly* dampened with either clean water or water containing a mild detergent to clean the outside of the Reference 3000 enclosure. Alternatively, you can use isopropyl alcohol. Do not use a wet rag or allow fluid to enter the Reference 3000 enclosure. Do not immerse the Reference 3000 in any type of cleaning fluid (including water). Do not use any abrasive cleaners.

## Service

Your Reference 3000 Potentiostat/Galvanostat/ZRA has no user-serviceable parts inside. Refer all service to a qualified service technician.



**Warning:** Never operate the Reference 3000 with any cover or panel on the chassis open. Dangerous voltages may be present at several points within the Reference 3000 chassis, including PC board traces. Always remove the power connection before opening the Reference 3000 case.

## RFI Warning

Your Reference 3000 Potentiostat/Galvanostat/ZRA generates, uses, and can radiate radio-frequency energy. The radiated levels are low enough that the Reference 3000 should not create an interference problem in most industrial laboratory environments.

The Reference 3000 has been tested for both radiated and conducted RF interference, and has been found to be in compliance with FCC Part 18 and EN 61326:1998—*Electrical equipment for measurement, control, and laboratory use— EMC Requirements*.

## Electrical Transient Sensitivity

Your Reference 3000 Potentiostat/Galvanostat/ZRA was designed to offer reasonable immunity from electrical transients, including transients on the incoming AC Mains supply and Electrostatic Discharge. It has been tested for compliance with EN 61326:1998—*Electrical equipment for measurement, control, and laboratory use—EMC Requirements* describing acceptable limits for Electrical Transient susceptibility in Laboratory Test equipment. The Reference 3000 is not rated for continuous use when subject to ESD events. It should suffer no permanent damage when subject to the standard ESD events defined in EN61326, but may cease normal operation until it is powered down and restarted.

In severe cases, the Reference 3000 could malfunction as a result of electrical transients such as a static discharge. If you are having problems in this regard, the following steps may help:

If the problem is static electricity (sparks are apparent when you touch the Reference 3000 or its cables):

- Placing your Reference 3000 on a static control work surface may help. Static-control work surfaces are now generally available from computer-supply houses and electronics tool suppliers. An antistatic floor mat may also help, particularly if a carpet is involved in generating the static electricity.
- Air ionizers or even simple air humidifiers can reduce the voltage available in static discharges.

If the problem is AC power-line transients (often from large electrical motors near the Reference 3000):

- Try plugging your Reference 3000 into a different AC power branch circuit.
- Plug your Reference into a power-line surge suppressor. Inexpensive surge suppressors designed for use with computer equipment are now generally available.

Contact Gamry Instruments, Inc. if these measures do not solve the problem.

## CE Compliance

The European Community has instituted standards limiting radio-frequency interference emitted by electronic devices, setting limits for susceptibility of apparatus to RF energy and transient events, and mandating safety requirements. Gamry Instruments, Inc. has designed and tested the Reference 3000 to comply with these standards.

The relevant CE regulations include EN 61010 and EN 61326.

## RoHS Compliance

The Reference 3000 has been built using lead-free components and lead-free solder. It is in compliance with the European RoHS initiative.

## Chapter 2: Introduction

### About this Manual

This manual covers the installation, safety, and use of the Gamry Instruments Reference 3000 Potentiostat/Galvanostat/ZRA. It also includes information about the Reference 3000's AE Auxiliary Electrometer option.

This manual describes use of a Reference 3000 with Revision 7.00 (and later revisions) of the Gamry Framework software. It is equally useful when setting up a newly purchased potentiostat or modifying the setup of an older potentiostat for use with new software.

You will find dry technical material such as specifications and connector pin-out diagrams in the Appendices.

This manual does not discuss software installation or software operation in any detail. Software support for the Reference 3000 is described in the Gamry Framework's Help system.

All the Gamry Instruments' applications running under the Gamry Framework control the Reference 3000 via a PSTAT object. See the Framework's Help system for information concerning PSTAT objects and their functions.

### About the Reference 3000

The Reference 3000 Potentiostat is a research-grade electrochemical instrument packaged in a small, easy-to-handle case. It is the larger, higher-current sibling of Gamry's extremely popular Reference 600+ potentiostat. It is especially useful when currents higher than the 600 mA current-limit of the Reference 600+ are required.

Typical applications for the Reference 3000 include research regarding batteries, fuel cells and super-capacitors. It should also prove useful in studies involving electrochemical synthesis, electroplating and corrosion. While it can apply and measure ampere-level currents, it is also an excellent small-signal potentiostat that can work with picoampere and sometimes even femtoampere current levels.

The Reference 3000 offers measurement capabilities similar to instruments many times larger in size, weight, and price. The Reference 3000 can operate as a potentiostat, a galvanostat, or a ZRA (zero-resistance ammeter). A new stack mode allows precision control and measurement of battery-stack voltages as large as  $\pm 36$  V.

The Reference 3000 offers two different compliance-voltage and compliance-current settings. You can choose to operate the Reference 3000 set for compliance of  $\pm 1.5$  A and voltages up to  $\pm 30$  V, or you can choose to operate at  $\pm 3$  A and voltages up to  $\pm 15$  V. This choice cannot be changed in the middle of an experimental run.

Reference 3000 features include:

- Eleven-decade current auto-ranging,
- Electrical isolation from earth ground,
- Switchable compliance-current and compliance-voltage settings,
- Current-interrupt *iR*-compensation, and
- Both analog and digital filtering.

A sine-wave generator on the Reference 3000 allows its use for impedance measurements at frequencies up to 1 MHz. Data can be acquired at frequencies up to 300 000 points per second, allowing cyclic voltammetry at scan rates of 1500 V/s with 5 mV per point resolution.

A unique DSP (Digital Signal Processing) data-acquisition mode allows the Reference 3000 to reject noise from the instrument itself, from the electrochemical cell, and from the laboratory environment. In many cases where other instruments require a cell in a Faraday shield to make quiet measurements, the Reference 3000 can be used with the cell exposed on a bench top.

The Reference 3000 offers an unprecedented combination of high speed, high sensitivity, and low noise. State-of-the-art analog components were used throughout the design. In all design decisions, performance weighed more heavily than product cost.

The Reference 3000, like all Gamry potentiostats, requires a computer for its use. Unlike most of Gamry's older potentiostats, the Reference 3000 connects to the computer through a USB connection. The USB connection has become truly universal, with USB ports found on all modern computers. Gamry's software currently supports up to 16 Reference 3000 Potentiostats connected to one computer.

The Reference 3000 is isolated from earth ground. It can therefore be used to make measurements on cells that contain an earth-grounded metal. A few of examples of such systems include are autoclaves, large metal storage tanks, stress apparatus, and capillary electrophoresis detectors.

### **About the Auxiliary Electrometer Option**

The Reference 3000 Potentiostat can be equipped with a unique Auxiliary Electrometer option. This factory-installed option is especially useful when you need to measure the performance of individual cells in a multi-cell fuel cell or battery stack.

Up to eight completely independent voltages can be measured using this option. The measurements are fully differential, so cell voltages at any point in a stack can be measured. Each input can measure a  $\pm 5$  V signal superimposed on a common-mode voltage that can be as large as  $\pm 36$  V! The input impedance is greater than  $10^{11} \Omega$ , so the inputs can even be connected to small diameter Luggin probes.

This option can be used to simultaneously measure electrochemical impedance on up to eight cells in a cell stack. This is often of great interest because cells in a fuel cell or battery stack are not identical.

The AE is not restricted to energy-conversion and -storage applications. The electrometer inputs can measure virtually any voltage. You can measure voltages from temperature, pressure, or strain transducers or voltages of multiple reference electrodes in a cell.

### **Notational Conventions**

In order to make this manual more readable we have adopted some notational conventions. These are used throughout this manual and all other Gamry Instruments manuals:

- Numbered lists. A numbered list is reserved for step-by-step procedures, with the steps always performed sequentially.
- Bulleted list. The items in a bulleted list, such as this one, are grouped together because they represent similar items. The order of items in the list is not critical..

## Chapter 3: Instrument Circuitry

### Reference 3000 Schematic/Block Diagrams

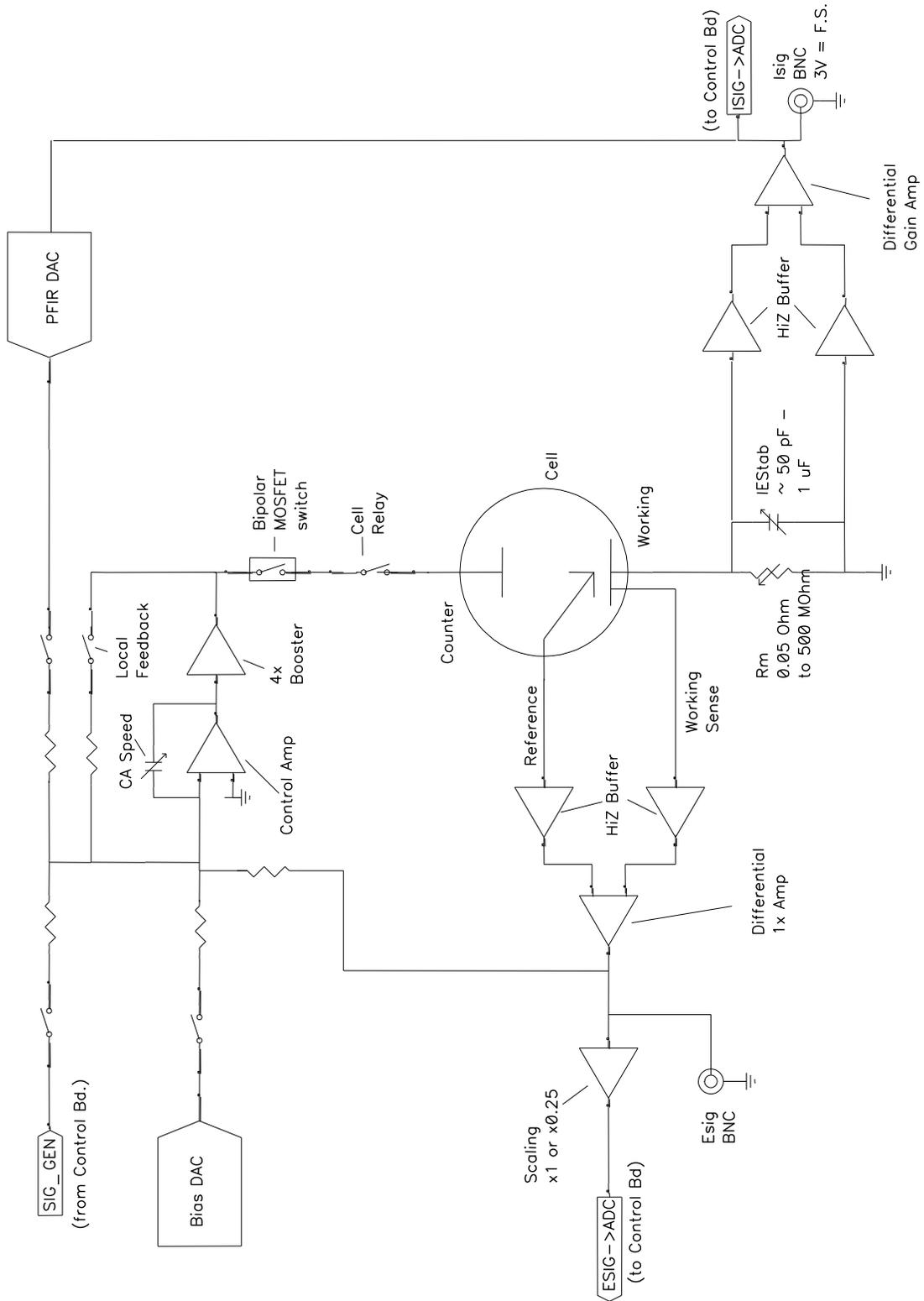
If you are not familiar with electronic schematics or potentiostats, you probably want to skip this chapter. This information is for expert use only and is not required for routine use of the Reference 3000.

The following figures are partly schematic diagrams and partly block diagrams. They are intended to show the basic principles of the Reference 3000 circuitry without the confusion of the full circuitry details. The complexity of the Reference 3000 can be quite daunting: the Reference 3000 circuit boards contain more than 3000 components connected by almost 2500 circuit nets!

The schematic/block diagram figures show:

- The potentiostat board and heat sink board in a potentiostatic control mode,
- The control board circuits for signal generation,
- The control board circuits for signal conditioning and A/D conversion,
- The Auxiliary ADC channel input switching,
- The microprocessors in the Reference 3000,
- DC-DC power conversion,
- The optional Multi Channel (MCE) circuitry.

**Figure 3-1**  
**Reference 3000 Potentiostat Board in Potentiostat Mode**  
**Simplified Schematic/Block Diagram**

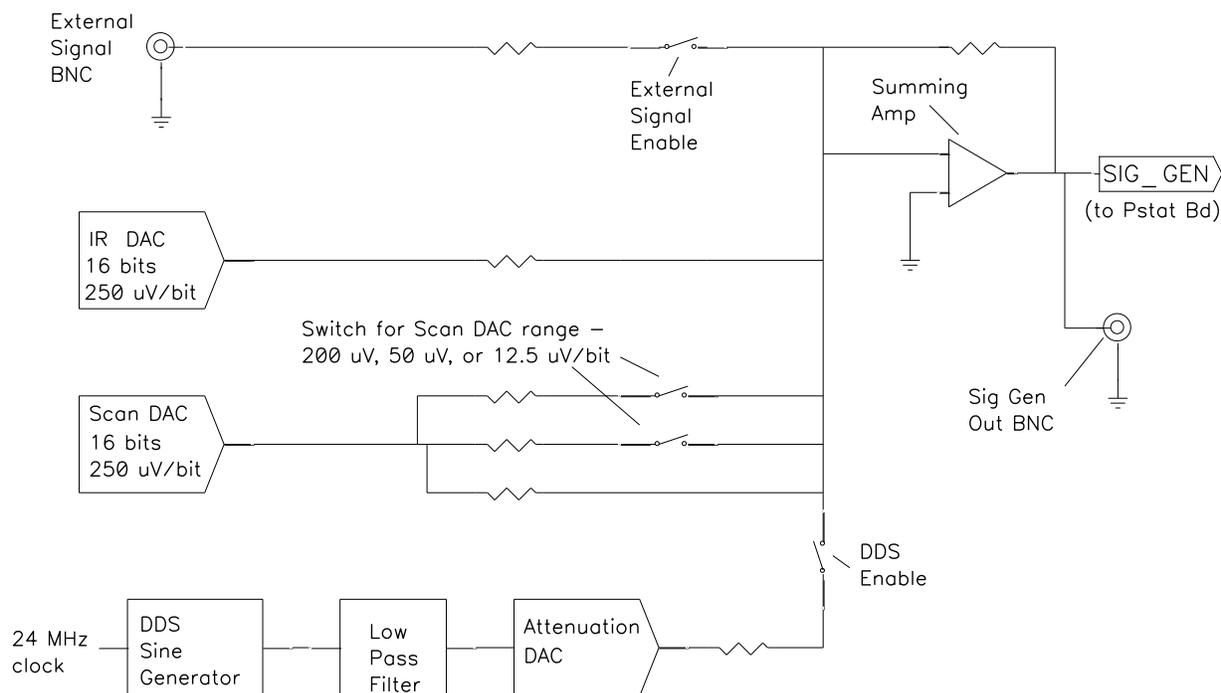


Notes for Figure 3-1

- The 4× booster following the Control Amp can operate with two combinations of compliance current and compliance voltage. One is  $\pm 1.5$  A at  $\pm 30$  V, the other is  $\pm 3$  A at  $\pm 15$  V.
- Only Potentiostat Mode circuitry is shown in this figure. In this mode the voltage difference between the Reference and Working Sense leads (called  $E_{sig}$ ) is feedback into the control amplifier.  
In Galvanostat Mode, the feedback is from  $I_{sig}$ .  
In the ZRA and stack modes, the feedback is from a differential amplifier measuring the difference between the Counter Sense and Working Sense leads of the cell cable. The counter sense circuitry is not shown. It is conceptually similar to the voltage-sensing circuit that generates  $E_{sig}$ , except that it can measure voltage differences as large as  $\pm 36$  V.
- The Bias DAC and PFIR (Positive Feedback  $IR$ -compensation) DAC are set using a computer bus that is not shown.
- Switches are either reed relays or MOS switches as appropriate. All switches are under computer control (obviously, for the Reference 3000 does not have a knob-and-dial front panel).
- The variable current-measurement resistor,  $R_m$ , is one of eleven fixed-value resistors selected using relays. The resistor's values vary by decades: 50 m $\Omega$ , 500 m $\Omega$ , 5 $\Omega$ , 50  $\Omega$  ... 500 M $\Omega$ . The lower-value resistors require software-gain corrections. Correction values are measured at Gamry's test facility and stored in an EEPROM on the Reference 3000 potentiostat board. Software calibration of the instrument by a customer does *not* change these  $R_m$  gain corrections.
- Other components shown as being variable (IEStab capacitor and CASpeed capacitor) are actually several fixed-value components switched into the circuit, and not continuously variable.
- The monitor BNC connectors for  $I_{sig}$  and  $E_{sig}$  are lightly filtered using an RLC circuit.
- The ADC channel for  $E_{sig}$  is actually switchable between  $E_{sig}$  (the reference voltage minus the working sense voltage) and  $Z_{sig}$  (the counter sense voltage minus the working sense voltage). The  $Z_{sig}$  connection allows the Reference 3000 to measure the voltage of battery or fuel-cell stacks.
- The programmable attenuator on  $E_{sig}$  prior to the ADC channel scales the  $E_{sig}$  voltage to make it compatible with the A/D channel's  $\pm 3$  V input range. The 0.25 gain setting allows the Reference 3000 to measure potential signals slightly in excess of 10 volts (on a 12-volt full-scale range).  $I_{sig}$  is gained to be 3 V full-scale so it does not require a similar attenuation function.
- All the resistors summing voltages into the Control Amplifier input do not have values shown on the diagram; their values depend on scaling factors too complex to discuss in this chapter.
- Calibration components are not shown.
- Gamry's software can disconnect the signal generator from the Potentiostat. Once disconnected it can be used for other experimental-control tasks.
- Overload protection and overload detection are not shown. Good engineering practice demands that any possible misconnection of the cell leads will not damage the instrument. This practice has been followed in the Reference 3000 design.

The overload protection can handle overloads of up to 30 A for very short times. Fuses in the Working and Counter Sense leads always open up before overload conditions can damage the instrument. Misconnection of a battery, fuel cell, or super-capacitor stack can open the fuse, but will not cause hardware failures.

**Figure 3-2**  
**Reference 3000 Signal-Generation Circuitry**



Notes on Figure 3-2:

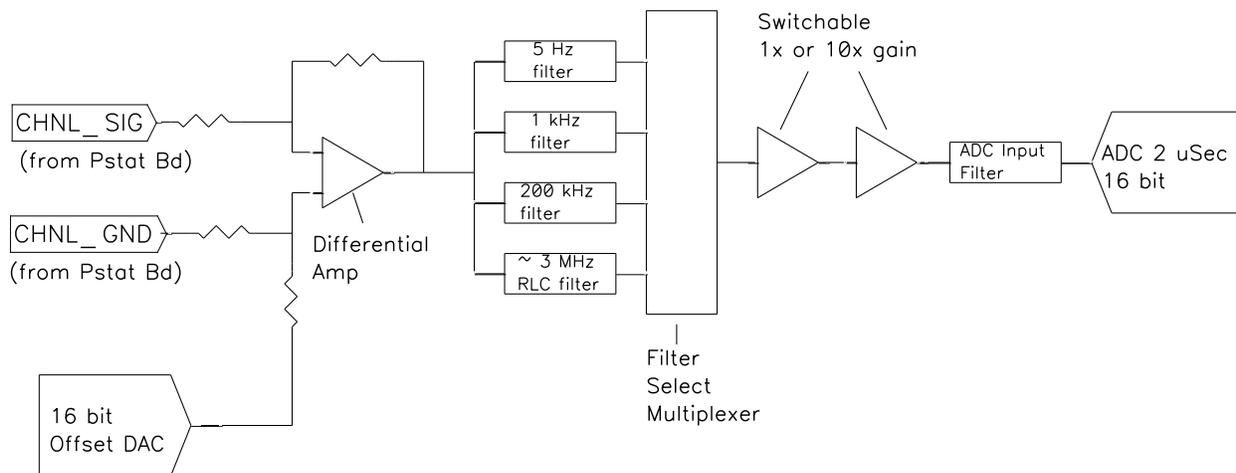
- All the resistors summing voltages into the Summing Amplifier input do not have values shown on the diagram. Their values depend on scaling factors too complex for this simplified diagram.
- The IR DAC has a  $\pm 8$  V full-scale range.
- Calibration components are not shown.
- The DDS can generate fixed-amplitude sine waves with frequencies between 1 MHz and 1 mHz. In practice, Gamry's electrochemical-impedance spectroscopy software uses the Scan DAC to generate sine signals if the frequency is below 1 Hz.

The low-pass filter removes high-frequency distortion in the "raw" DDS output.

The attenuator scales the DDS. The maximum output signal is 5.979 V peak-to-peak and the minimum is approximately 11  $\mu$ V peak-to-peak.

- The BNC connector for Sig Gen out is lightly filtered using an RLC circuit.

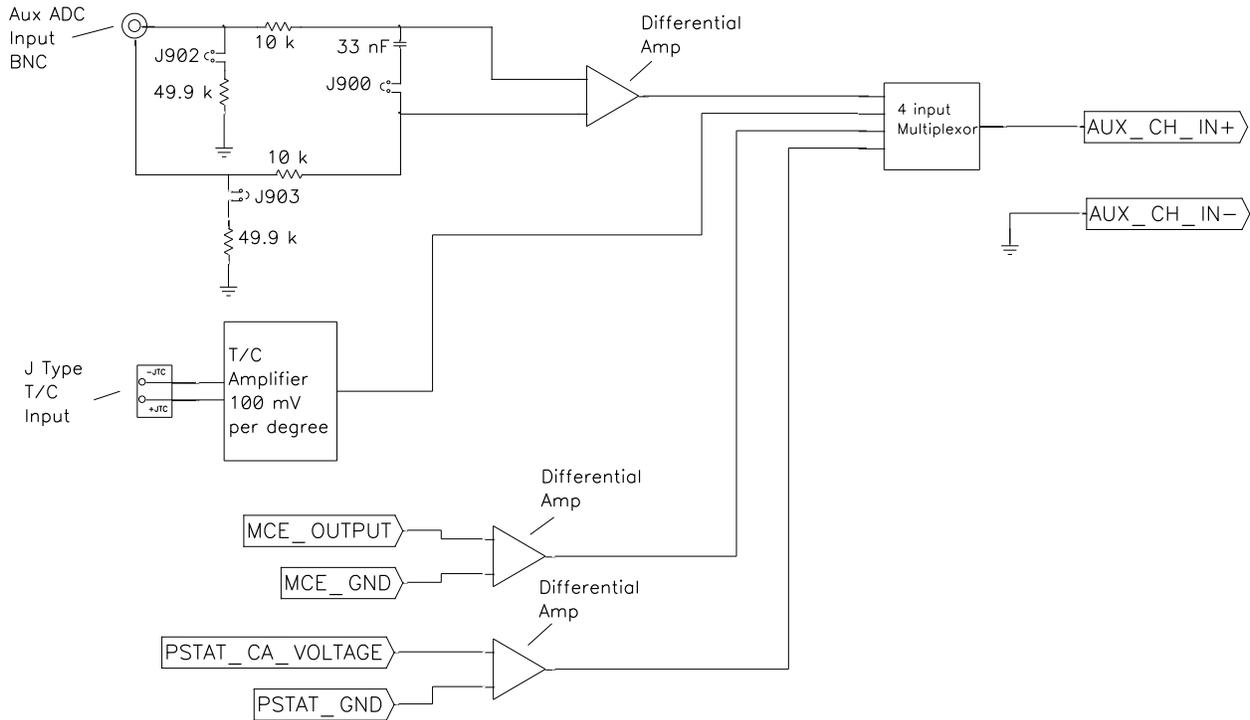
**Figure 3-3**  
**One A/D Signal Chain in the Reference 3000**



Notes for Figure 3-3:

- This diagram shows one of three identical ADC channels. One channel is dedicated to measurement of the potentiostat's current signal, another is used to measure the cell or stack voltage, and the third is switched between a wide selection of possible signals. See Figure 0-4  
 All three A/D converters are triggered simultaneously to start a conversion. This trigger and the pulse updating the Scan DAC voltage are under the control of a hardware state-machine. This insures that all waveform and data-acquisition timing is tightly controlled and reproducible point-to-point.  
 By default, the data acquisition is synchronized with the 300 kHz power-supply switching frequency, to reduce noise from the power supply. Data-acquisition times that are a multiple of 3.333  $\mu$ s maintain this synchronization.
- All analog signals that cross from the Potentiostat Board to the Control Board or vice versa are received differentially as shown here.
- The 5 Hz, 1 kHz, and 200 kHz filters are two-pole Butterworth filters. The 3 MHz RLC filter has an arbitrary transfer function.
- All signal-channel components are selected for optimal DC accuracy, low noise, and high bandwidth.

**Figure 0-4**  
**Aux Channel Input Switching**



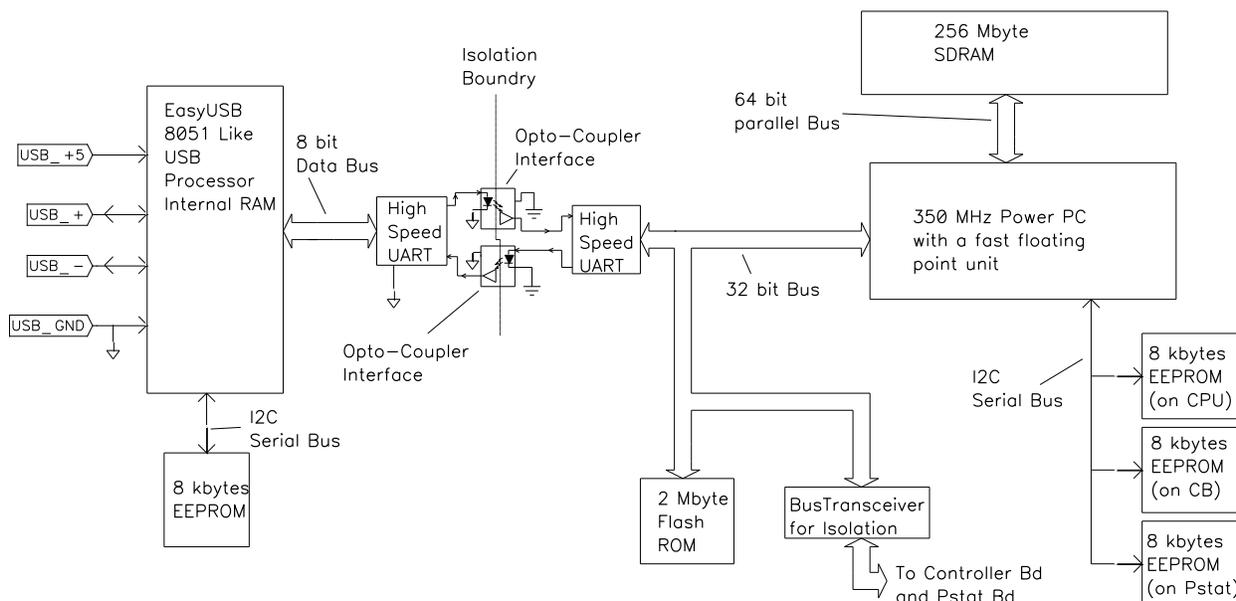
Notes for Figure 3-4:

- Two ADC Channels are dedicated to the potentiostat's voltage and current signals. This diagram shows the signals that can be connected to the third channel.
- The Aux ADC BNC input is a differential input. Some of this input's characteristics can be changed by either jumpers or CMOS switches. Early Reference 3000's use jumpers to change the characteristics, while later units (shipped after the middle of 2009) use CMOS switches under software control to change the characteristics. Contact Technical Support at Gamry.com if you are uncertain which type of unit you have.

Jumper-configured Reference 3000's are shipped configured for input impedance of 100 kΩ and unfiltered operation. The jumpers shown in this diagram allow it to be configured for high input impedance or input filtering.

- The thermocouple input allows for connection of a K-type thermocouple. Note that this circuit must be calibrated to obtain reasonable accuracy. A section of the Reference 3000 calibration procedure allows for user-calibration of this input.
- The other two inputs to the Aux channel can be used to measure the AE (Auxiliary Electrometer) signal or a Control Amp voltage signal.

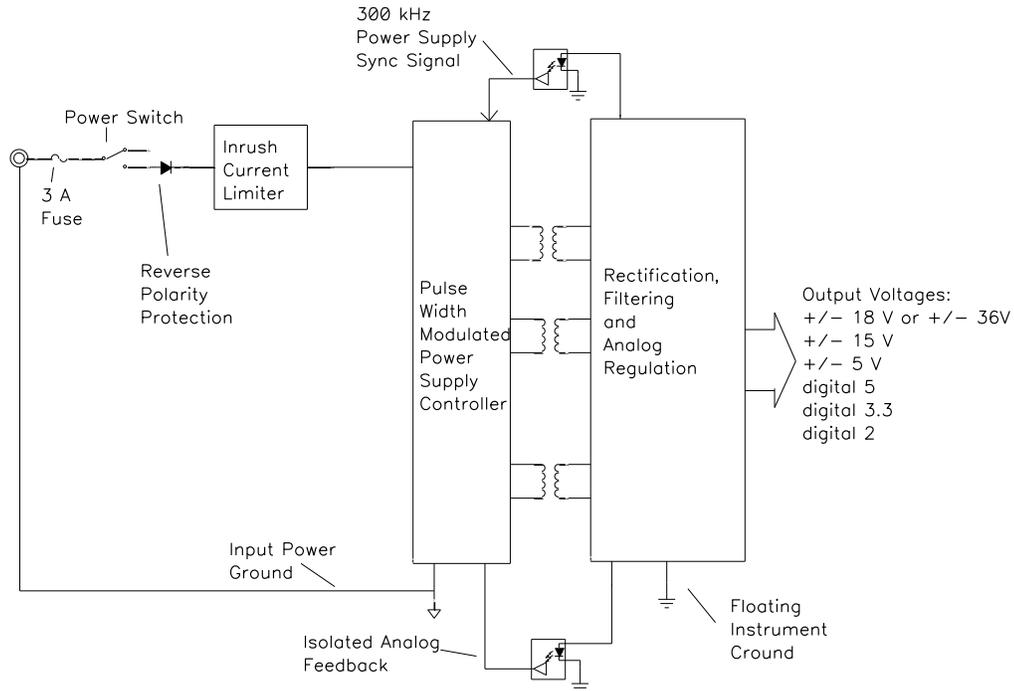
**Figure 3-5**  
**Microprocessors in the Reference 3000**



Notes for Figure 3-5:

- Note the lack of a ground connection between the USB bus and the Reference 3000 circuitry.
- The EasyUSB firmware is loaded into EasyUSB RAM on power-up. The USB firmware can be updated over the USB via a selection in the Reference 3000 section in the Instrument Manager.
- The Power PC firmware is also transferred from ROM into RAM on power-up. The Power PC firmware can also be updated over the USB via a selection in the Reference 3000 section of the Instrument Manager. Time-critical sections of the Power PC code are kept in the processor's fast cache memory.
- The term UART refers to a Universal Asynchronous Receiver Transmitter. It converts parallel data to a self-clocking serial bit stream. The UARTs send data at 6 Mbits/second.
- The Bus Transceiver isolates bus activity on the Controller and Potentiostat boards. Only reads and write-to locations on these boards generate bus activity. This reduces noise pick-up.
- Each board in a Reference 3000 has local non-volatile data storage. This is used to save calibration data and board-revision information. Unlike previous Gamry Instruments potentiostats, the Reference 3000 calibration data is stored in the instrument, not in a data file. When a Reference 3000 is moved from one computer to another, its calibration remains valid.

**Figure 3-6**  
**DC-DC Power Conversion**



Notes for Figure 3-6:

- Note the ground isolation between the input power and the Reference 3000 circuitry. The Reference 3000 chassis is connected to the Floating Instrument Ground. Transformers and isolators are the only components connected between the grounds.
- The 300 kHz power-supply sync signal is derived from the same clock used to control data acquisition. Data points taken at an integer multiple of 3.333  $\mu$ s/point are synchronized with the power supply, minimizing the effect of power-supply noise on the data.
- Additional circuitry that is not shown protects the Reference 3000 against ESD (electrostatic discharge) and electrical surges. Note that the Reference 3000 is also protected against damage if an incorrect polarity power input is connected to the unit.

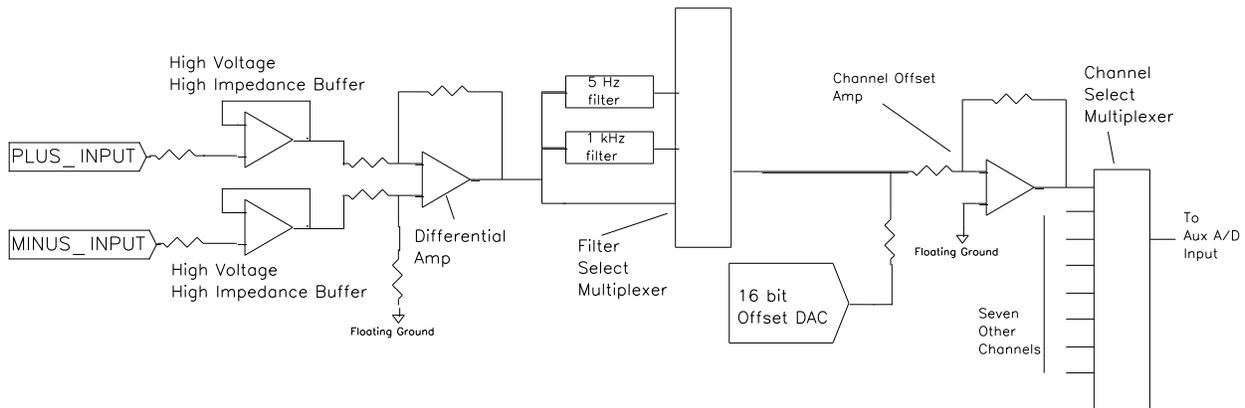


**Caution:** Do not use a DC power source other than the AC-adaptor model provided with your Reference 3000. Other replacements may void the performance and/or safety characteristics of the Reference 3000.

Power input voltages less than 20 V or greater than 26 V can damage the Reference 3000's power supply.

- The incoming DC voltage must be between 22 and 26 V. With inputs below 22 V, the PWM (Pulse Width Modulator) may be unable to regulate the supply. Above 26 V, the PWM may not start up.

**Figure 3-7**  
**One AE Channel**



Notes for Figure 3-7:

- One of eight identical channels is shown.
- The input buffers work over the entire Reference 3000 compliance voltage range.
- The maximum useable differential voltage between the two inputs on a channel is  $\pm 5$  V.
- Each channel has its own filtering. The Aux A/D Channel filter is not useful for switched inputs.
- The  $\times 10$  and  $\times 100$  gains in the Aux A/D channel can be used to improve A/D resolution.
- The inputs have an input impedance of greater than  $10^{11} \Omega$  as long as the product is powered.
- The inputs will not be damaged by connection to voltages as large as  $\pm 36$  V versus the Reference 3000's ground, regardless of the compliance-voltage setting. This is true even when the Reference 3000 is not powered up. They cannot measure voltages larger than the compliance voltage.



## Chapter 4: Installation

This chapter of the Gamry Instruments Inc. Reference 3000 Operator's Manual covers normal installation of the Reference 3000. We assume the Reference 3000 is installed as part of a Gamry Framework-based electrochemical measurement system containing a Microsoft Windows®-compatible computer.

These instructions assume use with Gamry's Framework Software Revision 7.0 or higher.

**Figure 4 - 1**  
**Front View of a Reference 3000 without AE Option**



## Initial Visual Inspection

After you remove your Reference 3000 from its shipping carton, check it for any signs of shipping damage. If you find any damage, please notify Gamry Instruments, Inc. and the shipping carrier immediately. Save the shipping container for possible inspection by the carrier.



**Warning:** The “reinforced insulation” that keeps the operator from accessing the “hazardous live” voltages in the Reference 3000 can be rendered ineffective if the Reference 3000 is damaged in shipment. Do not operate damaged apparatus until a qualified service technician has verified its safety. Tag a damaged Reference 3000 to indicate that it could be a safety hazard.

If the Reference 3000 is taken from a cold location (for example outdoors in winter conditions) to a warm, humid location, water vapor may condense on the cold surfaces inside the Reference 3000, possibly creating a hazardous condition. The “reinforced insulation” that keeps the operator from accessing the “hazardous live” voltages in the Reference 3000 can be rendered ineffective if the Reference 3000 has condensed water inside its case. Before connecting power to a “cold” Reference 3000, allow at least one hour for the Reference 3000 to warm at room temperature.

## Physical Location

Normally you place your Reference 3000 on a flat workbench surface. You need access to the rear of the instrument because some cable connections are made from the rear. The Reference 3000 is generally operated in an upright position (see Figure 4-1). Operation in other positions is possible as long as you insure that air movement through the chassis is not restricted.



**Caution:** Do not block the airflow into or out of the Reference 3000 chassis. While the circuitry should suffer no damage due to excessive heat, the Reference 3000 enclosure may become uncomfortably hot to the touch if no air flows through the chassis. Running the Reference 3000 without adequate cooling could shorten the time to failure of some of the circuitry.

If you place your Reference 3000 within an enclosed space, make sure that the internal temperature within that space does not exceed the 45°C ambient temperature limit of the Reference 3000. Be particularly careful if a computer or other heat-dissipating equipment is mounted in the same enclosure as the Reference 3000.

The Reference 3000 has not been designed for outdoor use.

## Computer Requirements

Before you connect a Reference 3000 to a host computer, you must ensure that your computer meets these simple requirements:

- Microsoft® Windows® 7 or newer is required with Gamry Framework™ software version 7. Framework version 6 is compatible with Windows® XP and newer. Both 32-bit and 64-bit versions of these operating systems are supported.
- A USB port that supports high-speed (480 Mbits/second) USB 2.0 transfers.

Gamry's Windows®-based application software packages may impose additional, more stringent requirements. See the software documentation or contact a Gamry representative for details.

## Quick-start Guide for System Installation

Your shipment includes a short document entitled *Quick-start Installation Guide–USB Potentiostat*. It contains the latest instructions for installing Gamry hardware and software onto a computer system. If this document is missing, the following information should be sufficient for you to install the Gamry Framework Software and Gamry potentiostat onto your computer.

## Software Installation

The Reference 3000 is compatible with the Windows® Plug & Play configuration system. Like most Plug & Play hardware, it is best if you install the software for the Reference 3000 *before* you install the potentiostat hardware.

Gamry Software Setup program normally starts automatically when you place the Gamry Instruments Software disk (or Gamry Instrument Software flash drive) into your computer.

If you have inserted the Gamry CD or Flash Drive into your computer and the Gamry Setup program does not start automatically:

- 1) Navigate to the root folder of the device containing the Gamry Software (CD or flash drive) or to a Windows® folder containing the Gamry Software.
- 2) Run the program called `Autorun.exe` found in this folder.

If you do not know how to navigate to the Gamry Installation device, consult your local computer expert or network administrator or email [techsupport@gamry.com](mailto:techsupport@gamry.com).

## Reboot your Computer after Software Installation

Reboot your computer after the Gamry Setup program is done. The Setup program normally offers you the opportunity to do so. USB device drivers are usually loaded when Windows boots up. Following Setup, you may not be able to use your Reference 3000 until the drivers are loaded.



Device-driver installation may not occur until a while after the Windows® Desktop appears. On a slow computer, or a busy computer with lots of active applications, the delay before driver installation can be a minute or more.

**Figure 4 - 2**  
**Rear Panel of the Reference 3000**



### Power Cord and Power Connection

The Reference 3000 does not plug directly in the AC mains supply. Instead, the mains are connected to an external power supply, which supplies a regulated 24-V DC output. This regulated DC is then connected to the DC **Power In** jack on the rear of the Reference 3000 (see Figure 4-2).

The external power supply provided with the Reference 3000 is rated for operation from 100 to 240 V AC, at frequencies from 47 to 63 Hz. It should be usable worldwide.



**Caution:** If your facility owns both Reference 600's and Reference 3000's, you must insure that the smaller power adapter from the Reference 600 is **not** used to power a Reference 3000. The Reference 3000 will not power up with the smaller adapter. Fortunately, neither the Reference 3000 nor the small power adapter will be damaged if connected in error.

The Reference 3000 external power supply is delivered with a line cord suitable for use in the United States. In other countries, you may have to replace the line cord with one suitable for your type of electrical outlet. You must always use a line cord with a CEE 22 Standard V female connector on the apparatus end of the cable. This is the same connector used on the US standard line cord supplied with your Reference 3000. See Chapter 1 for specific safety information regarding line cord selection.

## Power-up Test

Before you make any other connections to your Reference 3000, check that the Reference 3000 is at least nominally functional.

One quick test is to power up the Reference 3000 and watch the blue **Power** LED indicator on the front panel of the Reference 3000 (see Figure 4 - 1). After connecting DC power to the Reference 3000, turn on its rear panel **Power** switch (see Figure 4-2). The **Power** LED should illuminate for a second or so, flash three times, then remain on. If this sequence doesn't happen, see Appendix F for a list of blink codes.

The status of the other LED indicators is not important at this time.

Each flash of the **Power** LED upon startup corresponds to successful conclusion of one portion of the Power PC's power-up self-test.

If the **Power** LED goes on, then turns off and stays off, the Reference 3000 is not working properly! Contact Gamry Instruments or your local Gamry Instruments representative as soon as possible if this power-up test fails.

## USB Cables

The Reference 3000 connects to the computer using a standard High Speed USB A/B cable. A suitable cable was shipped with your Reference 3000. If this cable is lost, you can get a replacement at almost any computer retailer. The replacement cable should be rated for USB 2.0 High Speed USB operation.

An A/B USB cable has different connectors on each end. The end with a wider, rectangular connector plugs into a USB port on your computer (or a similar port on a USB hub). The end with a nearly-square connector plugs into the USB port on the Reference 3000 (see Figure 0-2).

The USB connection can be "hot-plugged". This means that both the computer and the Reference 3000 can be powered up before the USB cable is plugged in. Unlike many other instrument system connections, you need not power down the system before plugging in the USB.

You may also safely remove the USB cable without powering down the Reference 3000 and your computer. Be aware however, that this may have undesirable consequences if the system is currently taking data or performing an electrochemical experiment.

## Front Panel USB LED

The front panel **USB** LED provides a simple test of two aspects of normal Reference 3000 USB operation. It has three *normal* states:

• Unlit	The USB cable is disconnected or the USB connection is disabled by the host computer,
• Solid green	A valid cable-connection is made and the Reference 3000 USB processor is receiving power from the USB cable,
• Flashing yellow	Valid USB messages are being transferred between the computer and the Reference 3000.

The flashing state is seen only when Gamry Instruments application software is running.

One additional **USB** LED indication is possible. A solid red **USB** LED occurs during a firmware update. The firmware update process is described later in this chapter.

## First-time Device Installation in Windows®



These instructions presume you have already installed Gamry software Revision 7.0 or higher.

### Running the Framework

Regardless of your electrochemical application, Gamry recommends running the Gamry Framework after you install new Framework software or add a potentiostat to your system. The Framework Instrument Manager allows you to:

- Rename potentiostats,
- Calibrate potentiostats,
- Manage potentiostat firmware,
- Authorize specific applications for use with specific potentiostats

Run the Gamry Framework by clicking on the icon it installed on the Windows® desktop. You can connect and power any Gamry potentiostats either before or after you start the Framework.

### Framework Device Status Bar

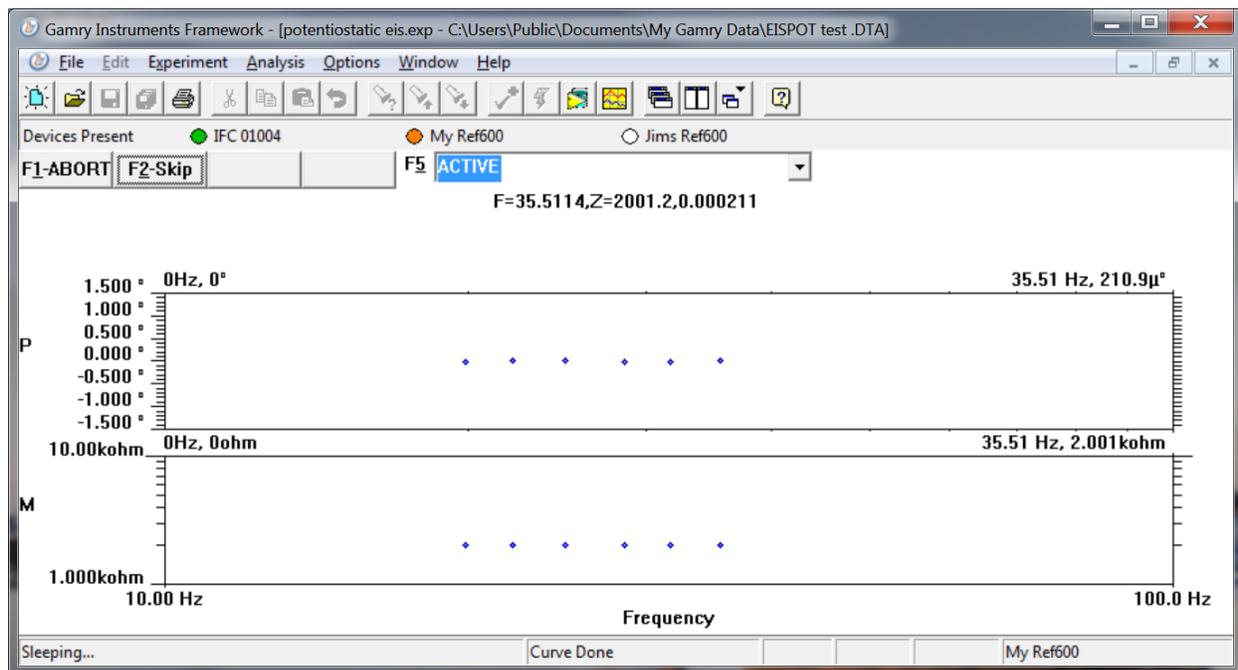
By default, the Gamry Framework shows a Device Status Bar under its main menu (see Figure 4-3). If you don't see the Device Status Bar when you run the Gamry Framework, it has been disabled in the Framework **Options** menu.

Potentiostat Devices (instruments) that are connected to the computer appear on this bar. The round indicator associated with each device shows its status:

<b>Green</b>	The device is available to run experiments
<b>Yellow</b>	The device is currently running an experiment
<b>White</b>	The device is connected to the system, but is not usable. This is generally the result of a revision mismatch between the Framework software and the device's firmware. You can use the Framework Instrument Manager to fix the mismatch.

The screen capture below shows a Framework screen with three USB instruments connected.

**Figure 4-3**  
**Framework With Three Potentiostats and One Running Test**



The Interface 1000 (IFC 01004) in this system is shown with a green indicator because it is installed and ready to run. The Reference 600 labeled **My Ref600** has a yellow indicator because it is recording the EIS spectrum shown on the screen. The Reference 600 labeled **Jims Ref600** has a white indicator, showing it is plugged in but cannot be used. This is an indication of obsolete firmware.

Though no Reference 3000 was used in this example, its status indicator behaves in the same manner described above.

## Gamry Instrument Manager

Use Gamry's Instrument Manager dialog box to make changes to the configuration of your Reference 3000 system. Get access to this dialog box via the **Options** menu in the Gamry Framework.

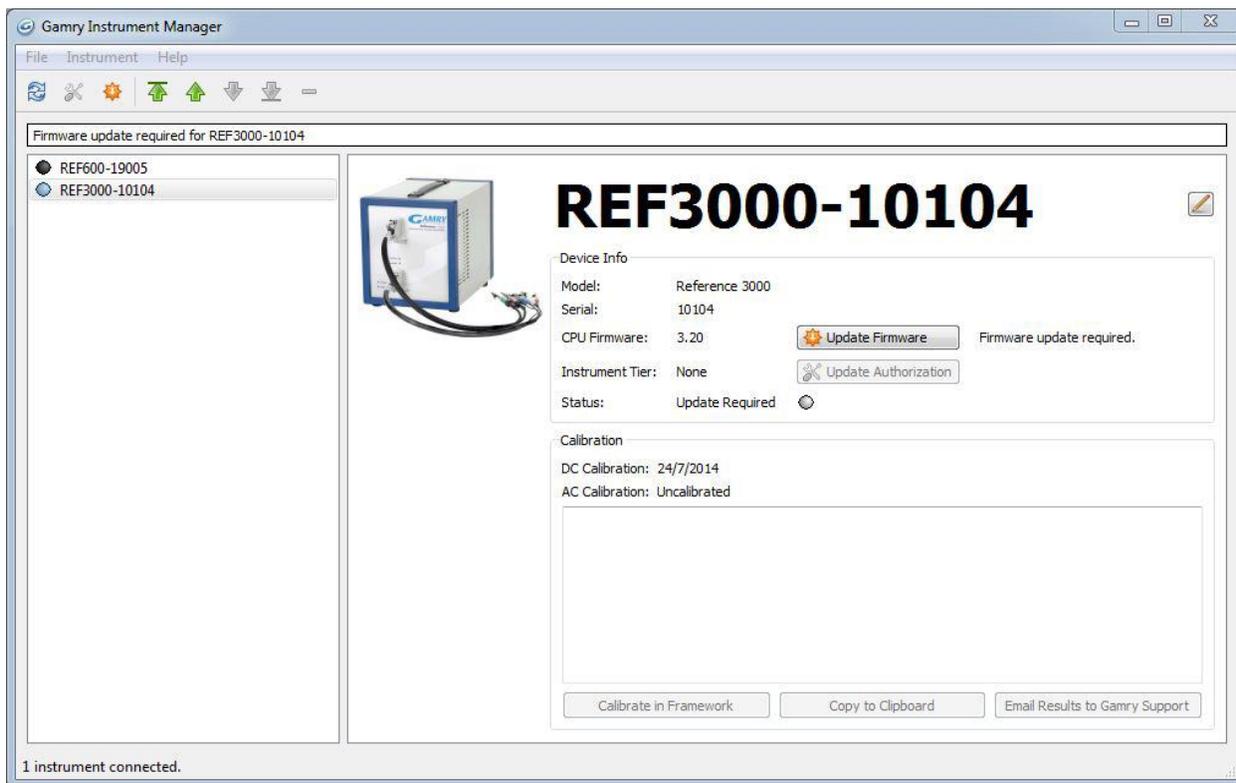
Use the Instrument Manager to:

- Rename potentiostats
- Delete potentiostats that are not currently connected to the computer
- Select the order in which potentiostats appear in menus
- Authorize potentiostat use with applications packages
- Update firmware within potentiostats

Run the Instrument Manger by selecting **Options>Instrument Manager...** on the Framework Menu.

Figure 4-4 shows an **Instrument Manager** dialog box for a two-potentiostat Framework session with a Reference 600+ and a Reference 3000.

**Figure 4-4**  
**Instrument Manager Dialog Box**

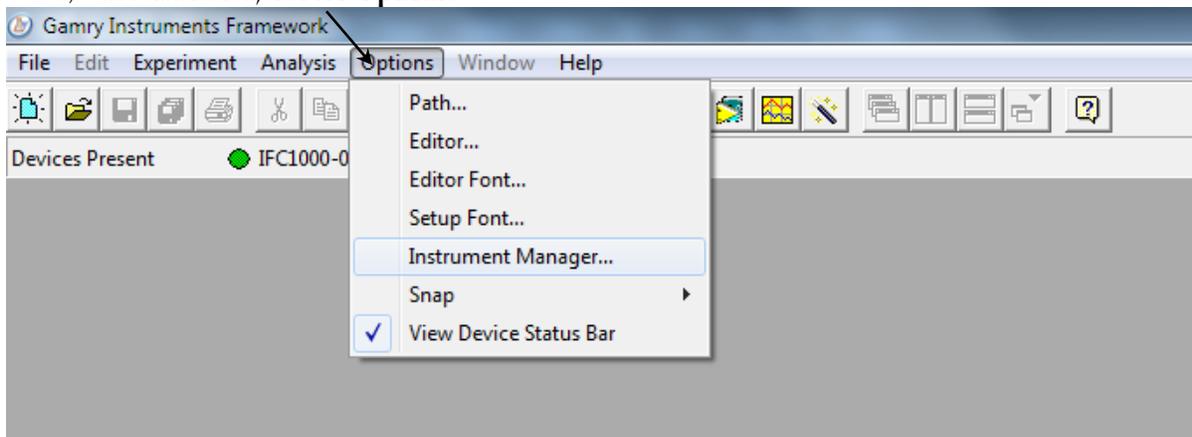


Each Gamry potentiostat in the system appears in the list on the left. All Gamry Instruments potentiostats that are known to the system are displayed in the **Instrument Manager**. Select an instrument by clicking on its name. Selecting an instrument that is connected and idle blinks its Power LED. After a moment, your potentiostat should appear next to **Devices Present** along with a green virtual LED. Repeat for additional potentiostats.

### Changing the Label

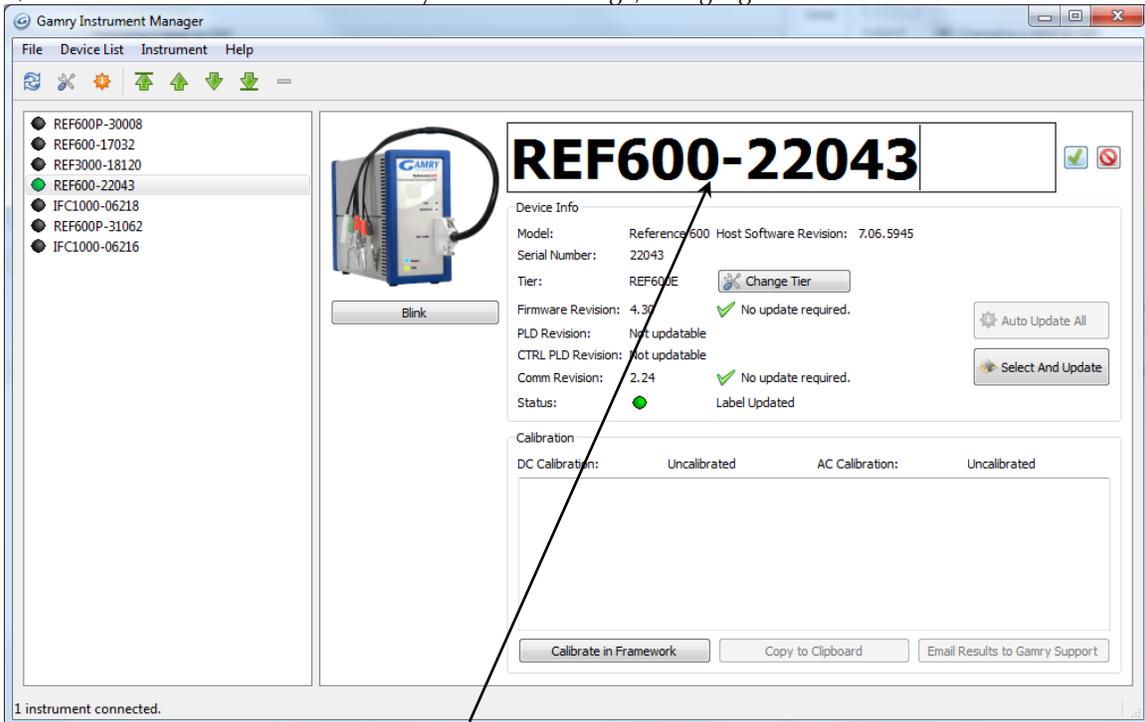
If you purchase additional Gamry Instruments application software, you can do so using the Framework software's Instrument Manager. The dialog box also allows you to change the Label for your device:

- 1) In Framework, choose **Options**.



A drop-down menu appears.

- 2) Choose **Instrument Manager....**  
The **Instrument Manager** window appears.
- 3) Click the instrument whose label you wish to change, to highlight that instrument.



- 4) Click the label itself, and then enter a new, unique name.

## Firmware Update

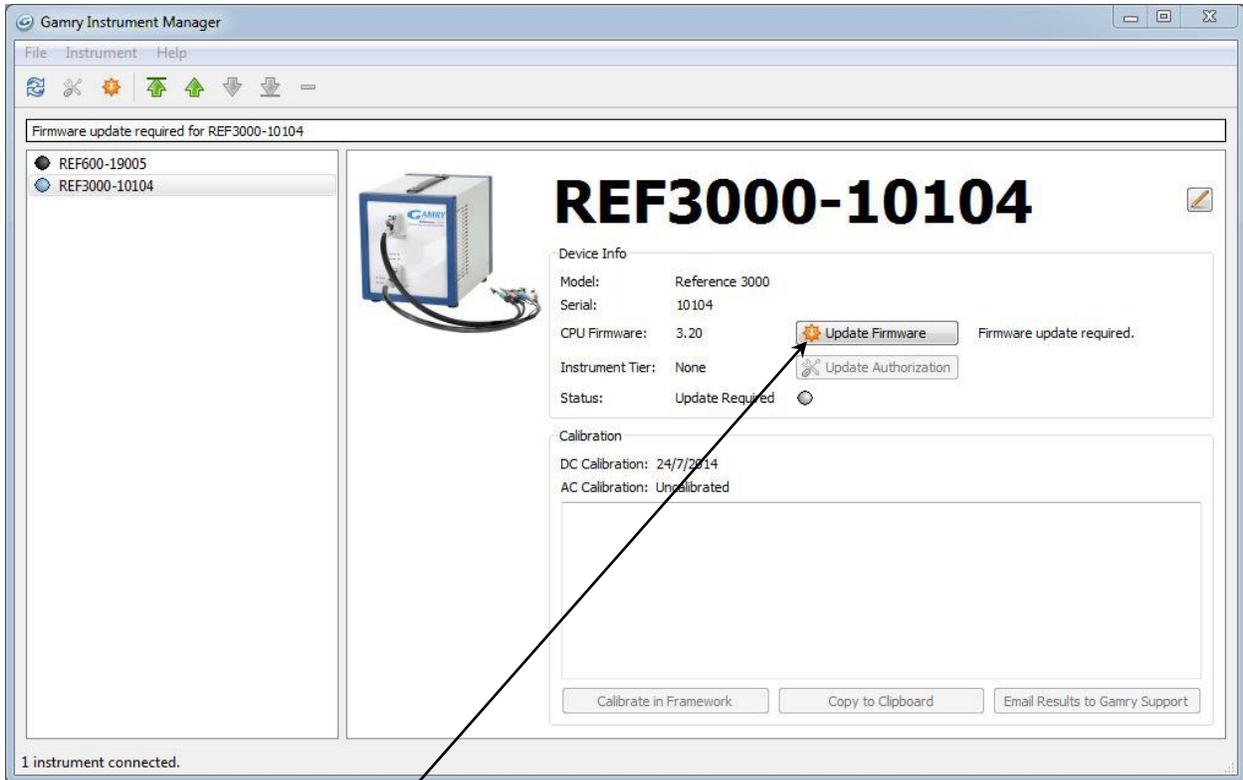
Your Reference 3000 was shipped with the latest version of all its firmware. From time to time, Gamry makes changes to the instrument's firmware code, and a firmware update is required to make use of the new or improved code.

There are two firmware images that you can update on your Reference 3000.

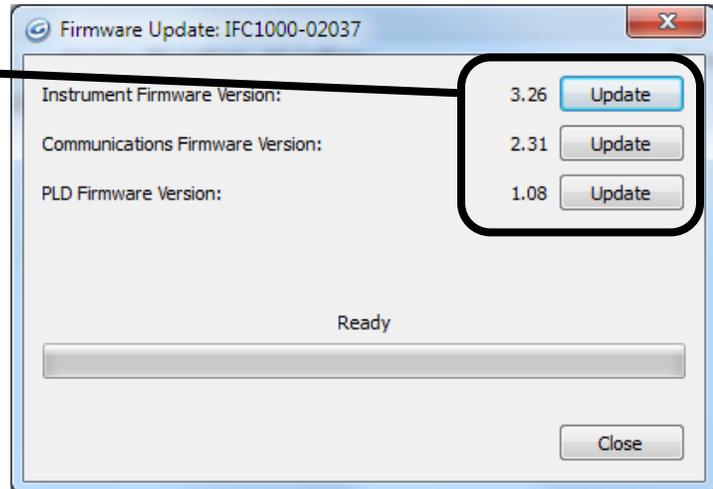
- Instrument Firmware      Handles most of the functions of the Reference 3000.
- Communications          Handles the USB communications between your Reference 3000 and the host  
Firmware                      computer

Appropriate update files (firmware images) can be obtained from the Gamry Instruments website at [www.gamry.com](http://www.gamry.com). Download the file containing the new image and save it onto your computer's hard disk. Alternatively, every Gamry Software disk (or Gamry Software Flash Drive) contains a firmware folder that contains firmware files compatible with that CD's Framework revision.

1. Start the Firmware Update process using the Framework **Options>Instrument Manager...** command
2. In the list on the left side, click on the instrument whose firmware you wish to update, to highlight that instrument.



3. Click the **Update Firmware** button. The **Firmware Update** window appears.
4. Click the **Update** button on the type of firmware you wish to update. You are then prompted for a file. Navigate to the file's location (on the Gamry software disk or on your computer's hard drive) and then click the **Open** button. The update procedure begins. A status bar shows the progress of the update. The **USB** LED on the Reference 3000 should also turn red during the procedure. When the update is successful, click the **OK** button, and your Reference 3000 is ready for use.



## Chapter 5: Calibration

### Introduction

There are three different types of calibration for the Reference 3000 potentiostat:

- Instrument DC and AC calibration
- Cable calibration
- Low I Range DC Calibration

Calibration can be initiated through Framework™ software, or directly through Gamry Instrument Manager (GIM). Gamry recommends calibrating the instrument at least once per year, or when the quality of your data is in question. To initiate calibration, take the following steps:

Click the **Calibrate in Framework** button, located in the Calibration area of GIM. This launches the appropriate calibration routine for the instrument selected in GIM. Follow the instructions given in Framework. See the relevant Calibration Quick Start Guide for additional details.

If there are any failures during calibration, click the **Email Results to Gamry Support** button in GIM. One of our support engineers will review the results and provide appropriate advice.

You run the Instrument Manager by selecting **Options/Instrument Manager...** on the Framework Menu.

Calibrate each potentiostat installed in your system. A calibration utility is provided with the Gamry Framework.

The calibration for the Reference 3000 has been divided into three sections: Instrument DC and AC Calibration, cable calibration, and Low I DC Cal. Gain access to all three calibration procedures via the **Utility** selection on the Framework's **Experiment** drop-down menu.

The Reference 3000 recognizes the type of cable connected to its cell connector. It maintains a separate AC calibration table for each type of cable. The Gamry Framework will not let you use AC calibration data recorded using a 60 cm shielded cable for experiments run using a Twisted Pair cable optimized for low-impedance EIS measurements.

### DC and AC Calibration

These two procedures use an external resistive Calibration Cell (Figure 5-1).

Figure 5-1

2 k $\Omega$  Calibration Cell





**Caution:** The standard Reference 3000 calibration calls for an external resistive dummy cell. Your Reference 3000 was shipped with a Calibration Cell, which includes a 2 k $\Omega$ , 0.05% accurate resistor. After calibration, please place this Calibration Cell in a safe place where you can find it if your unit requires recalibration.

If you do need to recalibrate and you cannot find your Calibration Cell, you can perform DC Calibration using a different 2 k $\Omega$  resistor. Its power-rating is unimportant. Some performance checks in the calibration process may fail if the resistor's inaccuracy exceeds 0.2% (4  $\Omega$ ).

We do **not** recommend AC Calibration with any resistor other than the one on the Calibration Cell. The Calibration Cell was designed to separate the working electrode leads from the counter and reference electrode leads. If you perform AC Calibration without adequate separation between these leads, you will see a phase-shift in your high-frequency EIS data. 1.4 pF of stray capacitance across a 2000  $\Omega$  resistor causes a 1° phase-shift at 1 MHz.

Enclose the resistor used for calibration in a Faraday shield during the calibration process. Connect the Faraday shield to the black lead on the standard cell cable. A Faraday shield suitable for calibration is included in every Reference 3000 shipment.

Earth-grounding the Reference 3000 is also recommended during calibration, but is not essential.

Potentiostat calibration is only required infrequently. Recalibrate your Reference 3000 under the following circumstances:

- It has been about one year since your last calibration.
- Your potentiostat has been serviced.
- You notice breaks or discontinuities in the data-curves recorded with your system.
- The system is being run in an environment that is very different from the previous operating environment. For example, if the Reference 3000 was calibrated at 15°C and you are now operating it at 30°C, you should recalibrate.

## Low I Range DC Calibration

The standard Reference 3000 calibration is performed with the cell leads connected to a 2 k $\Omega$  resistor. During the calibration procedure, DC current-range offsets are recorded with the cell switch turned off. A DC current measurement is made on each of the eleven current ranges in the Reference 3000. The measured current on each range is the sum of current contributions from:

- The input current of the I/E Converter input amplifier,
- The input current of the Working sense input amplifier,
- The input current of the Reference input amplifier,
- The input current of a Counter Sense input amplifier,
- Current leakage in the cell switch.

In most real-world experiments, the cell is turned on, and the I/E converter does not measure the last three terms listed above. These currents still exist, but they are generally sourced by the low-impedance counter-electrode lead. During Low I DC Calibration the first two terms are directly measured in order to improve pA accuracy of the instrument.

The current contributions from each source on the above list are (at most) a few pA, so they are insignificant on all but the most sensitive Reference 3000 current range: the 300 pA range.

This effect can cause differences of up to 8 pA between DC current measured with the cell turned off and current measured with the cell turned on. In most cases the difference is smaller: one or two pA.

Only two Reference 3000 applications are sensitive enough that this current measurement offset causes problems. One is Physical Electrochemistry (Cyclic Voltammetry for example) with small electrodes, and the other is EIS on high-impedance samples such as barrier coatings. Incorrect DC current readings in EIS can slow the experiment, for the automatic ranging algorithms in the Electrochemical Impedance Spectroscopy software can make poor range-choices given incorrect data. This can significantly lengthen the time needed to measure an EIS spectrum.

Most corrosion experiments or macro-electrode measurements involve currents much too large to be affected by this difference.

The Gamry Framework also includes a special “Low I DC Current” calibration procedure that corrects the Reference 3000 offsets to minimize this problem. The procedure uses a script that:

- 1) Asks you to disconnect the reference, counter, and counter sense leads from the calibration cell,
- 2) Measures the I/E input and Working sense amp input currents on the 300 pA range,
- 3) Replaces the 300 pA current range offset measured in the full DC calibration with the improved value.

The error sources listed above are both time- and temperature-dependent, so we recommend frequent “Low I DC Calibration”—if you need accurate measurement of absolute currents at pA levels. The procedure runs fairly quickly, so daily or weekly calibration ought not be too inconvenient.



The Low I DC Calibration is not a full calibration. You must run a **full** DC Calibration on your Reference 3000 **before** you run the Low I DC calibration. Remember that the Reference 3000 must have a full DC calibration on the **same type of cable** you use for the Low I DC Calibration.

## Cable Calibration

This procedure uses the external resistive 2 k $\Omega$  Calibration Cell.





**Caution:** The Reference 3000 cable calibration calls for an external resistive Calibration Cell. Your Reference 3000 was shipped with a 2 k $\Omega$  Calibration Cell including a 2 k $\Omega$ , 0.05% accurate resistor. After calibration, please place this Calibration Cell in a safe place where you can find it if your unit requires recalibration.

Cable calibration is only required infrequently, if you feel that the cable capacitance is a problem for your experiments, especially if you see excessive phase glitches on high-impedance samples.

### Procedure to calibrate the cable

- 1) Connect the **Chassis Ground** on the back of your potentiostat to a known, good earth ground.

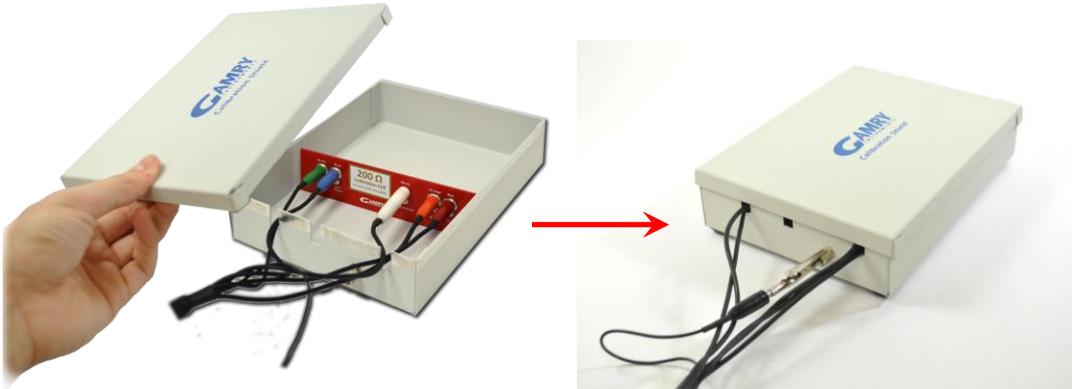


- 2) Connect the cell cable to the correct color-coded receptacles on the 2 k $\Omega$  Calibration Cell.

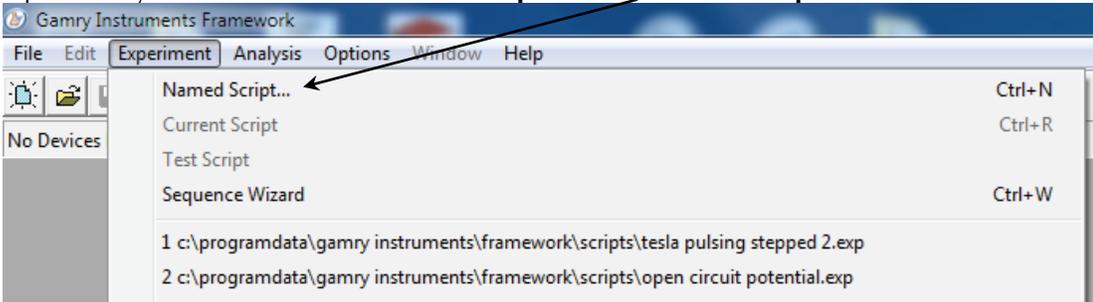


## Calibration--Cable Calibration

- 3) Place the Calibration Cell inside the Calibration Shield, close the lid, and connect the black floating-ground lead of your cell cable to the Shield's grounding post.



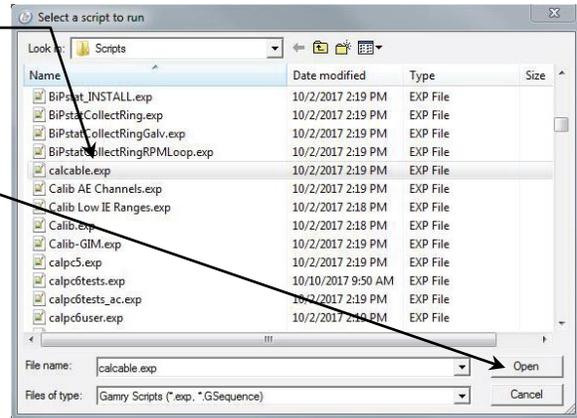
- 4) Open Gamry Framework™ software. Select **Experiment > Named Script...**



The **Select a script to run** window appears.

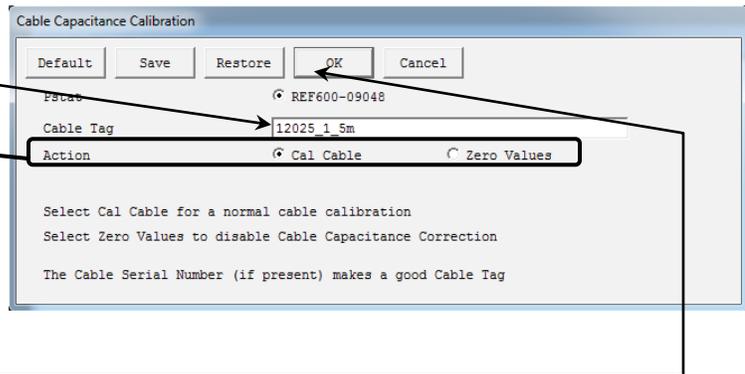
## Calibration--Cable Calibration

From the list of scripts, choose `calcable.exp`, then click the **Open** button.



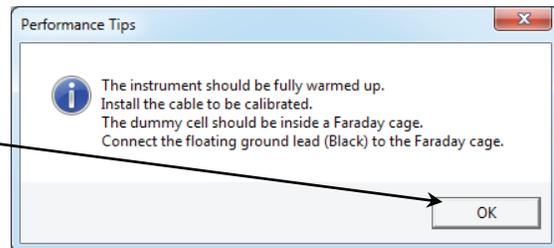
The **Cable Capacitance Calibration** window appears.

- 5) In the **Cable Tag** field, enter a unique name for the cable you are calibrating. Choose the desired **Action** radio button:
- To calibrate the cable, choose **Cal Cable**.
  - To reset the values to zero (if, say, the calibration doesn't work), choose **Zero Values**.
- Click the **OK** button.



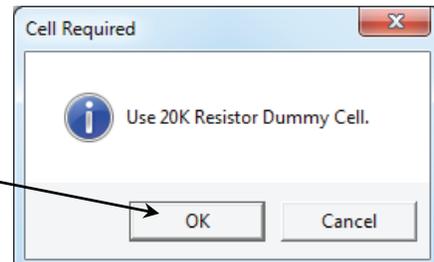
The **Performance Tips** window appears.

- 7) Make sure that all of the tips are true, then click the **OK** button.



The **Cell Required** window appears.

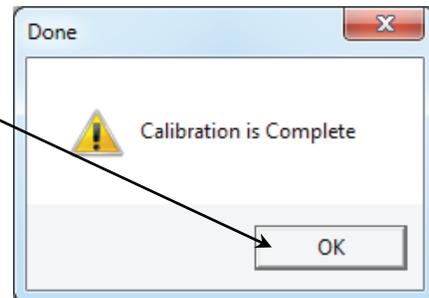
- 8) Make sure that the correct Calibration Cell is attached, then click the **OK** button.



The calibration runs.

The **Done** window appears.

- 9) Click the **OK** button to acknowledge completion.



## Chapter 6: Cell Connections

### Cell Cable Overview

The Cell Connectors are two 15-pin D-connectors on the front of the Reference 3000.

The upper (female) connector is labeled Counter/Working. It carries the cell current between the Counter electrode wire and the Working electrode wires and the instrument

The lower male connector is labeled Sense Inputs. It contains only high-impedance inputs used to sense potentials in the cell.

Gamry's standard cell cables always come in pairs. Each cable has a D-connector on one end and a number of leads to connect to electrodes in an electrochemical cell. The D-connector end of the cable is connected to the appropriate D-Connector on the front of the Reference 3000. The male and female cables cannot be interchanged.

Every Reference 3000 is shipped with a pair of standard shielded cell cables. The Gamry part number for a set of these two cables is cell cables is 985-00111. They are both 60 cm complex cables, with D-connectors on one end, and color-coded banana plugs and pin sockets on the other end.

In some cases, your system may also include special-purpose cell cables. The special- purpose cell cables include documentation describing their use.

Always screw both cell cables into place, because cables can fall off the unit. This can be disastrous if it occurs during an experiment.

### Ancillary Apparatus

Do not use the Reference 3000 with ancillary apparatus connected directly to any of the cell leads. Examples of ancillary apparatus include DVMs, oscilloscopes, chart records, and data loggers. Ammeters and voltmeters, regardless of their specifications, almost always create problems when connected to the Reference 3000 cell leads. Ancillary measurement devices can be connected to monitor points on the rear panel of the Reference 3000.

### AE Connections

The AE (Auxiliary Electrometers) is an option for the Reference 3000. Its inputs often connect to an electrochemical device, such as a battery or fuel-cell stack. The inputs can also connect to external measurement devices, such as a pressure sensor.

AE connections are not described here. See Chapter for AE connection information.

### Fuses in the Cell Cable

The Reference 3000 could be damaged if currents much larger than 3 A were to flow into or out of the Counter electrode or Working electrode leads. Improper connection to a battery, fuel cell, or super-capacitor could cause this type of damaging current to flow. All Reference 3000 Counter/Working cell cables contain fuses in the cable to protect the instrument if it is misconnected. A later section of this manual discusses the fuses and their replacement.

### Normal Cell Connections

This section assumes that you are using standard, shielded cell cables. This information does not depend on the length of the cables.

The cell end of the standard cell cables terminates in a number of banana plugs and two pin jacks. Each termination comes with a removable alligator clip. Table 6-1 identifies the terminals of the cables.

**Table 6-1**  
**Cell-cable Terminations: Potentiostat and Galvanostat Modes (Voltages less than or equal to 12 V)**

Color	Type	Name	Normal Connection
Blue	Banana Plug	Working Sense	Connect to working electrode
Green	Banana Plug	Working Electrode	Connect to working electrode
White	Pin Jack	Reference	Connect to reference electrode
Red	Banana Plug	Counter Electrode	Connect to counter electrode
Orange	Banana Plug	Counter Sense	Used in ZRA mode; connect to counter electrode
Black	Pin Jack	Floating Ground	Leave open or connect to a Faraday shield

Connect both the blue and green cell leads to the working electrode. (The working electrode is the electrode being tested.) The blue pin-jack connection senses the voltage of the working electrode. The green working-electrode connection carries the cell current. The working electrode may be as much as 250 mV above the circuitry ground (floating ground).

Connect the white pin jack to the cell's reference electrode, such as an SCE or Ag/AgCl reference electrode. The measured cell potential is the potential difference between the blue and white cell connectors.

You may need to connect the Reference 3000 to a two-terminal device (such as a commercial battery). In this case, connect both the white cell and red cell leads to one side of the device, and the blue and green cell leads to the other side. Try to connect the white and blue leads as close to the device as possible.

Connect the red banana plug to the counter or auxiliary electrode. The counter electrode is usually a large inert metal or graphite electrode. The counter electrode terminal is the output of the Reference 3000's power amplifier.

The orange lead is only used in ZRA mode and Stack Mode. Automatic switching to ZRA mode is possible if this lead is connected to the counter electrode. If you will not be using ZRA or Stack mode, this lead can be left open or connected to the Counter electrode.

The black pin jack is connected on the Reference 3000 end to Floating Ground. This is the circuitry ground for the analog circuits in the Reference 3000. In most cases, leave this terminal disconnected at the cell end. When you do, take care that its metal contact does not touch any of the other cell connections.

If your cell is a typical glass laboratory cell, all of the electrodes are isolated from earth ground. In this case, you may be able to lower noise in your data by connecting the Reference 3000's Floating Ground to an earth ground.



**Caution:** If any electrode in your cell is at earth ground, **never** connect the Reference 3000 chassis to earth ground. Autoclaves, stress apparatus, and field measurements may involve earth-grounded electrodes. A binding post on the rear panel of the Reference 3000 is provided for this purpose. A water pipe can be a suitable earth ground.



**Warning:** Make sure that your earth-ground connection is made to a legitimate source of earth ground. Consult a qualified electrician if you are uncertain how to obtain an earth ground. Connecting the Reference 3000 to an incorrect and unsafe voltage can create a safety hazard (see Chapter 1 for details).

If you are measuring very small currents, you may find that a metal enclosure completely surrounding your cell (a Faraday shield) significantly lowers measured current-noise. Usually you connect this Faraday shield to both earth ground and Floating Ground. The Floating ground on the black cell lead is a convenient source of ground.

If any electrode in your cell is connected to earth ground, **only** connect your Faraday shield to the black cell lead (Floating Ground).

You can remove the alligator clip on any cell connection to gain access to the underlying banana plug or pin jack. To permanently change the terminations on your cell cable, feel free to remove the banana plugs and replace them with your new termination. Gamry Instruments can also provide additional standard or special cell cables.

## ZRA Mode Cell Connections

The Reference 3000 can function as a precision Zero-resistance Ammeter (ZRA), which maintains two metal samples at the same potential and measures the current flow between the samples. It can also measure the potential of the samples versus a reference electrode.

The cell cable connections for ZRA mode are shown in Table 6-2. The connections are very similar to those for the potentiostat and galvanostat modes. A second working electrode is substituted for the counter electrode, and the Orange Counter Sense lead **must** be connected.

**Table 6-2**  
**Cell Cable Connections for ZRA Mode**

Color	Type	Name	Normal Connection
Blue	Banana Plug	Working Sense	Connect to metal sample #1
Green	Banana Plug	Working Electrode	Connect to metal sample #1
White	Pin Jack	Reference	Connect to a reference electrode
Red	Banana Plug	Counter Electrode	Connect to metal sample #2
Orange	Banana Plug	Counter Sense	Connect to metal sample #2
Black	Pin Jack	Floating Ground	Leave open or connect to a Faraday shield

The counter sense and the working sense lead are each connected to different metal samples. In the ZRA mode, the Reference 3000 is normally programmed to maintain zero volts between these leads. It therefore maintains the two metal samples at the same voltage.

The white pin jack on the cell cable is normally connected to a reference electrode. The potential between this lead and the working sense lead is reported as the cell potential.

If you don't have a reference electrode in your cell, we recommend that you connect the white reference lead to the working electrode. In theory, the measured potential is exactly zero when this is done. In practice, A/D noise and offset will create a small potential signal with a value very close to zero.

### Stack Mode Cell Connections

Batteries, fuel cells, and super-capacitors are often connected with several individual cells connected in series to allow higher-voltage operation. This type of connection is a stack connection. Special experiment scripts allow the Reference 3000 to control and measure stack voltages as large as its compliance voltage ( $\pm 15$  V or  $\pm 30$  V). These scripts refer to Stack Mode cell connections. The cell connections in Stack Mode differ from those in Potentiostatic and Galvanostatic modes.

Voltages in Stack Mode are measured as the voltage difference between the Counter Sense input and the Working Sense Input. A special high-voltage electrometer allows the orange lead to operate at high voltage and still draw minimal current from the system under test.

Connections in Stack mode are very similar to ZRA mode. The cell cable connections for Stack mode are shown in Table 6-3.

**Table 6-3**  
**Cell Cable Connections for ZRA Mode (Voltages greater than 12 V)**

Color	Type	Name	Normal Connection
Blue	Banana Plug	Working Sense	Connect to first end of a stack.
Green	Banana Plug	Working Electrode	Connect to first end of a stack.
White	Pin Jack	Reference	Can be connected to a reference electrode
Red	Banana Plug	Counter Electrode	Connect to the second end of a stack
Orange	Banana Plug	Counter Sense	Connect to the second end of a stack
Black	Pin Jack	Floating Ground	Leave open or connect to a Faraday shield

The Counter Sense and the Counter leads are connected to one end of a stack. The Working and the Working Sense leads are connected to opposite ends of a Stack.

The white pin jack on the cell cable can be connected to a low-voltage point in the stack. Some Gamry scripts allow the voltage difference between the white and blue leads to be read, even though the Reference 3000 is in Stack Mode. The white pin can be connected to a reference electrode—provided the difference between the working and the reference does not exceed 12 V.

The orange lead can be connected to second end of stack or a reference electrode.

If the instrument is connected in stack mode and the reference input is not used, it should be connected to the floating ground wire. A pin-plug shorting bar is provided for this purpose.

If you don't have a reference electrode in your cell, we recommend that you connect the white reference lead to the working electrode. In theory, the measured potential is exactly zero when this is done. In practice, A/D noise and offset create a small potential signal with a value very close to zero.

### Membrane Cell Connections

The Reference 3000 can be used with membrane cells. In this type of cell, a membrane separates two electrolyte solutions. Two reference electrodes are used—one in each electrolyte. Each electrolyte also contains a counter electrode. The Reference 3000 controls the potential across the membrane. Table 6-4 shows the cell connections used with a membrane-type cell.

**Table 6-4**  
**Cell Cable Connections for a Membrane Cell**

Color	Type	Name	Normal Connection
Blue	Banana Plug	Working Sense	Connect to reference electrode #1
Green	Banana Plug	Working Electrode	Connect to counter electrode #1
White	Pin Jack	Reference	Connect to reference electrode #2
Red	Banana Plug	Counter Electrode	Connect to counter electrode #2
Orange	Banana Plug	Counter Sense	Leave open (only needed in ZRA mode)
Black	Pin Jack	Floating Ground	Leave open or connect to a Faraday shield



Reference electrode #1 and counter electrode #1 must be on one side of the membrane, and reference electrode #2 and counter electrode #2 must be on the other side.

### Fuses in the Cell Cable

All standard Reference 3000 Counter/Working cell cables include fuses in the current-carrying leads. These fuses protect the instrument from the extremely large currents that can flow through an improperly connected electrochemical-energy generation or storage device (including batteries, fuel cells, and capacitors). For convenience, the term battery is used here to refer to all single-cell or stacked electrochemical devices that can source energy.

Grounding errors on a battery can be particularly dangerous, because they can result in the battery being shorted through the instrument. During the development of the Reference 3000, several prototype instruments—without fuses—were damaged when connected to a Li-ion battery stack.



**Warning:** The fuses in the Reference 3000 Counter/Working cell cable do **not** protect against a safety hazard. They are needed to prevent damage to the instrument if it is improperly connected.

Both the counter electrode lead and the working electrode lead must be fused. Two different cell-cable fuse arrangements have been built or are planned.

### In-line Fuse-Holders and Fuses

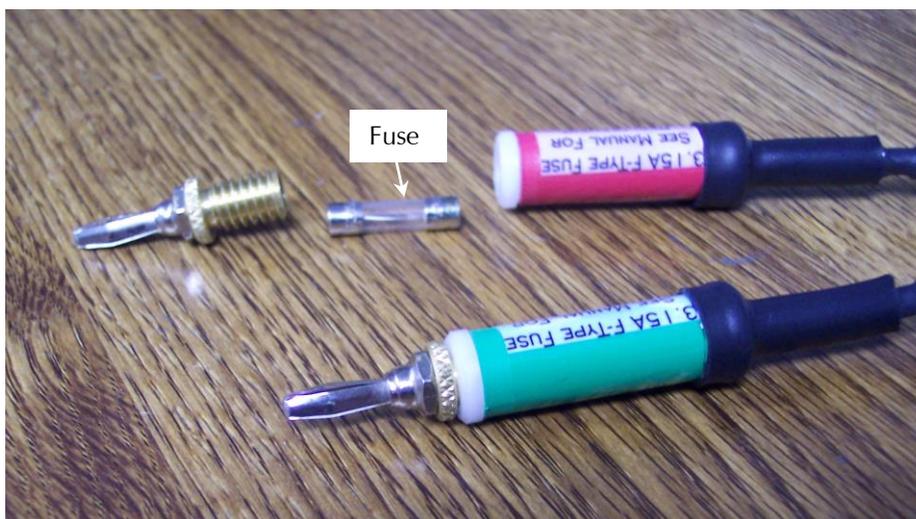
The In-line fuse design is provided with Counter/Working cables supplied with early Reference 3000 shipments. The fuses in the cable are plastic and brass fuse-holders located in-line with the current-carrying leads, as shown in Figure 6-5. The photograph in this Figure shows the Working and Counter electrode leads with in-line fuse-holders. The Counter electrode fuse-holder has been opened to show the fuse. The fuse-holder contains a hidden spring that keeps the fuse in contact with the cell lead.



Always turn off the Reference 3000, and disconnect both ends of the Counter/Working cable before checking or replacing the fuses in the cable.

In this fuse arrangement, you can remove the fuse from the cable by unscrewing the brass knurled nut on the fuse holder, just below the banana plug. After a fuse is removed, you can check it using an ohmmeter, such as that found on modern digital voltmeters. Do not trust a visual inspection of the fuse. A blown (open) fuse should always have a resistance of greater than 100  $\Omega$ . The resistance of a good fuse is very small.

**Figure 6-5**  
**Open Fuse-holder Showing Fuse**



Four replacement fuses should accompany every cell cable shipped by Gamry Instruments. The Gamry Instruments Part Number for the in-line fuses is 630-00019. If you need to buy replacement fuses locally, we currently only recommend Fast Acting (FF), 3.15 A, 5 × 20 mm cylindrical fuses from the Bussman Corporation. The Bussman Part Number is BK/GMA-3.15-R. Fuses with similar ratings from other manufacturers have not been tested, so we cannot recommend their use.



**Caution:** Always replace the fuses in a Reference 3000 cable with the recommended fuse. Use of an improper fuse, especially a fuse with a higher current-rating, could cause instrument failure if a battery cell is improperly connected. Use of a non-approved fuse will void Gamry's factory warranty.

#### **Fuses Located in the Cable Hood**

The in-line fuses in the Counter/Working are expensive and large enough to be awkward, especially when low-impedance connections are required. Newer Counter/Working cables use a simple design with fuses located within the cable hood. This approach is less expensive and allows lower-inductance cell connections.



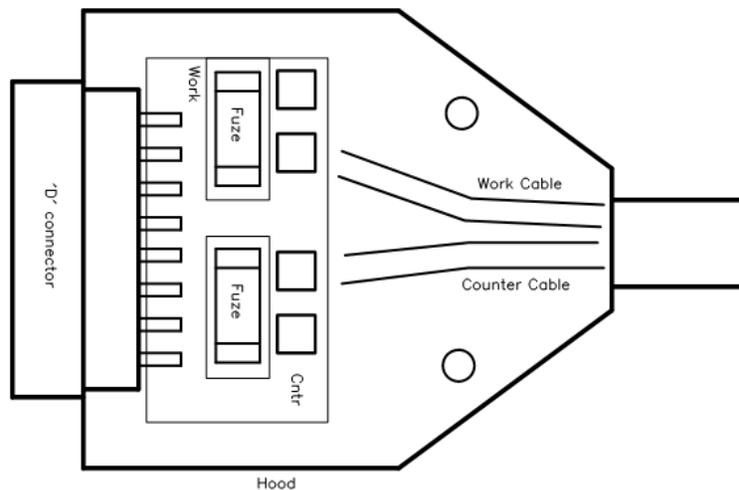
Always turn off the Reference 3000, and disconnect both ends of the Counter/Working cable before checking or replacing the fuses in the cable.

Access to the fuses requires removal of two screws located on opposite sides of the hood covering the D-connector end of the Counter/Working cable. A drawing of the D-connector end of a cable with the hood opened is in Figure 6-6. The small rectangular fuses snap into fuse-holders labeled **Work** and **Cntr**. To remove the fuse, either grip it with small pliers and gently lift the fuse out of the fuse-holder, or pry the fuse out of the fuse-holder using a small screwdriver or knife blade.

After a fuse has been removed, you can check it using an ohmmeter, such as that found on modern digital voltmeters. A blown (open) fuse should always have a resistance of greater than 100  $\Omega$ . The resistance of a good fuse is very small.

Don't forget the jackscrews when you reassemble the hood.

**Figure 6-6**  
**Drawing of Fuse-holder in the D-Connector Hood**



Four replacement fuses should accompany every Counter/Working cell cable shipped by Gamry Instruments. The Gamry Part Number for the small rectangular fuses is 630-00021. If you need to buy replacement fuses locally, we currently only recommend Very Fast Acting, 3.15 A, Nano Fuses from the Littelfuse Corporation. The Littelfuse Part Number is 04513.15MRL. Fuses with similar ratings from other manufacturers have not been tested, so we cannot recommend their use.



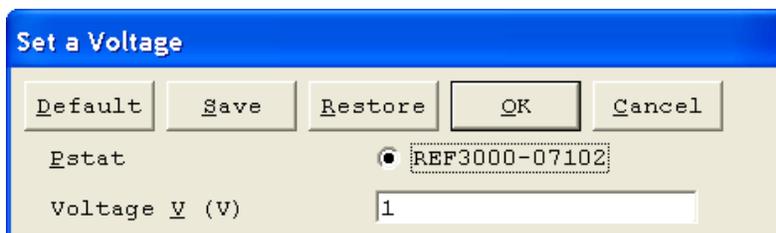
**Caution:** Always replace the fuses in a Reference 3000 cable with the recommended fuse. Use of an improper fuse, especially a fuse with a higher current-rating, could cause instrument failure if a battery cell is improperly connected. Use of a non-approved fuse will void Gamry's factory warranty.

### Testing For Open Fuses

A Gamry Framework test checks for blown fuses without having you remove the fuses. A simple Potentiostatic test is run on the Calibration Cell with the Gamry UDC4 Dummy Cell. The test is run using the “Set a Voltage.exp” script in the Framework’s Utilities Package.

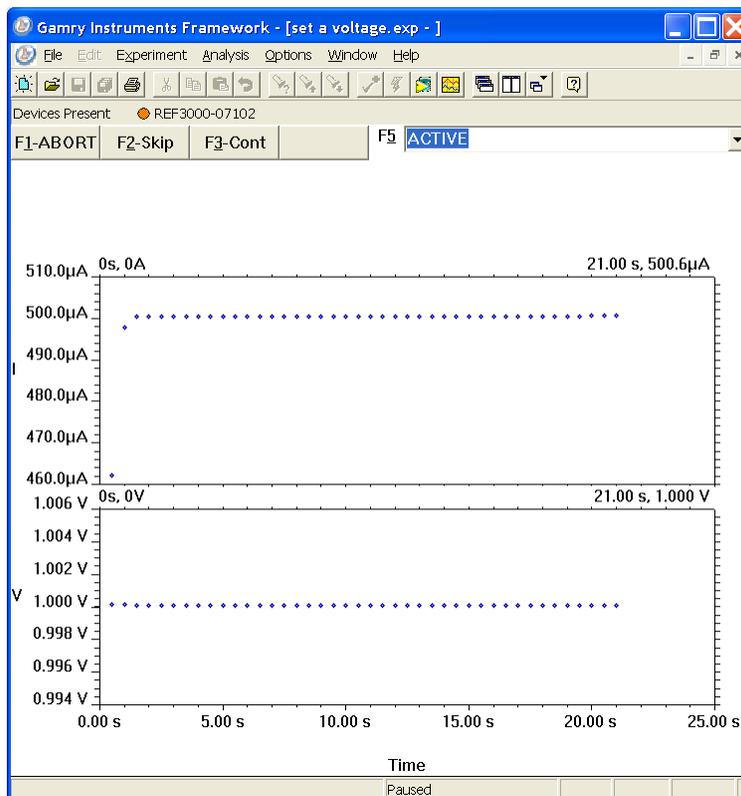
Connect the cell leads to the Calibration side of the UDC4. You do not need to place the UDC4 within a Faraday cage. Select the command **Experiment>Utilities>Set a Voltage** on the Framework’s menu bar. A dialog box similar to this appears:

**Figure 6-7**  
Setup Dialog Box for the “Set a Voltage” Script



Enter a **Voltage** of 1 V as shown above, then click the **OK** button. The Framework opens a runner window, and a graph of current versus time appears.

**Figure 6-8**  
Typical Runner Window with Good Fuses



We expect you to see one of two very different results:

### *Cell Connections--Fuses in the Cell Cable*

- If the instrument is working properly and the fuses in the cable are good, the measured current will be around 500  $\mu\text{A}$  as seen above. No overloads are seen.
- If the one or both fuses are open, all the current readings will be near zero, and a red **CA Overload** indication may be seen at the bottom of the runner window.

If this test indicates an open fuse, use the procedures described above to check **both** fuses. This test cannot tell which fuse is blown. Both fuses can blow at the same time.

If the fuse test indicates an open fuse, and the fuses both check out good with an ohmmeter, some other problem has occurred in the cables or the instrument. Contact Technical Support at Gamry Instruments at your earliest convenience.

*Cell Connections--Fuses in the Cell Cable*

## Chapter 7: Panel Indicators and Connectors

### Front Panel

The Reference 3000 front panel includes two connectors and four backlighted LED indicators.



The cell connections are discussed at great length in Chapter 6. A pin-out diagram of the two cell cable connectors is in Appendix B.

### Counter/Working Connector

The **Counter/Working** connector is a 15-pin female D-type connector. It contains the high-current connections between a Reference 3000 and an electrochemical test cell.

The **Counter/Working** connector is normally connected to a Gamry Instruments-supplied cell cable. Gamry's **Counter/Working** cables always include fuses that prevent instrument failure when a battery or other energy source is improperly connected to the cable.

In addition to the pins used for cell connections, the Reference 3000 **Counter/Working** Connector also uses five pins to read a cell cable ID. Gamry's software can compensate for the cell-cable characteristics for optimal system performance, especially in EIS (Electrochemical Impedance Spectroscopy).

### Sense Inputs Connector



The **Sense Inputs** connector is a 15-pin male D-type connector. It contains high-impedance voltage sense inputs. These inputs are used to measure voltages in the electrochemical test cell.

The **Sense Inputs** connector is normally connected to a Gamry Instruments-supplied cell cable.

In addition to the pins used for cell connections, the Reference 3000 **Sense Inputs** connector also uses five pins to read a cell cable ID. Gamry's software can compensate for the cell-cable characteristics for optimal system performance, especially in EIS (Electrochemical Impedance Spectroscopy).

### The Power LED

The **Power** LED is located on the lower left of the Reference 3000 front panel. It normally glows a steady blue, when the Reference 3000 is turned on and has passed some simple self-tests.

When the Reference 3000 is first turned on, the **Power** LED glows steadily for a second or two, blink three times, and then go to its normal steady blue output. Each blink in this sequence indicates successful completion of a portion of the Power PC's power-up self-test routine.

A Reference 3000's **Power** LED blinks when that instrument is selected in the Framework's Instrument Manager. This allows easy identification of a specific instrument in a MultEchem system without looking at the instrument Serial Label on the bottom of the instrument chassis.

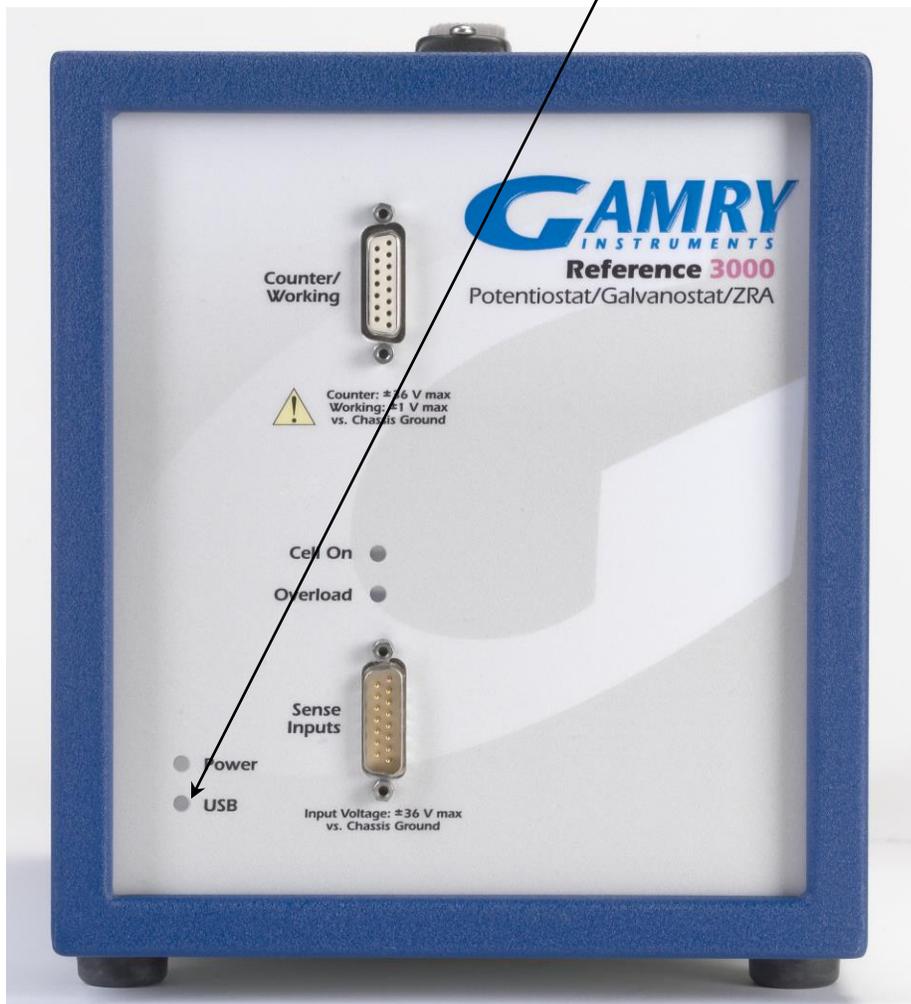
When the **Power** LED is off, one of the following is true:

- The rear-panel power switch is off.
- There is no DC +24 V supply connected to the rear panel DC **Power In** connector.
- The external DC power supply has no input power or is malfunctioning.
- One part of the Power PC's power-up self-test has failed.



**Caution:** The **Power** LED is used both to indicate power status and that power-up tests have passed. It therefore cannot be relied on as a true power-status indicator. Always unplug the DC **Power In** connection if you suspect your Reference 3000 is malfunctioning.

The USB LED



The **USB** LED is located just below the **Power** LED. It is a tri-color LED, capable of glowing green, orange, or red.

The USB LED is unlit when:

- The Reference 3000 is not powered.
- The Reference 3000 does not have a USB cable plugged into its rear-panel **USB** port.

- The computer end of the USB cable is not plugged into a USB port on a computer or hub.
- The USB cable is not supplying USB power to the Reference 3000.
- The computer has disabled the USB port going to the Reference 3000.

The **USB** LED glows a steady green if a valid USB connection is made and the Reference 3000's communication processor is receiving power through the USB cable.

The **USB** LED flashes orange whenever the Reference 3000 is receiving or transmitting valid USB messages to or from the host computer. It does not flash if there is USB traffic addressed to other devices on the USB bus, including messages aimed at a different Reference 3000.

The **USB** LED indicates a solid RED in one special condition: when a firmware download is taking place.



**Caution:** Interrupting a firmware update can cause a catastrophic failure of your system.

Do not turn off the Reference 3000, do not unplug the USB cable, and do not stop the operation of the host computer when the USB LED is a continuous red color.

**Do not interrupt a firmware update that is in progress.** An incomplete update can render a Reference 3000 inoperable until it is returned to Gamry for reprogramming.

Should the firmware update be interrupted, contact Gamry before starting the return process.

## Cell On LED

The **Cell On** LED glows yellow whenever the Reference 3000 is actively applying voltage or current to the electrochemical cell attached to the Cell Cable. Avoid touching the cell cable leads whenever the **Cell On** LED is lighted, because the quality of the data collected in your experiment may be compromised.



The **Cell On** LED does not indicate a dangerous condition when it is lighted. The voltages put out by a Reference 3000 are generally considered safe. Still, avoid touching the cell leads when the cell is on.

If you need to make changes to your cell leads, do this between experiments, when the **Cell On** LED is off and the potentiostat is inactive.

In a typical experimental sequence, the **Cell On** LED is off between experiments and during any open-circuit potential measurements. It glows yellow whenever the cell is polarized.

## Overload LED

The **Overload** LED is normally unlighted. When it glows red, this indicates that some circuit in the Reference 3000 has exceeded its normal operating limit. Conditions that generate Overloads include:

- The absolute value of the differential electrometer output voltage (the difference in voltage between the Working and Reference leads) exceeds 12 V. This condition is known as an E Overload.
- The control amplifier has lost control of the cell.

Remember that the Reference 3000 can be operating with compliance limits of  $\pm 1.5$  A at  $\pm 30$  V or with compliance limits of  $\pm 3.0$  A at  $\pm 15$  V.

The absolute value of the cell current may be trying to exceed the compliance current or the absolute value of the counter electrode voltage may be trying to exceed the compliance voltage setting. Either condition is a Control Overload.

- The absolute value of the cell current has exceeded full scale on the current range presently in use. This condition is known as an I Overload.

An Overload indication does not indicate an instrument failure or system malfunction. Many normal conditions can light the **Overload** indicator.



For example, transient (temporary) overloads **during** an experiment in which the cell voltage or current is being stepped or swept are often normal. Consider the case of an infinitely fast voltage step into a perfect capacitor. In theory, charging the capacitor requires an infinite current. The current spike seen at each step in a stepped-voltage waveform can easily light the **Overload** LED. The current spike normally decays to near zero before the actual current and voltage readings are taken.

Overload indications when the cell is being connected or disconnected are also common, and usually do not indicate a problem. Overloads can also be seen when one of the cell leads is disconnected from the other cell leads, even though the cell is off. Again, this does not indicate a problem.

A steadily glowing **Overload** LED **during** an experiment most likely indicates a problem is occurring. Possible causes include:

- One of the cell leads is disconnected (this is the most common cause),
- A gas bubble in the cell is blocking one of the electrodes,
- The potentiostat could be oscillating (see the next chapter).



A glowing red **Overload** LED does not necessarily indicate a system malfunction. The **Overload** LED can illuminate when one or more cell leads are disconnected, without indicating a problem with the system. The **Overload** LED can often illuminate momentarily during a swept or stepped experiment. The only **Overload** LED indication that definitely points towards a problem is a continuously glowing **Overload** LED during an experiment.

## Rear Panel

The rear panel contains one switch and a large number of connectors. See photograph below.

### Power In Jack

The Reference 3000 derives all its power from a +24 V DC supply connected to the **Power In** jack on the lower right side of the rear panel. The input current is less than 5 A.



The Reference 3000's AC Adapter is rated for operation from 100 to 240 V AC, 47 to 63 Hz. It should therefore be useful throughout the world.

We recommend that you always use the power adapter (power brick) supplied with your Reference 3000 to power to the instrument.

Input power from the power adapter comes from your AC power mains.





**Caution:** If your facility owns both Reference 600's and Reference 3000's, you must insure that the smaller power adapter from the Reference 600 is **not** used to power a Reference 3000. The Reference 3000 will not power up with the smaller adapter. Fortunately, neither the Reference 3000 nor the small power adapter will be damaged if connected in error..

While a Reference 3000 may work with other power sources, we cannot guarantee it will work to its full specifications. If you have to use the Reference 3000 with a different supply, make sure that the supply is regulated, has an output between 22 and 26 V and supplies at least 5 A of load current. Inrush current drawn by the Reference 3000 at "power on" has been known to cause improper operation of an external power supply, even though the supply is rated for more than 5 A of output current.



**Caution:** Power input voltages less than 20 V or greater than 32 V can damage the Reference 3000's DC-DC power supply.

## Power Switch

The **Power** switch is located just below the **Power In** jack. It switches the power from this jack to the input of the Reference 3000's DC-DC converter.

Normally, the DC power is connected before the **Power** switch is turned ON. However, no damage will occur if this switch is already in the ON position when the DC power is connected, or when the AC power input is connected to the external power supply.

## Chassis Ground

The rear panel **Chassis Ground** is intended for one use only. When the Reference 3000 is used with cells isolated from earth ground, connecting the **Chassis Ground** to earth ground may lower the noise measured in the system. Note that the chassis of the Reference 3000 is connected to Floating Ground. See Chapter for safety information concerning this connection.

Either a banana plug or the stripped end of a wire can be connected to the **Chassis Ground** binding post. The other end of the wire is then connected to



earth ground.

A black banana-plug-to-banana-plug lead is provided with your Reference 3000. You may find it useful when making this earth-ground connection.

## USB Port

The **USB** port on the rear panel of the Reference 3000 is a Type B connector as defined in Revision 1.1 and 2.0 of the USB Specification. Use a standard, shielded, Type A/B cable to connect this port to a computer's USB port or a USB hub (preferably an externally powered hub). The two ends of a Type A/B cable are different: the more-rectangular end plugs into the computer, and the more-square end plugs into the Reference 3000.

A suitable USB cable was included with your Reference 3000 shipment. If this cable is lost, replace it with a cable from your local computer retailer.

The Reference 3000 is a High Speed USB 2.0 peripheral, capable of data-transfer at 480 Mbits/second. If it is plugged into a computer port incapable of High Speed operation it will downgrade to USB 1.1 full-speed operation (12 Mbits/second). Obviously data-transfer speed will be slower if this occurs.

The Reference 3000 **USB** port is compatible with Revision 1.1 and 2.0 of the USB specification. It supports the Windows Plug and Play mechanism, including dynamic connect/reconnect.

The front-panel **USB** LED should be green whenever a valid computer to Reference 3000 connection has been made and both the computer and Reference 3000 are fully powered.



## Thermocouple Input

The Reference 3000 has a **T/C Type K** jack for a type K thermocouple. The ISO standard calls for color-coded mini-thermocouple connections. Yellow is the color assigned to type K thermocouples. The mating connector on your thermocouple should therefore be yellow.

Possible uses for temperature measurement in an electrochemical test include:

- Looking for a temperature rise at end-of-charge on a battery.
- Measuring ambient temperature prior to a corrosion measurement.
- Measuring temperature in a cell before making a CV measurement that will be used to calculate reaction kinetics.

Gamry Instruments chose not to provide a thermocouple with the Reference 3000. There is simply too much variety in the mechanical design of thermocouple probes. Commercial thermocouples designed for measurement in air, on solid surfaces, and in immersion service are available from a variety of vendors. Make sure you get a type K thermocouple.

The Reference 3000 uses a temperature-measurement IC to convert the thermocouple output to a usable voltage. It outputs a voltage that is nominally 10 mV per degree Celsius. The IC used, the Analog Device AD594A, is rated for an accuracy of 3°C. Even this accuracy is only achieved when the Reference 3000 is calibrated. The scaling at the A/D converter is  $\pm 3$  V full scale, or  $\pm 300^\circ\text{C}$  full scale.

The Reference 3000 calibration script has an optional section for thermocouple calibration. An ice-water bath and a beaker of boiling water provide convenient standards for a two-point calibration.



**Caution:** One side of the thermocouple is connected to the Reference 3000's Floating Ground. An improper connection to the thermocouple input can compromise the Reference 3000's ability to float, and invalidate data collected on earth-grounded cells. A connection to a non-insulated thermocouple immersed in your electrochemical cell can also cause erroneous readings.

### Misc. I/O Connector

The **Misc.** (Miscellaneous) **I/O** connector is a multipurpose connector. It contains both digital and analog signals used to connect external devices to the Reference 3000.

All of its signals are isolated from both earth ground and the Reference 3000's Floating Ground. The device connected to this connector establishes a ground reference. This isolation allows the **Misc. I/O** connector to be connected to earth-grounded apparatus, without compromising the Reference 3000's ground isolation.

A full description of this connector can be found in Appendix C of this manual. This appendix includes details such as connector pin-out, output and input voltage-levels, and full signal descriptions.



The following list is a short description of the signals in the **Misc. I/O** Connector and their uses:

- Sync Out and Sync In signal allow two or more Reference 3000s to use one data-acquisition clock.
- Four digital outputs can be used to turn on external devices under control of an Explain experimental control script.
- Some of Gamry applications assign three of the digital output to control stirring, flow of de-aeration gas, and formation of mercury drops on a mercury drop electrode.
- Four digital inputs that can be read in an Explain experimental control script.
- A 12-bit D/A converter used to set “continuously variable” settings, such as electrode rotation rate on a rotating disk electrode.
- A 5 V isolated power supply that can provide up to 50 mA of current for external circuitry.



**Caution:** Floating operation of the Reference 3000 can be compromised by improper connections to the **Misc. I/O** Connector. We do not recommend use of standard 15-pin shielded cables with this connector. Custom cables with the shield connected to pin 6 of the D-connector are preferred.

### I Monitor BNC

The **I Monitor BNC** connector represents the output of the Reference 3000's current measurement circuit. With the exception of the filtering described below, it is the raw signal. It is high bandwidth on the less-sensitive current ranges. The effective bandwidth of the current signal falls as you reach the nA and pA current ranges. IE Stability capacitors further slow the response.

The outer shell of this BNC connector is connected to the Reference 3000's floating ground.



**Caution:** The shell of the **I Monitor BNC** is connected to the Reference 3000's Floating Ground. Connection of this BNC to a piece of earth-ground-referenced equipment can compromise the Reference 3000's ability to float, and invalidate data collected on earth-grounded cells.



Scaling on this signal is  $\pm 3$  V for  $\pm$  the nominal full scale current on the selected current range. Cathodic currents cause a positive output voltage. If the software is auto-ranging the current-range selection, this signal is discontinuous at each range change.

The **I Monitor** BNC connector is lightly filtered using an RLC circuit. It has a bandwidth of approximately 3 MHz when connected to a high-impedance input. This bandwidth is further reduced if a coaxial cable is connected to the BNC. Its output impedance is approximately 200  $\Omega$  in parallel with 220 pF.

### E Monitor BNC

The **E Monitor** BNC connector is the output of the Reference 3000's differential electrometer circuit. With the exception of the filtering described below, it is a buffered representation of the voltage difference between the white and blue cell cable leads. It has a high bandwidth.

The outer shell of the BNC connector is connected to the Reference 3000's floating ground.



**Caution:** The shell of the **E Monitor** BNC is connected to the Reference 3000's Floating Ground. Connection of this BNC to a piece of earth-ground-referenced equipment can compromise the Reference 3000's ability to float, and invalidate data collected on earth-grounded cells.

The **E Monitor** BNC connector is lightly filtered using an RLC circuit. It has a bandwidth of approximately 3 MHz when connected to a high-impedance input. This bandwidth is further reduced if a coaxial cable is connected to the BNC. Its output impedance is approximately 200  $\Omega$  in parallel with 220 pF.

### Ext. Sig. In BNC

The **Ext. Sig. In** BNC connector allows you to add a voltage to the Reference 3000's signal generator. This signal is summed with the other signal-generator sources, including the IR DAC, the Scan DAC, and the DDS output.

The outer shell of the BNC is connected to the Reference 3000's floating ground.



**Caution:** The shell of the **Ext. Sig. In** BNC is connected to the Reference 3000's Floating Ground. Connection of this BNC to a piece of earth-ground-referenced equipment can compromise the Reference 3000's ability to float, and invalidate data collected on earth-grounded cells.

The signal generator output is usually directly connected to the potentiostat's input. When the cell is turned on in potentiostat mode, the feedback is such that a negative signal-generator output creates a positive differential electrometer signal, which corresponds to a negative working electrode versus reference electrode voltage.



The polarity of the **Ext. Sig. In** signal is inverted at the signal generator's output. As described above, a negative input signal on this BNC creates a positive change in the working electrode versus reference electrode voltage. The input impedance of this signal is 3 k $\Omega$  in parallel with 15 pF.

### Sig Gen. Out BNC

The **Sig Gen. Out** BNC connector allows you to monitor the "signal generator" signal being sent from the Reference 3000's controller board to the potentiostat board. This signal has a high bandwidth. The signal output range is -15 V to +15 V.

The outer shell of the BNC is connected to the Reference 3000's floating ground.



**Caution:** The shell of the **Sig. Gen. Out** BNC is connected to the Reference 3000's Floating Ground. Connection of this BNC to a piece of earth-ground-referenced equipment can compromise the Reference 3000's ability to float, and invalidate data collected on earth-grounded cells.

The **Sig. Gen. Out** BNC connector is lightly filtered using an RLC circuit. It has a bandwidth of approximately 3 MHz when connected to a high-impedance input. This bandwidth will be further reduced if a coaxial cable is connected to the BNC. Its output impedance is approximately 200  $\Omega$  in parallel with 220 pF.

### Aux. In BNC

The **Aux. In** BNC connector allows you to measure a voltage from outside the Reference 3000 using the Reference 3000's internal A/D. The scaling is  $\pm 3$  V in equals  $\pm 30\,000$  A/D counts. This is a resolution of 100  $\mu$ V per bit. The results are reported in volts. The input is differential (see Appendix D).

The allowed input voltage range is  $\pm 5$  V.

Consult Appendix D for additional information concerning this connector.

### Expansion Interface

The expansion interface is a D-connector on the Reference 3000 reserved for use with the Gamry Reference 30k Booster to provide additional cell current up to 30 A. A specially designed digital cable connects the Expansion Interface port to the booster. Consult the Reference 30k Booster manual for the details of setting up a booster.




**Caution:** Voltages outside the **Aux. In** range of  $\pm 5$  V could damage the Reference 3000 circuitry.

## Chapter 8: Auxiliary Electrometer Option

### Overview

The AE is a factory-installed option for the Reference 3000. The AE acronym stands for Auxiliary Electrometer.

The AE allows eight independent, high-voltage differential electrometer channels available to be read by the Reference 3000's A/D converter. Difference voltages (between the two inputs of each channel) of up to  $\pm 5$  V can be measured.

All AE inputs are rated to operate at all voltages available at the Reference 3000's Counter Electrode terminal. This allows operation between  $-18$  V and  $+18$  V in the 3 A/15 V compliance setting and between  $-36$  V and  $+36$  V in the 1.5 A/30 V compliance setting. These voltages are all versus the Reference 3000's Floating Ground.

The channels are all completely independent. One channel can measure the difference between  $-1$  V and  $-2$  V while a different channel measures the difference between 30 V and 31 V.

The primary function of the AE is simultaneous measurement of individual cells within a multi-cell battery, fuel cell, or super-capacitor stack. Both AC parameters and DC performance of the cells can be measured. The stack is often polarized using galvanostatic control. Alternatively, you can use the Reference 3000 in **Stack** mode to control the voltage of the entire stack or of one cell in the stack.

Regardless of the control mode used, the same cell current flows through all the cells in the stack. As a result, we only need voltage measurements to measure the current and voltage of each cell in the stack. You can also use the AE to use to measure non-electrochemical signals. The voltage input to an AE channel can be the output from a temperature, pressure, or other transducer.

### AC Performance and CMRR

Each AE channel has 2 differential inputs. The channel measures the difference voltage between these inputs, labeled as the **+input** and the **-input**.

The AE inputs can operate with input voltages as high as 36 V and can still maintain pA-level input currents. They are also capable of high-speed measurements. The AE is specified to have less than  $2^\circ$  of phase-shift for 100 kHz input signals applied to an input channel.

Another very important—although often disregarded—specification for differential inputs is common-mode rejection (CMR). CMR is a measure of how well differential inputs reject a signal applied equally to both inputs (often called a common-mode input). The ratio of output voltage to common-mode voltage is called the common-mode rejection ratio, CMRR. By convention, it has units of dB, a logarithmic scale where 20 dB represents a factor of 10. Assuming one volt of common-mode voltage, 20 dB of CMRR corresponds to 100 mV of output voltage, 40 dB corresponds to 10 mV, 60 dB corresponds to 1 mV, etc.

CMR is generally dependent on frequency. As frequency increases, CMRR falls. Each AE channel is specified to have better than 94 dB CMRR at frequencies between DC and 5 kHz, and better than 76 dB of CMRR between 5 kHz and 100 kHz.



AC CMR is highly dependent on resistance in the measurement leads. The AE's CMR specifications only apply when there is less than  $10 \Omega$  of resistance in the input leads. A typical aqueous reference electrode has  $500 \Omega$  of resistance. Gamry does not recommend use of reference electrodes in high-frequency AE measurements.

CMR is especially important when the AE is used to measure the voltage of individual cells in a battery or fuel-cell stack. Cells near the Working Electrode have relatively low DC voltages, because the working electrode voltage is near ground. Cells near the Counter electrode have higher DC voltages. When an AC signal is applied to the stack, the counter-electrode end of the stack has higher AC voltages.

Let's look at a hypothetical example. A battery stack has 22 cells, with an average DC cell voltage equal to 1.5 volts. The bottom cell in the battery stack is attached to the working electrode, and the top cell is attached to the counter electrode. A 100 kHz AC signal is applied, creating an average AC voltage of 10 mV<sub>rms</sub> per cell.

Assume the working electrode is at zero volts versus floating ground. The DC voltages on the top cell are 31.5 V and 33 V. This can also be described as 31 V of common-mode voltage plus a 1.5-volt differential voltage. The AC voltages on the top cell are 210 mV of common-mode voltage and 10 mV of differential voltage.

Applying the 94 dB minimum low-frequency CMRR specification, we can calculate the DC error in the voltage caused by the DC common-mode voltage.

$$\text{Maximum DC Error} = V_{\text{cm}} / \text{CMRR} = 31 \text{ V} / 94 \text{ dB} = 31 \text{ V} / 50\,000 = 620 \mu\text{V}.$$

This is quite small compared to the 1.5 V cell voltage, so we can ignore it.

At 100 kHz, the CMRR is specified to be at least 74 dB.

$$\text{Maximum AC error} = V_{\text{cm}} / \text{CMRR} = 0.21 \text{ V} / 74 \text{ dB} = 0.21 \text{ V} / 5000 = 42 \mu\text{V}.$$

If this AC error is 90° out of phase with the true AC voltage on the cell, it creates a phase error of 0.2° in the measured AC voltage. This is not significant, because the AC accuracy specification at 100 kHz is ±2°.

## Experiments

The AE option is supported only by the Galvanostatic EIS, Galvanostatic Single-Frequency EIS, and Hybrid EIS Framework scripts, as well as the experiments performed through Gamry Electrochemical Energy Software.

## Connections Using Standard Cables

The AE connects to an electrochemical cell using one, two, three, or four cables. Other than labeling, all four cables are identical. Each cable supports two AE channels.



The cables are interchangeable, but we do not recommend using a numbered cable in a differently-numbered connector. The connections get much too confusing.

Table 8-1 shows the pin-out of these cables.

**Table 8-1**  
**AE Cable Connections**

Pin	Type	Name	Wire Color	Connector Color	Normal Connection
1	Analog Input	Odd Ch –	Black	Yellow	Low side of odd channel reference side
2	Analog Input	Odd Ch +	Black	Purple	High side of odd channel working side
3	Ground	Floating Ground	--	--	
4	Analog Input	Even Ch –	Red	Yellow	Low side of even channel reference side
5	Analog Input	Even Ch +	Red	Purple	High side of even channel working side
6	Ground	Floating Ground	--	--	
7	Digital input	!Cable			Ground if cable present
8	Digital In	!Power On			Ground if AE powered
9	Ground	Floating Ground			

Note that pins 8 and 9 **must** be grounded on any cable that is used to take voltage readings.

The analog inputs are both labeled (with labels on the wires) and color coded. The wires are black for odd-numbered channels (Channel 1, 3, 5, and 7) and red for the even-numbered channels (Channels 2, 4, 6, and 8).

The working side plug (the positive input) is purple, and the reference side (the negative input) is yellow.

### Connections Using Custom Cables

You can build custom cables for AE connections. Follow the pin designations in the table above. Pins 7 and 8 must be grounded.

### AE Specifications

See Appendix E.



## Chapter 9: Stability in Potentiostat Mode

### Capacitive Cells and Stability

All potentiostats can become unstable when connected to capacitive cells. The capacitive cell adds phase-shift to the potentiostat's feedback signal (which is already phase-shifted). The additional phase-shift can convert the potentiostat's power amplifier into a power oscillator.

To make matters worse, almost all electrochemical cells are capacitive because an electrical double-layer forms next to a conductor immersed in a solution.

Potentiostat oscillation is an AC phenomenon. However, it can affect both AC and DC measurements. Oscillation often causes excessive noise or sharp DC shifts in the system's graphical output. The Reference 3000 potentiostat can be stable on less-sensitive current ranges and unstable on more-sensitive current ranges. Whenever you see sharp breaks in the current recorded on the system, you should suspect oscillation.

The Reference 3000 has been tested for stability with cell capacitors between 10 pF and 0.1 F. In all but its fastest control amp speed setting, it is stable on any capacitor in this range—as long as the impedance in the reference-electrode lead does not exceed 20 k $\Omega$ . With reference-electrode impedances greater than 20 k $\Omega$ , the Reference 3000 may oscillate. The RC filter formed by the reference-electrode impedance and the reference terminal's input capacitance filters out the high-frequency feedback needed for potentiostat stability.

Longer cell cables make the problem worse by increasing the reference terminal's effective input capacitance.

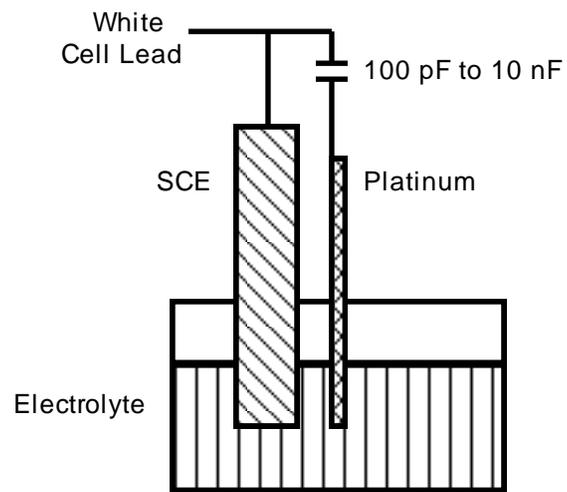
Even when the system is stable (not oscillating), it may exhibit ringing whenever there is a voltage step applied to the cell. The Reference 3000's D/A converters routinely apply steps, even when making a pseudo-linear ramp. While this ringing is not a problem with slow DC measurements, it can interfere with faster measurements. The steps taken to eliminate potentiostat oscillation also help to minimize ringing.

### Improving Potentiostat Stability

There are a number of things that you can do to improve an unstable or marginally stable Reference 3000 potentiostat/cell system. This list is not in any particular order. Any or all of these steps may help.

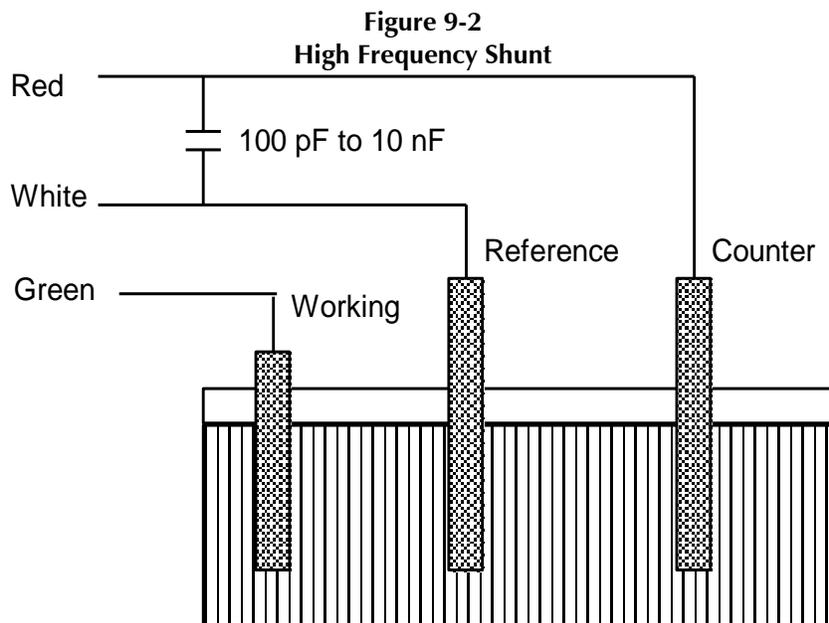
- Slow down the potentiostat. The Reference 3000 has five control-amplifier speed settings, which you select in the software. Slower settings are generally more stable.
- Increase the Reference 3000's I/E stability setting. The Reference 3000 includes three capacitors that can be placed in parallel with its I/E converter resistors. These capacitors are connected to relays that are under software control. Contact your local Gamry Instruments' representative for more information concerning changes in these settings.
- Lower the reference-electrode impedance. Make sure that you don't have a clogged reference-electrode junction. Avoid asbestos-fiber reference electrodes and double-junction electrodes. Avoid small-diameter Luggin capillaries. If you do have a Luggin capillary, make sure that the capillary's contents are as conductive as possible.
- Add a capacitively-coupled low-impedance reference element in parallel with your existing reference electrode. The classic fast combination reference electrode is a platinum wire and a junction-isolated SCE. See Figure 9-9. The capacitor insures that DC potential comes from the SCE, and AC potential from the platinum wire. The capacitor value is generally determined by trial and error.

**Figure 9-1**  
**Fast Combination Reference Electrode**



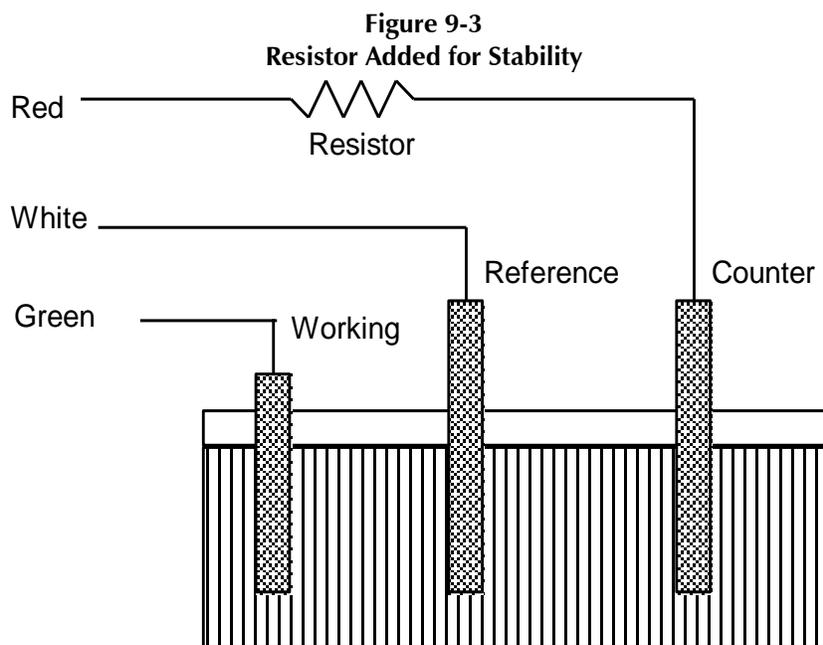
- Provide a high-frequency shunt around the cell. A small capacitor between the red and white cell leads allows high-frequency feedback to bypass the cell. See Figure 9-2. The capacitor's value is generally determined by trial and error. Ten nF (10 000 pF) is a good starting point.

In a sense, this is another form of an AC-coupled low-impedance reference electrode. The counter electrode is the low-impedance electrode, eliminating the need for an additional electrode in the solution.



- Add resistance to the counter-electrode lead. See Figure 9-3. This change lowers the effective bandwidth of the control amplifier. As a rule of thumb, select the resistor to give one volt of drop at the highest current expected in the test being run. For example, if you expect your highest current to be around 1 mA, you can add a 1 k $\Omega$  resistor.

This resistor has little or no effect on the DC accuracy of the potentiostat. It can create problems in high-speed experiments such as fast CV scans or EIS, which need high bandwidth.



## Chapter 10: Measurement of Small-current Signals

### Overview

The Gamry Instruments Reference 3000 is a high-performance measurement instrument used for all types of electrochemical testing. Unlike many other electrochemical instruments, it offers outstanding performance for both tests with small-current signals and high impedances, and for tests involving large currents and very small impedances.

Chapter is a discussion of the latter problem, when currents are large, voltages and cell impedances are small, and inductance limits measurements. These measurements include those encountered in research on:

- Batteries
- Fuel cells
- Super-capacitors

Problems in the measurement of very small currents are discussed here. Examples of this type of testing include:

- Cells for testing painted metal samples
- Cells for testing corrosion of bare metals
- Microelectrode cells
- Most cells for fast CV (cyclic voltammetry)
- Super-capacitors

## Description of the Problem

The Reference 3000 is a very sensitive scientific instrument. It can theoretically resolve current changes as small as 100 femtoamperes ( $1 \times 10^{-13}$  A). To place this current in perspective, 100 fA represents the flow of only about 600 000 electrons per second!

The small currents measured by the Reference 3000 place demands on the instrument, the cell, the cables, and the experimenter. Many of the techniques used in higher-current electrochemistry must be modified when used to measure pA currents. In many cases, the basic physics of the measurement must be considered.

This chapter discusses the limiting factors controlling low-current measurements. It includes hints on cell and system design. The emphasis is on EIS (Electrochemical Impedance Spectroscopy), a highly demanding application for the Reference 3000.

## Measurement System Model and Physical Limitations

To get a feel for the physical limits implied by very sensitive current measurements, consider the equivalent circuit shown in Figure 10-10. We are attempting to measure the cell impedance given by  $Z_{\text{cell}}$ . This model is valid for analysis purposes even though the real Reference 3000 circuit topology differs significantly.

In Figure 10-1:

$E_S$	ideal signal source
$Z_{\text{cell}}$	unknown cell impedance
$I_{\text{cell}}$	“real” cell current
$R_m$	current measurement circuit’s current-measurement resistance
$R_{\text{shunt}}$	unwanted resistance across the cell
$C_{\text{shunt}}$	unwanted capacitance across the cell
$C_{\text{in}}$	current-measurement circuit’s stray input capacitance
$R_{\text{in}}$	current-measurement circuit’s stray input resistance
$I_{\text{in}}$	measurement circuit’s input current

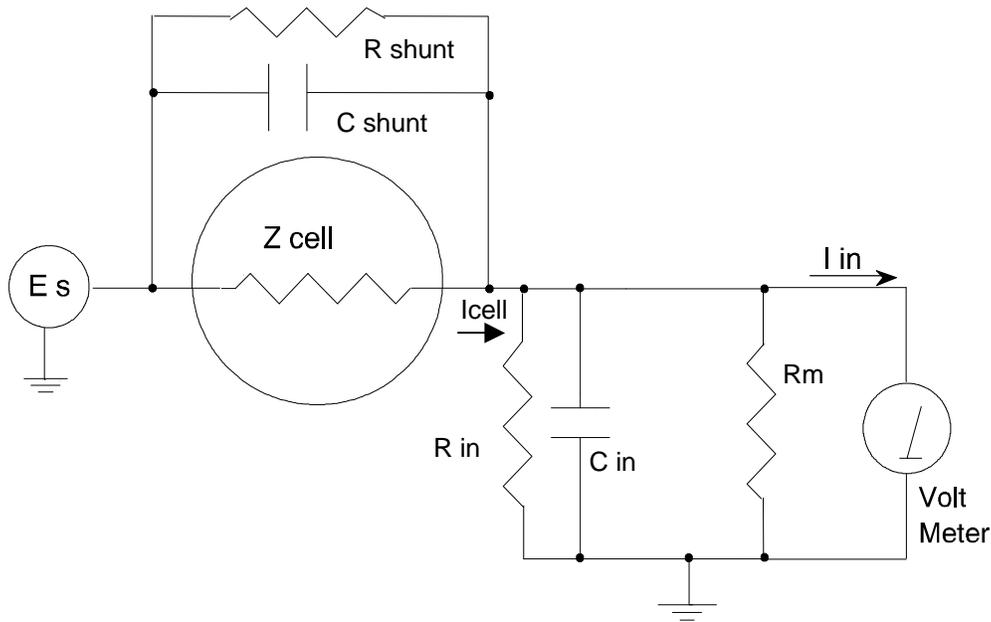
In the ideal current-measurement circuit,  $R_{\text{in}}$  is infinite while  $C_{\text{in}}$  and  $I_{\text{in}}$  are zero. All of the cell current,  $I_{\text{cell}}$ , flows through  $R_m$ .

With an ideal cell and voltage source,  $R_{\text{shunt}}$  is infinite and  $C_{\text{shunt}}$  is zero. All the current flowing into the current measurement circuit comes from  $Z_{\text{cell}}$ .

The voltage developed across  $R_m$  is measured by the meter as  $V_m$ . Given the idealities discussed above, use Kirchoff’s and Ohm’s laws to calculate  $Z_{\text{cell}}$ :

$$Z_{\text{cell}} = E_S \times R_m / V_m$$

**Figure 10-1**  
**Equivalent Measurement Circuit**



Unfortunately technology limits high-impedance measurements because:

- Current measurement circuits always have non-zero input capacitance, i.e.,  $C_{in} > 0$ .
- Infinite  $R_{in}$  cannot be achieved with real circuits and materials.
- Amplifiers used in the meter have input currents, i.e.,  $I_{in} > 0$ .
- The cell and the potentiostat create both a non-zero  $C_{shunt}$  and a finite  $R_{shunt}$ .

Additionally, basic physics limits high-impedance measurements via Johnson noise, which is the inherent noise in a resistance.

### Johnson Noise in $Z_{cell}$

Johnson noise across a resistor represents a fundamental physical limitation. Resistors, regardless of composition, demonstrate a minimum noise for both current and voltage, per the following equations:

$$E = (4kTR \delta F)^{1/2}$$

$$I = (4kT \delta F / R)^{1/2}$$

where:

$k$  = Boltzman's constant  $1.38 \times 10^{-23}$  J/K

$T$  = temperature in K

$\delta F$  = noise bandwidth in Hz

$R$  = resistance in  $\Omega$ .

For purposes of approximation, the noise bandwidth,  $\delta F$ , is equal to the measurement frequency. Assume a  $10^{11} \Omega$  resistor as  $Z_{cell}$ . At 300 K and a measurement frequency of 1 Hz this gives a voltage noise of 41  $\mu V$  rms. The peak-to-peak noise is about five times the rms noise. Under these conditions, you can make a voltage measurement of  $\pm 10$  mV across  $Z_{cell}$  with an error of about  $\pm 0.4\%$ . Fortunately, an AC measurement can reduce the bandwidth by integrating the measured value at the expense of additional measurement time. With a noise bandwidth of 1 mHz, the voltage noise falls to about 1.3  $\mu V$  rms.

Current noise on the same resistor under the same conditions is 0.41 fA. To place this number in perspective, a  $\pm 10$  mV signal across this same resistor will generate a current of  $\pm 100$  fA, or again an error of up to  $\pm 0.4\%$ . Reducing the bandwidth helps. At a noise bandwidth of 1 mHz, the current noise falls to 0.013 fA.

With  $E_s$  at 10 mV, an EIS system that measures  $10^{11} \Omega$  at 1 Hz is about  $2\frac{1}{2}$  decades away from the Johnson-noise limits. At 10 Hz, the system is close enough to the Johnson-noise limits to make accurate measurements impossible. Between these limits, readings get progressively less accurate as the frequency increases.

In practice, EIS measurements usually cannot be made at high-enough frequencies that Johnson noise is the dominant noise source. If Johnson noise is a problem, averaging reduces the noise bandwidth, thereby reducing the noise at a cost of lengthening the experiment.

## Finite Input Capacitance

$C_{in}$  in Figure 10-1 represents unavoidable capacitances that always arise in real circuits.  $C_{in}$  shunts  $R_m$ , draining off higher-frequency signals, limiting the bandwidth that can be achieved for a given value of  $R_m$ . This calculation shows at which frequencies the effect becomes significant. The frequency-limit of a current measurement (defined by the frequency where the phase error hits  $45^\circ$ ) can be calculated from:

$$f_{RC} = 1/(2\pi f R_m C_{in})$$

Decreasing  $R_m$  increases this frequency. However, large  $R_m$  values are desirable to minimize the effects of voltage drift and voltage noise in the I/E converter's amplifiers.

A reasonable value for  $C_{in}$  in a practical, computer-controllable, low-current measurement circuit is 20 pF. For a 6 nA full-scale current range, a practical estimate for  $R_m$  is  $10^7 \Omega$ .

$$f_{RC} = 1/6.28 (1 \times 10^7)(2 \times 10^{-12}) \approx 8000 \text{ Hz}$$

In general, stay two decades below  $f_{RC}$  to keep phase-shift below one degree. The uncorrected upper frequency-limit on a 6 nA range is therefore around 80 Hz.

You can measure higher frequencies using the higher-current ranges (i.e., lower-impedance ranges) but this reduces the total available signal below the resolution limits of the "voltmeter". This exercise forms one basis to the statement that high-frequency and high-impedance measurements are mutually exclusive.

You can use software correction of the measured response to improve the useable bandwidth, but not by more than an order of magnitude in frequency.

## Leakage Currents and Input Impedance

In Figure 10-1, both  $R_{in}$  and  $I_{in}$  affect the accuracy of current measurements. The magnitude error caused by  $R_{in}$  is calculated via:

$$\text{Error} = 1 - R_{in}/(R_m + R_{in})$$

For an  $R_m$  of  $10^7 \Omega$ , an error  $< 1\%$  demands that  $R_{in}$  must be greater than  $10^9 \Omega$ . PC-board leakage, relay leakage, and measurement-device characteristics lower  $R_{in}$  below the desired value of infinity.

A similar problem is the finite input-leakage current  $I_{in}$  into the voltage-measuring circuit. It can be leakage directly into the input of the voltage meter, or leakage from a voltage source (such as a power supply) through an insulation resistance into the input. If an insulator connected to the input has a resistance of  $10^{12} \Omega$  between +15 V and the input, the leakage current is 15 pA. Fortunately, most sources of leakage current are DC and can be tuned out in impedance measurements. As a general rule, the DC leakage should not exceed the measured AC signal by more than a factor of 10.

The Reference 3000 uses an input amplifier with an input current of around 5 pA. Other circuit components may also contribute leakage currents. You therefore cannot make absolute current measurements of very low

pA currents with the Reference 3000. In practice, the input current is approximately constant, so current differences or AC current levels of less than one pA can usually be measured.

## Voltage Noise and DC Measurements

Often the current signal measured by a potentiostat shows noise that is not the fault of the current-measurement circuits. This is especially true when you are making DC measurements. The cause of the current noise is noise in the voltage applied to the cell.

Assume that you have a working electrode with a capacitance of 40  $\mu\text{F}$ . This could be represented by a 1  $\text{cm}^2$  polished bare metal immersed in an electrolyte solution. A rough estimate of the capacitance of the electrical double-layer formed by a metal/electrolyte interface is 20  $\mu\text{F}/\text{cm}^2$ . The area is the microscopic area of the surface, which is larger than the macroscopic geometric area, because even a polished surface is rough. The impedance of this 40  $\mu\text{F}$  electrode, assuming ideal capacitive behavior, is given by:

$$Z = 1/j\omega C$$

At 60 Hz, the impedance magnitude is about 66  $\Omega$ .

Apply an ideal DC potential across this ideal capacitor and you get no DC current.

Unfortunately, all potentiostats have noise in the applied voltage. This noise comes from the instrument itself and from external sources. In many cases, the predominant noise frequency is the AC power-line (mains) frequency.

Assume a realistic noise voltage,  $V_n$ , of 10  $\mu\text{V}$  (this is lower than the noise-level of most commercial potentiostats). Further, assume that this noise voltage is at the North American power-line (mains) frequency of 60 Hz. The noise voltage creates a current across the cell capacitance:

$$I = V_n/Z \approx 10 \times 10^{-6}/66 \approx 150 \text{ nA}$$

This rather large noise current prevents accurate DC current measurement in the low nA or pA ranges.

In an EIS measurement, you apply an AC excitation voltage that is much bigger than the typical noise voltage, so this is not a significant problem.

## Shunt Resistance and Capacitance

Non-ideal shunt resistance and capacitance arise in both the cell and the potentiostat. Both can cause significant measurement errors.

Parallel metal surfaces form a capacitor. The capacitance rises as the metal's area increases and as the separation distance between the metals decreases.

Wire and electrode placement have a large effect on shunt capacitance. If the clip leads connecting to the working and reference electrodes are close together, they can form a significant shunt capacitor. Values of 1 to 10 pF are common. This shunt capacitance cannot be distinguished from "real" capacitance in the cell. If you are measuring a paint film with a 100 pF capacitance, 5 pF of shunt capacitance is a very significant error.

Shunt resistance in the cell arises because of imperfect insulators. No material is a perfect insulator (infinite resistance). Even PTFE, which is one of the best insulators known, has a bulk resistivity of about  $10^{12} \Omega\cdot\text{m}$ . Worse yet, surface contamination often lowers the effective resistivity of good insulators. Water films can be a real problem, especially on glass.

Shunt capacitance and resistance also occur in the potentiostat itself. The Reference 3000 Potentiostat mode specifications in Appendix A contain equivalent values for the potentiostat's  $R_{\text{shunt}}$  and  $C_{\text{shunt}}$ . These values can be measured by an impedance measurement with no cell.

In most cases, the cell's shunt resistance and capacitance errors are larger than those from the potentiostat.

## Hints for System and Cell Design

The following hints may prove helpful.

### Faraday Shield

A Faraday shield surrounding your cell is **mandatory** for very low-level measurements. It reduces both current noise picked up directly on the working electrode and voltage noise picked up by the reference electrode.

A Faraday shield is a conductive enclosure that surrounds the cell. The shield can be constructed from sheet metal, fine-mesh wire screen, or even conductive paint on plastic. It must be continuous and completely surround the cell. Don't forget the areas above and below the cell. All parts of the shield must be electrically connected. You will need an opening in the shield large enough to allow a cell cable to enter the shield.

The shield **must be electrically connected** to the Reference 3000's floating-ground terminal.

An additional connection of both the shield and the Reference 3000 floating ground to an earth ground may also prove helpful.



Only connect the Reference 3000 ground to earth ground if all conductive cell components are well-isolated from earth ground. A glass cell is usually well isolated. An autoclave is generally not well isolated.

### Avoid External Noise Sources

Try to keep your system away from electrical noise sources. Some of the worst are:

- Fluorescent lights
- Motors
- Radio transmitters
- Computers and computer monitors

Try to avoid AC powered or computerized apparatus within your Faraday shield.

### Cell Cable Length and Construction

The Reference 3000 is shipped with a 60 cm shielded cell cable. We also offer extended length cables and unshielded cables as options at extra cost.

Cell cables longer than 1 meter **will** result in degraded instrument performance. Increased noise and decreased stability both can occur. However, with most cells, the instrument will work acceptably with an extended cell cable, so our advice is to try it. As a rule, do not attempt to use current-interrupt *IR*-compensation with cell cables longer than 5 m.

We do not recommend that you use the Reference 3000 with any cables not supplied by Gamry Instruments. The Reference 3000 cable is not a simple cable like a typical computer cable. The Reference 3000 cable includes a number of individually shielded wires contained within an overall shield. We pay careful attention to issues such as shield isolation, isolation resistance, and capacitance.

If you do need a special cable, contact us with your requirements.

## Lead Placement

Many experiments with the Reference 3000 involve cells with small capacitances, the value of which may be important.

In these cases, the capacitance between the Reference 3000's cell leads can result in an error. The Reference 3000 alligator clips can have 10 pF or more of mutual capacitance if they are run alongside each other.

If you wish to avoid excessive capacitance:

- Place the leads as far apart as possible. Pay special attention to physical separation between the working electrode/working sense leads and the counter/counter sense/reference electrode leads.
- Have the leads approach the cell from different directions.
- Remove the alligator clips from the leads. In extreme cases you can replace the banana plugs and pin jack with smaller connectors. If you do so, be careful not to compromise the isolation between the center conductor and the shield.

The cell leads must not be moved during an experiment measuring small currents. Both microphonic and triboelectric effects can create spurious results when the cell cables are moved.

## Cell Construction

If you need to measure small currents or high impedances, make sure that your cell construction does not limit your response.

A cell where the resistance between the electrodes is only  $10^{10} \Omega$  cannot be used to measure  $10^{13} \Omega$  impedances. In general, glass and PTFE are the preferred construction materials for cells. Even glass may be a problem when it is wet.

You also must consider  $C_{\text{shunt}}$ . Make the "inactive" portion of your electrodes as small as possible. Avoid placing electrodes close together or parallel with each other if you are measuring high impedances.

## Reference Electrode

Keep your reference electrode impedance as low as possible. High-impedance reference electrodes can cause potentiostat instability and excessive pickup of voltage-noise.

Try to avoid:

- Narrow-bore or Vycor<sup>®</sup>-tipped Luggin capillaries.
- Poorly conductive solutions, especially in Luggin capillaries.
- Asbestos-thread and double-junction reference electrodes.

Reference electrodes often develop high impedances as they are used. Anything that can clog the isolation frit can raise the electrode impedance. Avoid using saturated KCl-based references in perchlorate-ion solutions.

## Instrument Settings

There are several things to remember in setting up a very sensitive experiment.

- In EIS, use the largest practical excitation. Don't use a 10 mV excitation on a coated specimen that can handle 100 mV without damage.

- Avoid potentials where large DC currents flow. You cannot measure 1 pA of AC current on top of 1 mA of DC current.

### **EIS Speed**

In EIS, do not expect the Reference 3000 to measure  $10^{13} \Omega$  impedances at 1 kHz. Many of the factors listed above limit the performance.

As a rule of thumb, the product of impedance,  $Z$ , times frequency,  $f$ , should be less than  $10^9 \Omega \cdot \text{Hz}$  for good EIS measurements with a Reference 3000.

$$Z \cdot f < 10^9 \Omega \cdot \text{Hz}$$

### **Ancillary Apparatus**

Do not use the Reference 3000 with ancillary apparatus connected directly to any of the cell leads. Ammeters and voltmeters, regardless of their specifications, almost always create problems when connected to the Reference 3000 cell leads.

### **Floating Operation**

The Reference 3000 is capable of operation with cells where one of the electrodes or a cell surface is at earth ground. Examples of earth-grounded cells include: autoclaves, stress apparatus, pipelines, storage tanks and battleships. The Reference 3000's internal ground is allowed to float with respect to earth ground when it works with these cells, hence the name "floating operation".

Instrument performance can be substantially degraded when the Reference 3000 is operated in a floating mode. The instrument specifications only apply on isolated cells with the Reference 3000 earth-ground referenced (not floating).

Special precautions must be taken with the cell connections when the Reference 3000 must float. Make sure that all the cell connections are isolated from earth ground. In this case, you must disconnect the chassis-ground terminal of the Reference 3000 from earth ground.

Finally, ancillary apparatus connected to the Reference 3000 must be isolated. External voltmeters, ammeters, FRAs, etc. must be isolated. This includes devices connected to the monitor connectors located on the Reference 3000 rear panel.



## Chapter 11: EIS Measurement of Small Impedances

### Overview

The Gamry Instruments Reference 3000 is a high-performance measurement instrument used for all types of electrochemical testing. Unlike many other electrochemical instruments, it offers outstanding performance for tests with signals consisting of small currents and high impedances, and for tests involving large currents and very small impedances.

Problems in the measurement of very small currents were discussed in the previous chapter.

This chapter is a discussion of a very different problem, when currents are large, voltages and cell-impedances are small, and inductance limits measurements. These measurements include those encountered in research on:

- Batteries
- Fuel cells
- Super-capacitors

Results from fast transient techniques (CV, chronopotentiometry, etc.) or high-frequency EIS measurements on low-impedance systems are often limited by the cell cable and connections to the cell. An improper cell cable or a proper cell cable badly connected can cause significant errors in the data obtained.

This chapter should answer these questions:

- Why is galvanostatic operation preferred when measuring low impedance?
- What are the sources of error in measuring low-impedance cells?
- Why use four-terminal measurement techniques
- What is mutual inductance?
- How do I connect a cable to my cell to minimize errors?

### Why Galvanostatic Mode?

Even though potentiostatic EIS is the most commonly used EIS technique, it is poorly suited to impedance measurements of low-impedance batteries, fuel cells and super-capacitors. This is why:

Current, voltage, and impedance are related through Ohm's Law. Assume that a high-rate battery has an impedance of 1 m $\Omega$ . A voltage of 1 mV across a 1 m $\Omega$  impedance leads to 1 A of current. No commercial potentiostat is specified to control a cell's potential (typically 0.5 V to 4.5 V) with less than 1 mV of absolute error. When a potential with a 1 mV (or larger) error is applied to a low-impedance battery or fuel cell, a very large DC current flows his current (given enough time) can alter a battery's state-of-charge.

Conversely, a galvanostat can easily control ampere currents to an accuracy of a few milliamperes. The voltage on a battery or fuel cell is usually unaffected when the galvanostat is connected: the DC current is zero or some user-defined value.

A modern EIS system with AC-coupling or offset and gain in the voltage measurement can measure microvolts of AC voltage superimposed on a large DC voltage, as long as that DC voltage is stable.

### DC Errors and Four-terminal Measurements

Four-terminal measurements are a common technique used in precision measurement of small impedances. In a four-terminal measurement, a nominally two-terminal device, such as a resistor or a battery, is connected using four leads. Two of the leads carry the current that must flow through the device to make the measurement. The other leads measure the voltage created by that current.

An illustration of two-terminal measurements versus four-terminal measurements is provided in Figure 11-1. Both schematic diagrams show a resistance measurement made by passing a known current through an unknown resistance,  $R_{\text{test}}$ . The wires in the circuit have a resistance  $R_{\text{wire}}$ . A voltmeter with high input-impedance reads a voltage that is divided by the current value to calculate the value of  $R_{\text{test}}$ .

In the two-terminal case, the voltage measurement is made using the same wires that carry the current. The voltmeter measures:

$$V = I_{\text{test}} \times (2 \times R_{\text{wire}} + R_{\text{test}})$$

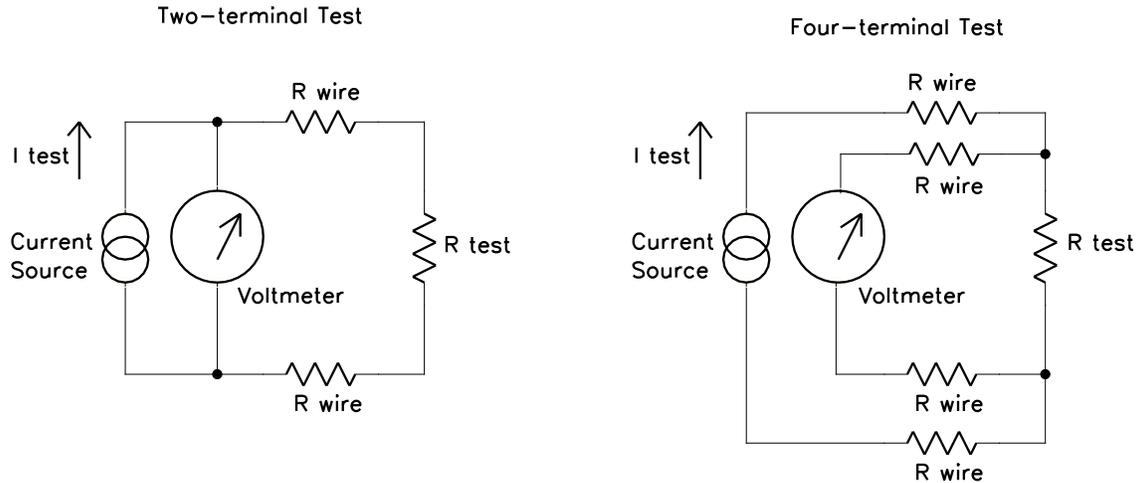
The calculated  $R_{\text{test}}$  is too high, since the resistance of the wires is added to the unknown resistance.

In the four-terminal case, the voltmeter uses two additional wires to measure the voltage close to  $R_{\text{test}}$ . The current is carried through the original pair of wires. There is no current through the voltmeter wires, so the wire resistance does not create voltage drop. The voltmeter measures:

$$V = I_{\text{test}} \times R_{\text{test}}$$

The addition of two wires to the circuit eliminates the error caused by the resistance of the wires.

**Figure 11-1**  
**Two and Four Terminal Measurements**



Four-terminal measurements are also useful in AC measurements, although there is a factor called mutual inductance (discussed below) that makes the AC case more complicated. Ignoring this complication for a moment, a simple extension of the discussion above shows that four-terminal measurements can also eliminate the effects of wire inductance.

In the real world, true four-terminal measurements are rarely possible. There is almost always some metallic conductor shared by both current-carrying leads and the voltage sensing leads. The metal volume shared between the current-carrying function and the sensing function can be minimized, but never eliminated.

### What is Mutual Inductance?

Before we can define mutual inductance, we must define some terms. As discussed above, four leads connect to the cell in an electrochemical system used to test small impedances. We group them into two pairs.

One pair is the counter and working leads (red and green). They carry the cell current so we call them the *current-carrying leads*.

The reference and working sense leads (white and blue) form the second pair. They measure the voltage across two points in the cell. These leads are called the *sense leads*.

A “mutual inductive” effect limits the ability of any system to measure small impedances at higher frequencies. The term *mutual inductance* describes the influence of the magnetic field generated by the current-carrying leads on the sense leads.

In essence, the current-carrying leads are the primary windings of a transformer and the sense leads are the secondary windings. The AC current in the primary creates a magnetic field that then couples to the secondary, where it creates an AC voltage.

You can minimize the unwanted effect in a number of ways:

- Avoid higher frequencies
- Minimize the net field generated by the current-carrying leads.
- Separate the current-carrying leads from the sense leads.
- Minimize pickup of the field in the sense leads.

Each of these ways are discussed below.

### **Avoid high frequencies**

Mutual inductance is an inductive effect. The voltage error is given by:

$$V_s = M di/dt$$

where  $V_s$  is the induced voltage on the secondary,  $M$  is the coupling constant (with units of Henries), and  $di/dt$  is the rate of change in the *primary* current.  $M$  depends on the closeness of the coupling, and can range from zero up to the value of the primary inductance (the inductance in the current-carrying leads).

Assuming a constant-amplitude waveform in the primary,  $di/dt$  is proportional to frequency. There is always a frequency below which the effect of mutual inductance errors is unimportant. Unfortunately, many electrochemical systems need information at frequencies above this limit.

### **Minimize the Net Magnetic Field**

A current passing through a wire creates a magnetic field. The field strength is proportional to the current. Fortunately, passing the same current in opposite directions through adjacent wires tends to cancel the external field. This also minimizes the net inductance in the wires. In all Gamry Instruments Reference 3000 Counter/Working cables, the current-carrying leads are bound together.

From basic physics, the  $\mathbf{E} \otimes \mathbf{B}$  cross-product relationship for current through a wire obeys the Right-Hand-Rule: if your right thumb points in the direction of the current flow in a wire, when you curl your fingers around the wire, the magnetic field curves around the wire in the same direction as your fingers. The current in the primary wires is flowing in opposite directions in the two wires of the cable, so your thumb points in opposite directions for each wire, causing some cancellation of the fields. If the wires were superimposed, in exactly the same place, the cancellation would be perfect.

Because the wires cannot be in identically the same location, the cancellation is imperfect, and some net magnetic field is always present. The more the wires are separated, the larger the net field.

The most common arrangement for inductance and field minimization is the twisted pair. Two insulated wires are simply twisted together. A coaxial-wire arrangement with current flowing in opposite directions in the center conductor and the outer conductor is also effective.

### **Separate the pairs**

If you place a magnetic-field probe near a wire passing current, you measure a field inversely proportional to the square of the distance between the probe and the wire.

In an electrochemical system, the probe is our sense wiring. Separating the sense wires from the current-carrying wires can dramatically reduce the magnetic coupling, reducing errors in the EIS measurement.

The Reference 3000 has two cell cables, so that we can separate the current-carrying wires from the sense wires. The current-carrying pair is in the Counter/Working Cable and the sense pair is in the Sense Cable.

### Twist the Sense Wires

The concept of a magnetic-loop probe is useful in understanding why twisted wire minimizes magnetic pickup. A loop of wire in a changing magnetic field sees a loop voltage proportional to the area of the loop.

Twisting the sense wires together helps in two ways, even though twisting the wire forms loops:

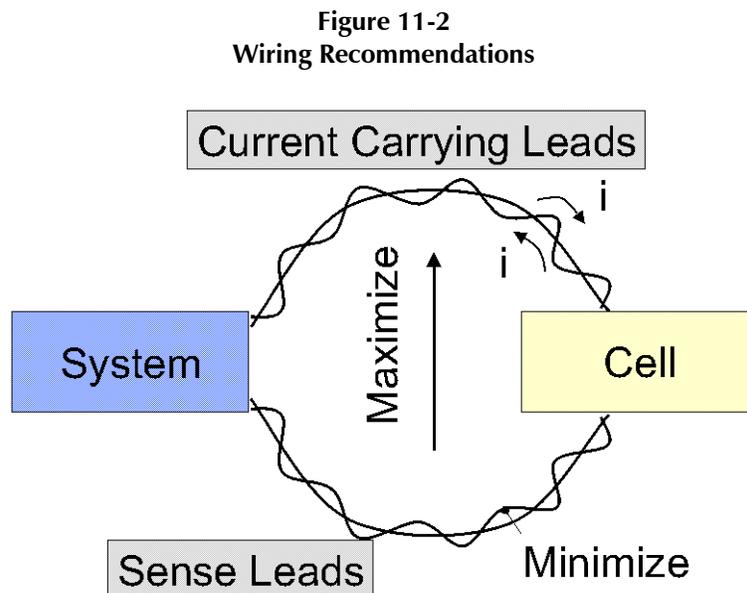
- The twisted wires are forced to lie close to each other, minimizing the loop areas.
- Adjacent loops pickup voltages of opposite polarity, resulting in cancellation.

### How Should You Hook Up Your Cell?

Always use four-terminal connections to the cell. Try to avoid conductors that are shared by both the current-carrying path and the voltage-sensing path.

If your experiments are in the region where mutual inductance may limit performance, keep the voltage-sensing leads in a twisted pair and the current-carrying leads in a different twisted pair. Keep the pair of sensing wires far away from the pair of current-carrying wires. Try to arrange each pair so that they approach the cell from opposite directions.

These recommendations are summarized in the Figure below.



## Appendix A: Reference 3000 Specifications

All specifications are at an ambient temperature of 25°C, with the Reference 3000 powered using the power adapter shipped with the unit, standard shielded 60 cm cell cables, and the cell enclosed in a Faraday shield. All specifications are after software calibration.

A numbered note qualifies many of the specifications. Many of these notes describe the method used to measure a specification. The notes are found at the end of this appendix.

All specifications are subject to change without notice.

### Control Amplifier

High-Voltage, Low-Current Mode

Compliance Voltage	Min. Typ.	$\pm 30$ $\pm 31.5$	V	Note 1a
Output Current	Min.	$\pm 1500$	mA	Note 2

Low-Voltage, High-Current Mode

Compliance Voltage	Min. Typ.	$\pm 15$ $\pm 16.2$	V	Note 1b
Output Current	Min.	$\pm 3000$	mA	Note 2

Both Modes

Unity Gain Bandwidth	Typ.	1100, 330, 50, 5.0, 0.5	kHz	Notes 3, 4
Slew Rate	Typ.	10, 3, 0.5, 0.06, 0.006	V/ $\mu$ s	Notes 3, 4

### Differential Electrometer

Maximum Input Voltage	Min.	$\pm 10$	V	Note 5
Input Current	Max.	6	pA	Note 6
Input Resistance Differential (between inputs) Common Mode (input to ground)	Typ.	100 1	T $\Omega$	Note 7
Input Capacitance Differential (between inputs) Common Mode (input to ground)	Typ.	0.2 12	pF	Note 7
Bandwidth (–3 dB)	Min.	15	MHz	Note 8
CMMR DC to 100 kHz 100 kHz to 1 MHz	Min.	80 60		Note 9

### Voltage Measurement

A/D Full-Scale Ranges	Typ.	$\pm 12.0, \pm 3.0, \pm 0.3, \pm 0.03$	Volts	Note 3
Resolution	Typ.	400, 100, 10, 1	$\mu$ V/bit	
Zero-Offset Error	Max.	1	mV	Note 11
Gain Error	Max.	0.2	%	Note 11

Appendix A: Reference 3000 Specifications--How Should You Hook Up Your Cell?

Offset Range	Typ.	$\pm 10$	V	
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**Current-to-Voltage Converter**

Maximum Full-Scale Range		3000	mA	Note 12
Minimum Full-Scale Range		300 3 (after $\times 100$ gain)	pA pA	Note 12
Voltage across $R_m$	Typ.	150	mV at full scale	Note 13
Output Voltage (at BNC and ADC in)	Typ.	3.0	V at full scale	
Input Offset Current	Max. Typ.	5 2	pA	Note 14
Range Zero Offset	Max.	0.05	% of range	Note 14
Gain Tolerance 3000 mA to 3 nA ranges 300 pA range	Max.	0.2 0.5	% of reading	Note 14
Zero drift	Typ.	0.03	% FS/ $^{\circ}$ C	Note 15
Bandwidth 3000 mA to 300 $\mu$ A ranges 30 $\mu$ A range 3 $\mu$ A range	Typ.	>10 >1.5 >0.15	MHz	Note 16

**Current Measurement**

Resolution	Typ.	0.00333...	% FS/bit	
Offset Range	Typ.	$\pm 100$	% of range	
Post- Offset Gain	Typ.	1 $\times$ , 10 $\times$ , 100 $\times$		
Accuracy	Typ.	Dominated by current-to-voltage error (see above)		

**Potentiostatic Mode**

Applied Voltage Range	Min.	$\pm 11$	V	Note 5
Accuracy DC zero offset Gain	Max.	1 0.2	mV % setting	Note 17
DC Bias	Typ.	$\pm 8$	V	
Scan DAC ranges	Typ.	$\pm 6.4$ , $\pm 1.6$ , $\pm 0.4$	V	
Drift	Max.	<20	$\mu$ V/ $^{\circ}$ C	Note 18
Noise and Ripple 1 Hz to 1 kHz 1 Hz to 200 kHz	Typ.	<5 <20	$\mu$ V rms	Note 19

**Galvanostatic Mode**

Maximum Full-Scale Current		±3000	mA	Note 12
Minimum Full-Scale Current		300	pA	Note 12
Accuracy		Dominated by current-to-voltage converter accuracy shown above		
Sig Gen Voltage for Full-Scale Current		±3.0 V		

**Auxiliary A/D Input (see Appendix D)**

Range (differential)	Typ.	±3	volts	
Input voltage range (either input)	Max.	±3.6	volts	
Gain Error	Max.	0.2	%	
Input Impedance a) as shipped b) re-jumpered for high-Z input	Typ.	100 10	kΩ GΩ	Note 20

**Auxiliary D/A Output**

Range	Typ.	0 to 4.096	V	
Resolution	Typ.	1	mV	

**Environmental**

Operating Temperature Range		0 to 45	°C	
Relative Humidity	Max.	90 (non-condensing)	%	
Storage and Shipping Temperature		-25 to 75	°C	
Maximum Shipping acceleration		30	G	

**General**

Power Input Voltage	Range	22 to 26	V	
Power	Max.	120	W	
Leakage Current (floating, earthed Working Electrode)		± 1 nA		Note 21
Dimensions (approximate) (w×h×d)		20 × 23 × 30	cm	Note 22
Weight (approximate)		5	kg	Note 22
Dimensions of External Power Adapter (approximate)		7.5 × 5 × 22	cm	Note 23
Weight of External Power Adapter (approximate)		900	g	Note 23

NOTES:

1. **a)** Measured in potentiostatic mode with a high-power 20 Ω load connected from counter-electrode lead to the reference lead, and a 2 Ω load between the reference lead and the working-electrode lead. The compliance voltage is measured using an external voltmeter across the 22 Ω load. Under these conditions, the output current is approximately 1.4 A.
- b)** Measured in potentiostatic mode with a high-power 3 Ω load connected from counter-electrode

lead to the reference lead, and a 2  $\Omega$  load between the reference lead and the working-electrode lead. The compliance voltage is measured using an external voltmeter across the 5  $\Omega$  load. Under these conditions, the output current is approximately 5 A.

2. Measured with a four-terminal 0.05  $\Omega$  load, in potentiostatic mode.
3. Unity gain bandwidth and slew rate are correlated. Each has five settings, with the highest slew-rate occurring at the highest bandwidth, down to the lowest slew-rate occurring at lowest bandwidth. Both are measured with 20 k $\Omega$  between counter and reference, and 100  $\Omega$  between the reference and the working and working-sense leads.
4. Measured with an external function generator connected to the **Ext. Sig. In** BNC.
5. The Differential Electrometer Amps are Analog Devices AD8065 Op amps specially selected for low Vos drift versus temperature. These amplifiers have a dual-input stage, with a JFET input over most of their input range and a bipolar transistor input at input voltages greater than +9.5 V. They are only a high-impedance buffer from -12 V to +9.5 V, though they are a unity gain buffer over their whole input-voltage range.
6. The input current of the JFET inputs on the AD8065s is less than 6 pA. When the bipolar input is operative, the input current can be in the microamperes. The specified current is only applicable at voltages of 2 V or less.
7. The differential impedance is measured between the Reference and Work Sense inputs. This is the impedance you measure when you record the EIS spectrum of an infinite impedance cell. There is also a common mode resistance and capacitance associated with the differential electrometer inputs. These values tell you how much the electrometer response is modified by a resistance in series with the source.
8. The bandwidth is for a sine-wave source with a 50  $\Omega$  output impedance driving either input. The bandwidth is well in excess of this specification, which is limited by the measurement equipment used in testing the Reference 3000.
9. CMRR is *common-mode rejection ratio*. It specifies the ability of the differential electrometer to reject signals connected to both inputs. The CMRR is measured driving both inputs with a sine-wave source with a 50  $\Omega$  output impedance, and measuring the error as a function of frequency. Resistance in either input causes a loss of CMRR.
10. Voltage measurement is actually performed with a  $\pm 3$  V signal input to the ADC signal chain. A  $\div 4$  attenuator divides down higher-voltage electrometer outputs so they fit into a  $\pm 3$  V input, thus making a  $\pm 12$  V full-scale range. Gains of 10 and 100 are available to generate 300 mV and 30 mV ranges.
11. The total error in a voltage measurement is:

$$\text{Error} = \text{Zero Offset Error} + \text{Gain Error} \times \text{Voltage}$$

For a 1 V signal, the error can be as high as 3 mV.

12. There are 11 hardware current ranges, separated in sensitivity by decades. The ranges are: 300 pA, 3 nA, ... 300 mA, 3 A full scale. The  $\times 10$  and  $\times 100$  gains add two virtual ranges of 30 pA and 6 pA full scale.
13. The voltage across the current-measurement resistor,  $R_m$ , is as shown. Slightly higher voltages may be seen at the working-electrode terminal on the cell cable, because the cable has both resistive and inductive impedance.
14. The total error in a current measurement is:

$$\text{Error} = \text{Input Current Offset} + \text{Range Zero Offset} \times \text{FS Current} + \text{Gain Tolerance} \times \text{Measured Current}$$

For small currents (pA) the first term is usually dominant.

For large currents ( $\mu\text{A}$ ), the first term can usually be ignored.

The units for the error are amperes.

15. Drift can be approximated by simple drift in the Range Zero Error. In reality all three terms in the equation above can have drift.

- 16.** The Current-to-Voltage converter bandwidth is a function of the current range, the cell cable, and the IEstability setting. The bandwidth can be very low on very sensitive ranges. Longer cell cables add capacitance and slow the current measurement.
- 17.** The total error in a voltage setting is:

$$\text{Error} = \text{DC Zero Offset} + \text{Gain} \times \text{Voltage Setting}$$

For a 1 V signal, the error can be as high as 3 mV.

- 18.** This specification is guaranteed by design. It is not tested.
- 19.** This specification is measured by applying zero voltage across a 1  $\Omega$  resistor and measuring current noise on the 30  $\mu\text{A}$  scale. 1  $\mu\text{V}$  of voltage noise will create a current of 1  $\mu\text{A}$ . The filters in the ADC Chain for the I Signal are used to limit the bandwidths as shown in the spec. Signal averaging via Gamry's DSP mode further reduces the measured noise.
- 20.** See Appendix D.
- 21.** Isolation quality has both DC factors and AC factors (predominately at the 300 kHz power-supply frequency). Only the DC leakage current is shown here. Consult Gamry's technical support for additional information.
- 22.** Excluding external power adapter and any cables supplied with unit.
- 23.** Excluding removable line cord.



## Appendix B: Reference 3000 Cell Connectors

Chapter 6 describes the connections between a cell cable and an electrochemical cell. This appendix describes the other end of the cell cable.

Multiple pins assigned to the same signal are connected together on the Reference 3000's Potentiostat board. If you need to connect this signal outside the Reference 3000, you need a wire connected to **any one** of the D-connector pins.

**Table B-1**  
**Counter/Working Connector**

Pin(s)	Signal Name	Use
1,9	Working	Connected to the working electrode (see Chapter <b>Error! Reference source not found.</b> ).
2, 10	Working Shield	The shield for the working electrode. Connected to Floating Ground on D end of the cable. Left open at the cell end of the cell cable.
3	No connect	
4, 6,11,13	Ground	The potentiostat's floating ground. Can be used to shield the cell if very low currents need to be measured. Also used as a shield for the counter electrode cable.
5,12	Counter	Connected to the Counter Electrode
7	CBL_ID1	One of 4 cable ID bits. Used to identify the type of cell cable attached to the unit. Pull to a logic High through a resistor. Ground to set the bit low.
8	CBL_ID2	One of 4 cable ID bits. Used to identify the type of cell cable attached to the unit. Pull to a logic High through a resistor. Ground to set the bit low.
14	CBL_ID0	One of 4 cable ID bits. Used to identify the type of cell cable attached to the unit. Pull to a logic High through a resistor. Ground to set the bit low.
15	CBL_ID3	One of 4 cable ID bits. Used to identify the type of cell cable attached to the unit. Pull to a logic High through a resistor. Ground to set the bit low.

**Table B-2**  
**Sense Inputs Connector**

Pin(s)	Signal Name	Use
1	CBL_ID2	One of 4 cable ID bits. Used to identify the type of cell cable attached to the unit. Pull to a logic High through a resistor. Ground to set the bit low.
2	CBL_ID1	One of 4 cable ID bits. Used to identify the type of cell cable attached to the unit. Pull to a logic High through a resistor. Ground to set the bit low.
3,4	Ground	The potentiostat's floating ground. Can be used to shield the cell if very low currents need to be measured. Also used as a shield for the counter-electrode cable.
5,6,12,14	Reference Shield	The shield for the reference electrode input. Driven to the same potential as Pin 16. Left open at the cell end of the cell cable.
7,15	Work Sense Shield	The shield for the work-sense electrode input. Driven to the same potential as Pin 8. Left open at the end of the cell cable.
8	Work Sense	Connected to the working electrode in most cases (see Chapter 5). This lead has a 261 $\Omega$ resistor in the cell end of the cable. Custom cell cables are likely to require a similar resistor.
9	CBL_ID3	One of 4 cable ID bits. Used to identify the type of cell cable attached to the unit. Pull to a logic High through a resistor. Ground to set the bit low.
10	CBL_ID0	One of 4 cable ID bits. Used to identify the type of cell cable attached to the unit. Pull to a logic High through a resistor. Ground to set the bit low.
11	Counter Sense	Connected to the counter electrode in ZRA-mode and stack-mode experiments (see Chapter 5). This lead has a 261 $\Omega$ resistor in the cell end of the cable. Custom cell cables are likely to require a similar resistor.
13	Reference Electrode	Connected to the reference electrode in most cases (see Chapter <b>Error!</b> <b>Reference source not found.</b> ). This lead has a 261 $\Omega$ resistor in the cell end of the cable. Custom cell cables are likely to require a similar resistor.

## Appendix C: Misc. I/O Connector

This connector contains a number of signals; it connects the Reference 3000 to external apparatus. It is the miniature 15-pin female D-shaped connector on the rear panel of the Reference 3000.



**Caution:** The ground pins on this connector are not the Reference 3000 floating ground. The metal shell of the D-connector is connected to the Reference 3000's Floating Ground. Improper Misc. I/O connections can damage a Reference 3000 that is connected to a high-energy cell containing earth-grounded terminals.

The auxiliary analog output, derived from a D/A converter, is on this connector. The scaling is normally 1 mV per bit, for a range of 0 to 4.096 V full-scale.

The pin-out table for this connector is shown in Table C-1.

**Table C-1**  
**Misc. I/O Connector**

Pin	Name	Use
1	Analog Output High	The auxiliary output signal (DAC output).
2	Analog Output Low	The auxiliary output ground connection (ground).
3	Sync In	Used in slave mode: starts data acquisition 2.2 k $\Omega$ input impedance
4	Sync Out	A TTL pulse output before the start of a data point 330 $\Omega$ output impedance
5	no connection	
6	Ground	Digital ground
7	Digital Out 0	A TTL-compatible digital output 330 $\Omega$ output impedance
8	Digital Out 1	A TTL-compatible digital output 330 $\Omega$ output impedance
9	Digital Out 2	A TTL-compatible digital output 330 $\Omega$ output impedance
10	Digital Out 3	A TTL-compatible digital output 330 $\Omega$ output impedance
11	Digital In 0	A TTL-compatible digital input 2.2 k $\Omega$ input impedance
12	Digital In 1	A TTL-compatible digital input 2.2 k $\Omega$ input impedance
13	Digital In 2	A TTL-compatible digital input 2.2 k $\Omega$ input impedance
14	Digital In 3	A TTL-compatible digital input 2.2 k $\Omega$ input impedance
15	+5 V	Power: 50 mA maximum current



**Caution:** Floating operation of the Reference 3000 can be compromised by improper connection of cables to the **Misc. I/O** Connector. Do not use standard 15-pin shielded cables with this connector. Custom cables with the shield connected to pin 6 of the D-connector are required.



## Appendix D: Auxiliary A/D Input Characteristics

### Overview

The Controller board used in the Gamry Instruments Reference 3000 potentiostat has jumpers or switches that configure the input circuitry used for the Aux A/D function. Early units have jumpers, while later units have CMOS switches set using software. The changeover occurred in 2009.

We usually ask that you return your Reference 3000 to Gamry Instruments if you need to change the hardware jumpers. This information is provided so a **qualified** service technician can change the Aux A/D input jumpers in the field.

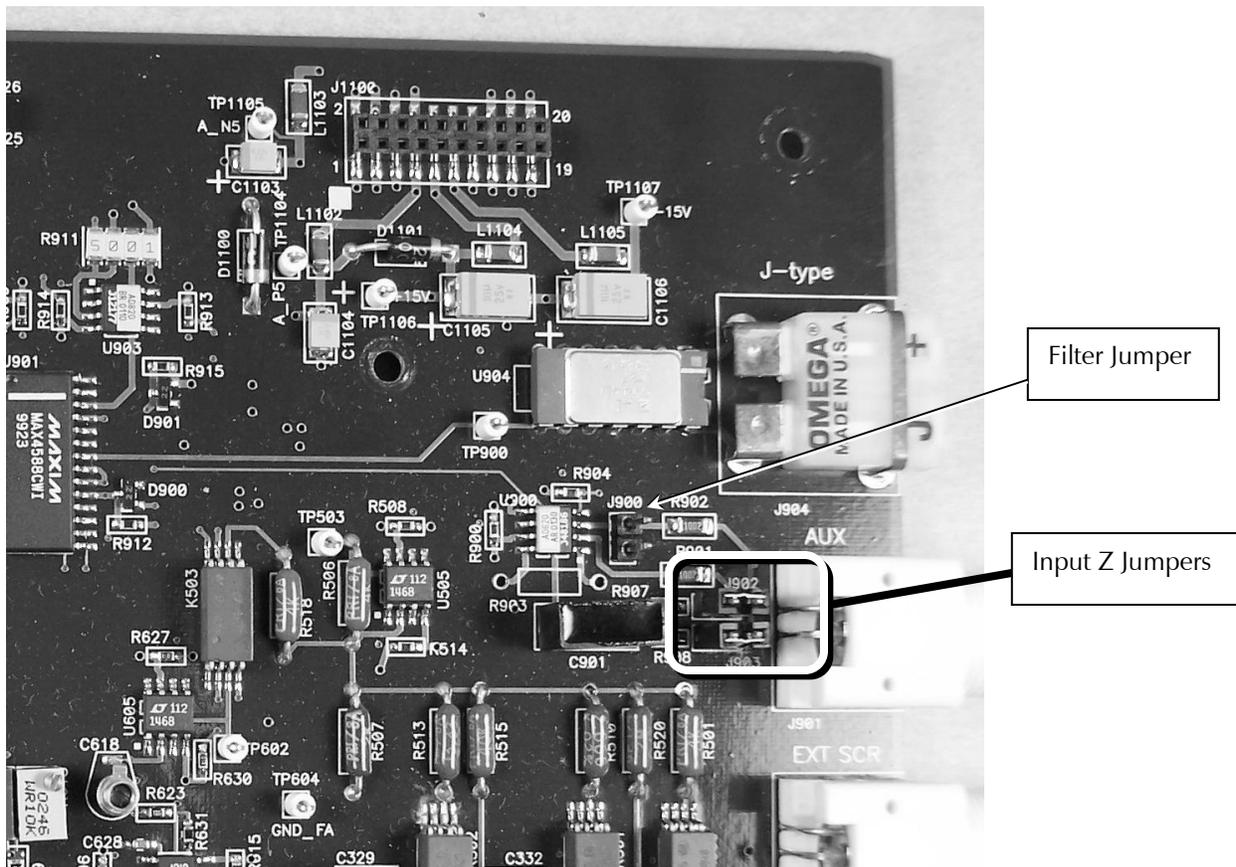
The CMOS switches are set using a function call in an Explain script. After the CMOS switches are set, they remain in the selected position until another script resets them. The settings return to their default values on reset or Power Up of the Reference 3000.

Call or e-mail your local Gamry representative if you need to change your Auxiliary A/D input characteristics, and you are uncertain which type of control board you have.

### Jumper Identification

The three jumpers that configure the Aux A/D input are in a cluster located at the upper right side of the Controller Card. See the figure below for jumper locations.

**Figure D-1**  
**Auxiliary A/D Input Configuration Jumpers**  
**View of Reference Family Controller Board**



## Input Impedance Selection

Two jumpers are associated with the input impedance, J902 and J903. With J902 and J903 installed, the Aux A/D input has a 100 k $\Omega$  input impedance. This is the default setting. With the jumpers installed, the potentiostat can be calibrated without a cable on the input BNC connector.

With both J902 and J903 removed, the Aux A/D input impedance is 10 G $\Omega$  (typically). This setting is suitable for use with a high-impedance source such as a reference electrode.



If you have removed jumpers J902 and J903, **do not** calibrate the potentiostat unless you have a cable connecting both Aux A/D inputs to floating ground.

The CMOS switch shorts the circuit nodes connected by these jumpers. As with the jumpers, in the low-impedance setting, the input impedance is set to 100 k $\Omega$ . In the high-impedance setting, the CMOS switches are opened, increasing the input impedance to about 10 G $\Omega$ .

## Bandwidth Selection

J900 (or on some boards J901) controls the bandwidth of the Aux A/D input. With J900 (or J901) removed, there is no filtering on this input, and the -3 dB bandwidth is greater than 300 kHz.

With J900 (or J901) in place (the default setting), a single-pole RC filter is used to limit noise into the A/D. The nominal cutoff frequency of this filter is 20 Hz.

Note that source impedances greater than 1 k $\Omega$  appreciably lower this cutoff frequency. When the Aux A/D input is driven by a source with an output impedance of 1 M $\Omega$ , the frequency cutoff is less than 0.25 Hz.

The CMOS switch performs the same function as the jumper.

## Aux A/D Specifications

Range	$\pm 3.276$ V	
Input Impedance	100 k $\Omega$ (approx)	Low Impedance Selected
	or	
	10 G $\Omega$ (typical)	High Impedance Selected
Input Bias Current	< 10 nA	High Impedance selected
Filter Cutoff	20 Hz $\pm$ 20%	With Low Bandwidth selected

## Function Call to Set the Aux A/D BNC Characteristics

The CMOS switch is set using an Explain function call. The syntax of this call is:

```
Pstat.SetAuxBNCImpedance (Impedance)
```

The variable passed to the function is a Boolean (with a value of TRUE or FALSE).

The following definitions can be used to make a script that calls this function more readable.

```
#define SETAUXBNCSETTINGS_IMPEDANCE_HIGH TRUE
#define SETAUXBNCSETTINGS_IMPEDANCE_LOW FALSE
```

A TRUE in the Impedance variable opens the switches across J902 and J903, selecting high input impedance. A FALSE selects 100 k $\Omega$  input impedance.

## Appendix E: Auxiliary Electrometer Specifications

Unless otherwise mentioned, all specifications apply at 22°C, zero common-mode voltage versus Ground FA, input voltages with  $Z_{out} < 10 \Omega$ , and all channel inputs (other than those of the channel under test) at zero volts versus Ground FA. All measurements are made with the Aux Channel A/D with normal Framework calibration.

### DC Voltage Measurement

Offset Voltage	$< 500 \mu V$	20°C to 25°C	At all gain ranges
Temp Drift	$< 10 \mu V/^{\circ}C$	from 0 to 45°C	
Ranges	$\pm 50 \text{ mV}, \pm 500 \text{ mV}, \pm 5V$	Full Scale	Differential Input Voltage
Resolution	$1.666 \mu V, 16.66 \mu V, 166.6 \mu V$	per bit	Differential Input Voltage
Gain Error	$< 0.3\%$		on all ranges
Offset Range	$\pm 5.12 \text{ V}$		offset of differential signal
Offset Resolution	$166.6 \mu V/\text{bit}$		
Input Current	$< 10 \text{ pA}$	zero volts input	measure by voltage

### Input Impedance

Common Mode	$> 30 \text{ G}\Omega$ in parallel with $< 100 \text{ pF}$	$-36V < V_{cm} < 36 \text{ V}$	versus floating ground
Differential	$> 100 \text{ G}\Omega$ in parallel with $< 40 \text{ pF}$	$-5V < V_{diff} < 5 \text{ V}$	measured between the two inputs

### Common Mode Rejection

CMR Range	$-36 \text{ V} < V_{in} < 36V$		versus Floating Ground
CMRR	$> 94 \text{ dB}$ $> 86 \text{ dB}$	at 3 Hz at 100 kHz	

### Crosstalk

Low Frequency	$< -90 \text{ dB}$	DC to 10 kHz
High Frequency	$< -80 \text{ dB}$	10 kHz to 100 kHz

### Other AC Specifications

Bandwidth	$> 2 \text{ MHz}$	- 3 dB
Phase Shift	$< 1^{\circ}$ $< 3^{\circ}$	DC to 20 kHz 20 kHz to 100 kHz
Noise	$< 4 \mu V_{pp}$ $< 10 \text{ nV}/\text{Hz}^{1/2}$	0.1 Hz to 10 Hz at 1 kHz



## Appendix F: Power LED Blink Codes

In the rare event that some electronics module fails, we include the start-up blink codes that the blue **Power** LED flashes. Upon start-up, view the **Power** LED. If it blinks more than five times, or flashes one of the blink-codes below, please call Gamry Instruments for service.

### Normal Start-up

The **Power** LED flashes three times.

### Failure in an electronics module

The **Power** LED flashes codes depending upon the failed module:

Power LED blink code	Problem
Twice, then once	Post SDRAM unidentified problem
Twice, then twice	Post SDRAM size error
Twice, then three times	Post ROM checksum error
Three times, then four times	Post Serial init. RX buffer
Three times, then five times	Post Serial init. TX buffer
Three times, then six times	Post Serial init. UART
Three times, then eight times	Post Communications module missing
Four times, then once	Post Control missing
Four times, then twice	Post potentiostat missing

If there are multiple problems, the communications board takes code priority over the memory module.

### Severe problem

The red LED, which normally switches off after a rapid boot-up, remains illuminated.



If you receive one of the above codes, please call Gamry Instruments Service Department for assistance.

--Other AC Specifications

## Appendix G: CE Certificate



### Declaration of Conformity

According to ISO/IEC Guide 22 and CEN/CENELEC EN 45014

Manufacturer's Name and Location:

**Gamry Instruments**  
**734 Louis Drive**  
**Warminster, PA 18974**  
**USA**

This declaration is for the Gamry Instruments product models: Reference 3000 Potentiostat/Galvanostat/ZRA.

The declaration is based upon compliance with the following directives:

EMC Directive 89/336/EEC as amended by 92/31/EEC and 93/68/EEC  
 Low Voltage Safety Directive 73/23/EEC as amended by 93/68/EEC

The declaration is based upon product compliance with the following standards as defined in report number R0295-000 from Ergonomics, Inc. for safety analysis and report number RSI-2772L from Radiation Sciences, Inc. for EMC test and analysis.

EMC Standards	Title	Class/ Criteria
EN 61000-4-2	EMC – Electrostatic discharge, Immunity	B
EN 61326:2002-2	EMC – Radiated Emissions	A

Low Voltage Directive Safety Standards	Title
EN 61010-1:2001	Safety requirements for electrical equipment for measurement, control and laboratory use, Part 1: General requirements.
EN 61010-2-081: 6/2003	Safety requirements for electrical equipment for measurement, control and laboratory use, Part 2 Particular requirements for automatic and semiautomatic laboratory equipment for analysis and other purposes

  
 \_\_\_\_\_  
 Signature

January 19, 2009  
 Date

Dr. Gregory A. Martinchek, PhD  
 Title: President

Formal signed declaration is on file at Gamry, Inc.

**Certificate of Conformance**



Redefining Electrochemical Measurement

**Declaration of Conformity**

This declaration is provided according to ISO/IEC 17050-1: 2004 and ISO/IEC 17050-2: 2004

Manufacturer's Name and Location:

Gamry Instruments, Inc  
734 Louis Drive  
Warminster, PA 18974 USA

This declaration is for the Gamry Instruments product models PC5 3000 Series product family. This includes:

1. Gamry's p/n 990-00224 PC5 3000 Reference Potentiostat/Galvanostat/ZRA and any variations thereof; and
2. Gamry's p/n 990-00227 PC5 3000 Reference Potentiostat/Galvanostat/ZRA with its Auxiliary Electrometer option installed and any variations thereof.

The declaration is based upon compliance with the following directives:

- EMC Directive 89/336/EEC as amended by 92/31/EEC, 93/68/EEC and 2004/108/EC.
- Low Voltage Safety Directive 2006/95/EC as dated 12 December 2006.

The declaration is based upon product compliance with the following standards as defined in report number R0422-000 from Ergonomics, Inc. for safety analysis and report number R-1160P from Retlif Testing Laboratories, Inc. for EMC test and analysis.

EMC Standards	Title	
<b>EMISSION</b>		
EN61326:2003	Electrical Equipment for Measurement, Control and Laboratory Use - EMC Emission Requirements-Second Edition	Pass
IEC61000-3-2	Harmonics	Pass
IEC61000-3-3	Flicker	Pass
CISPR 16-2:1996	Class A Power Line Conducted Emissions	Pass
CISPR 16-2:1996	Class A Electric Field Radiated Emissions	Pass
<b>IMMUNITY</b>		
EN 61000-4-2:1995	Electrostatic Discharge	Pass Criteria B
IEC 61000-4-3:1995	Radiated Field Immunity	Pass Criteria A
IEC 61000-4-4:1995	Electrical Fast Transient Immunity (AC Power)	Pass Criteria B
IEC 61000-4-5:1995	Surge Immunity	Pass Criteria B
IEC 61000-4-6:1996	Power Line Conducted Immunity	Pass Criteria A
IEC 61000-4-11:1994	Voltage Dips and Interrupts	Pass Criteria C
<b>Low Voltage Directive</b>		
<b>Safety Standards</b>	Title	
EN 61010-1:2001	Safety requirements for electrical equipment for measurement, control and laboratory use, Part 1: General requirements.	
EN 61010-2-081: 2003	Safety requirements for electrical equipment for measurement, control and laboratory use, Part 2 Particular requirements for automatic and semiautomatic laboratory equipment for analysis and other purposes	

  
Signature

Dr. Gregory A. Martinchek, PhD  
Title: President  
Gamry Instruments  
734 Louis Drive  
Warminster, PA 18974, USA

1/19/2009  
Date

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