# **FAS1 Femtostat**

**Operator's Manual** 

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Contact us at your earliest convenience. We can be contacted via:

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Contact <u>Techniical Support</u>

Gamry Instruments, Inc.

Mail 734 Louis Drive

Warminster, PA 18974

USA

If you write to us about a problem, provide as much information as possible.

If you are having problems with installation or use of your FAS1 Femtostat, it would be helpful if you called from a phone near to your computer, where you can type and read the screen while talking to us.

We are happy to provide a reasonable level of free support for registered users of our products. Reasonable support includes telephone assistance covering the normal installation and use of the FAS1 in standard computer hardware.

We provide a one year warranty covering both parts and labor. A service contract that extends the warranty is available at an additional charge.

Enhancements to the FAS1 that require significant engineering time on our part may be available on a contract basis. Contact us with your requirements.

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## **Chapter 1 -- Introduction**

#### **About This Manual**

This manual covers the installation and use of the Gamry Instruments' FAS1 Femtostat.

This manual describes use of an FAS1 with Revision 3.1 of the Gamry Framework software. It is equally useful when setting up a newly purchased potentiostat or modifying the setup of a two year old potentiostat for use with new software.

The bulk of Chapter 1 is an overview of the FAS1's design and modes of operation. Chapter 2 contains FAS1 installation instructions. Chapter 3 describes cell cable connections. Chapter 4 covers the difficult issues of potentiostat stability and approaches to prevent oscillation. Chapter 5 discusses the realities of low current, high impedance measurements.

You will find dry technical material such as specifications, DIP switch settings, and connector pin outs in the Appendices.

This manual does not discuss software installation or operation.

Software support for the FAS1 is described in the Installation Manual for each of the applications programs. All the Gamry Instruments' applications that run under the Gamry Framework control the FAS1 via a PSTAT object. See the Framework On-line Help for programming information.

### **CE Compliance Required for Sale in Europe**

The European Community has instituted standards limiting radio frequency interference from electronic devices and mandating several safety requirements. Gamry Instruments has modified its instruments to comply with these standards. We are shipping CE compliant instruments to all destinations.

The relevant CE regulations include EN55022 Class B and EN60950.

#### About the FAS1

The FAS1 Femtostat is a research grade electrochemical instrument optimized for the measurement of very small current signals. It can operate as a potentiostat, a galvanostat, or a ZRA (zero resistance ammeter).

The FAS1 is the low current member of Gamry Instruments' PC4 Potentiostat family. It shares a number of characteristics with the other members of this family, especially in the areas of signal generation and signal preconditioning prior to A/D conversion.

FAS1 features include 10 decade current autoranging, electrical isolation from earth ground, and extensive filtering. A sine wave generator on the FAS1 allows its use for impedance measurements up to 100 kHz.

The FAS1 consists of a printed circuit card that installs directly into a computer and an external measurement module. The printed circuit card will be referred to as the Controller Card. It requires one full length, full

height expansion slot in an AT compatible computer. The external module will be referred to as the Potentiostat Module. The cable that connects the Controller Card and the Potentiostat Module uses up a second card slot in the computer.

The Controller Card contains an ISA bus interface, an isolated power supply, a signal generator, and a high performance measurement system.

- The ISA bus (also known as the AT bus) interface communicates with the rest of the FAS1 over
  optocoupled serial lines. There is no ground connection between the ISA bus circuitry and the analog
  circuits in the FAS1.
- The isolated DC/DC converter on the Controller Card generates the power used by the Potentiostat Module as well as that used on the Controller Card.
- The standard "DC" signal generator on the Controller Card uses two 16 bit D/A converters. A DDS sine wave generator is packaged on a small "piggyback card" that plugs into the Controller card.
- The Controller Card measurement circuitry includes signal filtering, offset, and switchable gain on two
  independent measurement channels. The output of these channels is measured using a 16 bit A/D
  converter.

The Potentiostat Module is a digitally controlled analog potentiostat, with additional galvanostat and ZRA operating modes. Both the current and potential measurement circuits include both a low input current, slow amplifier and a much faster, higher input current amplifier. This allows for both high speed and low current operation using a single instrument.

### **Femtostat Schematic Diagram**

If you are not familiar with electronic schematics or potentiostats, you probably want to skip this section. This information is for expert use only and is not required for routine use of the FAS1 Femtostat.

Figure 1-1 is a highly simplified schematic diagram. It shows the analog portion of the Femtostat in its potentiostatic control mode.

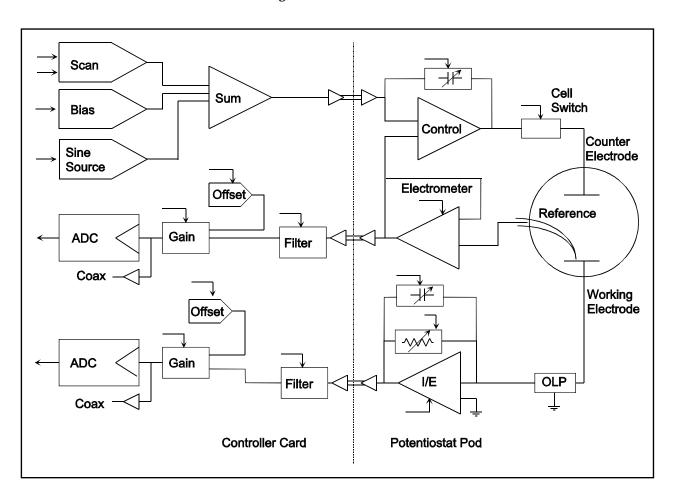


Figure 1-1 FAS1 Analog Circuits in Potentiostat Mode

#### A few points concerning this schematic:

- The circuits on the right side of the schematic are in the Potentiostat Module and those on the left are
  on the Controller Card. The dotted line shows the separation between the two portions of the
  instrument. The analog signals sent between the portions are received with differential amplifiers to
  eliminate grounding problems.
- Arrows pointing into a circuit indicate a computer control input.
- The labels CE, RE and WE stand for counter electrode, reference electrode and working electrode respectively.
- There are two 16 bit D/A converters generating the computer controlled portion of the applied cell voltage.
- The labels F/S on the Electrometer and the I/E converter indicate that the computer can switch these amplifiers between a fast and a slow configuration.
- The I/E converter feedback resistor consists of 9 decade resistors that can be switched in under computer control.
- The cell switch is actually two switches in series a relay for low leakage and an FET switch for fast response.
- The label OLP refers to overload protection.
- Gains and resistor values are not shown.
- Two capacitors can be switched across the I/E converter resistor. These capacitors are used for filtering and stability compensation.
- The control amplifier is shown at the upper right side of the schematic. Compensation capacitors can be switched across the control amplifier to adjust its bandwidth and improve potentiostat stability.
- The A/D converter is a 16 bit successive approximation type converter.
- Some analog circuits, including overload detection circuitry, positive feedback IR compensation, the auxiliary D/A converter, power circuits, and data acquisition controls are not shown.
- All digital circuits, including the AT bus interface, timers, state machines, optocouplers, and digital I/O are not shown.
- Timing for both data acquisition and D/A update in the signal generator is controlled by a state machine working with a crystal oscillator generated clock. A busy microprocessor in the computer cannot create timing jitter.

#### **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.
- Hexadecimal numbers. Hexadecimal numbers are used for hardware related items such as I/O addresses. The Gamry Framework and this manual use the C programming language convention: all hexadecimal numbers have a prefix of 0x. For example, the default I/O addresses used by a FAS1 Femtostat are 0x120 through 0x13F.
- File names and directories. Inside paragraphs, references to computer files and directories will be in quotes, for example: "WIN.INI" and "\FRAMEWORK\FRAMEWORK.EXE".

## Chapter 1 -- Introduction -- Notational Conventions

### **Chapter 2 -- Installation**

An FAS1 Femtostat is only useful after it has been installed in an AT compatible computer.

If you purchase an FAS1 in a system that includes both a computer and an applications software package, Gamry Instruments, Inc. will install the FAS1 (and the system software) to produce a "turn-key" system. You may ignore this chapter if you have purchased a turn-key system.

If you buy your own computer, add an FAS1 to an existing system, or move an old FAS1 to a new computer, you need to know how to install an FAS1 into a computer. Read on.

Software installation is discussed in the Installation Manual for each software package. It will not be discussed here.

### **Computer Requirements**

Before you install an FAS1 into your own computer you must make sure that your computer meets these simple requirements.

- A computer based on one of the following Intel microprocessors 486, Pentium™, Pentium II™ or a 100% compatible processor from another vendor.
- One full length, full AT height expansion slot. This slot must have a 16 bit ISA or EISA bus interface. The ISA bus interface is commonly referred to as the "AT Compatible" bus.
- A second slot which is used up by the minipanel and cable that connects the Controller Card and the Potentiostat Module. This slot does not need to be full length or height.
- Up to 18 watts of power supply capacity for each FAS1 Potentiostat. This is in addition to the power normally drawn by your computer and its expansion cards.

Most of the power is drawn from the computer's +12 volt supply. However, all four power supplies found in a PC ( $\pm 5$  and  $\pm 12$  volts) are required for FAS1 operation.

Gamry's Windows applications software packages may impose additional, more stringent requirements.

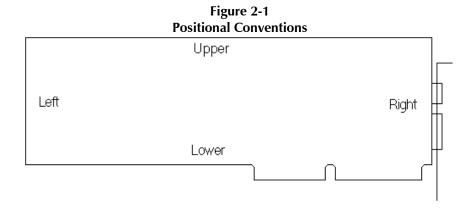
#### **Positional Conventions**

Throughout this manual, reference will be made to positions on the FAS1 Controller Board. In order to avoid confusion, we will define some conventions that describe positions on this card.

#### Assume:

- The Controller Card is lying on a table in front of you.
- The component side of the card is up.
- The card edge (where the card plugs into the computer) is facing you.

Under these assumptions, Figure 2-1 illustrates our positional convention.



### **Handling the FAS1 Controller Card**

The FAS1 Controller Card, like most electronic components, is susceptible to damage from static discharges and connection to live circuits. Some elementary precautions should be taken when handling and installing these cards.

- The Controller Card is shipped in an anti-static bag. Leave the card in its bag until you need to install or reconfigure it.
- Always turn off your computer before plugging in the card.
- If you need to leave the card out of its anti-static bag, such as when you change the DIP switch that configures the card, lay the anti-static bag on a flat surface, then lay the card on top of the bag.
- Prior to handling the card, you should momentarily ground yourself to eliminate any static charges on your body. A good way to accomplish this is to turn off your computer, then lightly touch your finger to an unpainted portion of the computer's metal chassis.
- Save the anti-static bag. You must use it if the card is shipped while not installed in a computer. This includes occasions when the card must be returned to Gamry Instruments, Inc. for repair.

### **Dip Switch for System Configuration**

The FAS1 Controller Card has one 4 pole DIP switch on it. You can use this switch to configure the card for use in a specific computer system.

In most cases you leave the switch in its factory set position and forget it is there. The factory settings should work for all single potentiostat systems where the computer contains only "common" expansion cards. Common is defined for purposes of this discussion as devices such as standard video cards, GPIB adapters, serial & parallel ports, disk controllers, etc.

You only have to set these DIP switch if you have an I/O address or interrupt level conflict between your FAS1 Controller Card and another expansion card. You normally discover these conflicts when the newly installed FAS1 doesn't work or causes failures in other cards that used to work.

The default factory settings for an FAS1 Controller Card are:

- Board I/O address range = 0x120-0x13F (hexadecimal). This range is not used by common AT compatible expansion cards.
- Board number = 1. This is the correct setting for systems containing a single FAS1 Femtostat. This setting must be changed on the second, third and fourth FAS1 added to a computer.

If you have questions about what the term I/O Address Range means, or think you may need to change the settings, consult **Appendix B.** 

The interrupt level used by the FAS1 can also conflict with the level used by other cards in your computer. This level is set via software settings, not via DIP switch changes, so it will not be discussed further here. It is discussed in **Appendix B**.

### **Installing the Controller Card in Your Computer**

NOTE: Please review the discussion on **Handling the FAS1 Controller Card** earlier in this chapter prior to proceeding.

The following procedure is used to install an FAS1 Controller Card in your computer. It assumes that you are using the default configuration for the installed card or that you have already configured the card for a non-standard configuration.

- 1. Turn off your computer.
- 2. Following your computer manufacturer's instructions, open up the computer to expose its expansion card slots.
- 3. Locate an empty full length expansion slot that has an AT (16 bit) interface. If necessary, remove the retaining screw and slot cover (the 'L' shaped metal bracket). Save the screw for use later.
- 4. Locate a second empty slot that is within 20 cm of the first. You may have to move some of your existing cards to get two suitable slots. Again, remove the retaining screw and slot cover, saving the screw for use later.
- 5. Remove the Controller Card from its anti-static bag.
- 6. Plug the Controller Card into the slot with an AT interface. Make sure the card seats securely in the edge card connector on the motherboard. Secure the card in the slot using the screw from Step 3.
  - NOTE: All the gold fingers on the lower edge on this card must be in a motherboard edge connector. If they are not, you have plugged the card into an XT (8 bit) interface slot.
- 7. Locate the Potentiostat Connector Mounting Bracket. It is a metal L shaped bracket with a D connector and cable mounted on it. Place this bracket in the card slot identified in Step 4. Secure the bracket using the screw from Step 4.
  - This bracket is used to interconnect the Controller Card and the Potentiostat module.
- 8. <u>Do not</u> close up the computer yet.

### Connecting the FAS1 Interconnection Cable to the Controller Card

The cable on the L bracket that you just added to the computer is connected to the Controller Card as follows:

- 1. Locate the 26 pin header (group of 26 pins) on the Controller Card. It is in the upper middle of the card.
- 2. Notice that one pin is missing from this header. This missing pin is used to "key" the connector and prevent incorrect cable installation.
- 3. Look at the 26 pin connector on the cable attached to the Potentiostat Connector Mounting Bracket. One of the 26 holes in this connector has been blocked.
- 4. Plug the connector into the 26 pin header. The blocked hole must line up with the missing pin on the header. If they do not line up, you will be unable to adequately seat the connector on the header.
- 5. Carefully double check your work.
  - CAUTION: The connector keying does not prevent one type of connector misalignment. You can easily miss an entire row of pins. Make sure that <u>both</u> rows of pins are in the connector.
- 6. Once this step is completed you may close up the computer.

### **Connecting the Potentiostat Module to the Controller Card**

Turn off your computer before making this connection.

The cable used to make this connection is provided with the FAS1. There is a 25 pin D connector on both ends of the cable.

The female end of this cable connects to the male 25 pin D connector on the Potentiostat Connector Mounting Bracket.

CAUTION: Many Computers include an XT compatible COM port that also uses a male 25 pin D connector. If your computer has two or more male 25 pin D connectors, <u>make sure</u> that you have connected to the correct one. Plugging the Potentiostat Module into a COM port could damage the Potentiostat Module or the computer.

The male end of the cable connects to the Potentiostat Module.

CAUTION: You must tighten the mounting screws that hold both connectors in place. Circuitry in the FAS1 could be damaged if this cable is accidentally unplugged while the computer is powered.

### **Application Software Installation and System Checkout**

Software installation is slightly different for each Gamry Instruments, Inc. application package. Refer to the software installation instructions in the Installation Manual for each application package in your system.

You should also perform the system checkout procedures for each application. Follow the instructions in each application's Installation Manual. The system checkout procedures check for correct hardware and software installation. They are not a comprehensive test of each facet of system operation.

#### **Calibration**

After you have run the system checkout procedure(s), you should calibrate each FAS1 Femtostat installed in your system. A calibration script is provided with the Gamry Framework. The Installation Manual for every major application package contains instructions for calibration using this script.

CAUTION: FAS1 calibration calls for an external resistive dummy cell. A suitable resistor was shipped with the FAS1. This resistor is a 100  $\Omega$  1% accurate resistor. Please place this resistor in a safe place where you can find it if your unit requires recalibration.

If you do need to recalibrate and you cannot find the resistor shipped to you, you can substitute another  $100\Omega$  resistor. Its wattage and tolerance are unimportant.

Potentiostat calibration is only required infrequently. You should recalibrate under the following circumstances:

- You are installing an FAS1 Femtostat into a new computer or moving an FAS1 into a different computer. The FAS1 should be calibrated in the new machine.
- 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.
- You have lost or replaced your "GAMRY.INI" file.

# **Chapter 3 -- Cell Lead Connections**

#### **Cell Connections**

Every FAS1 Femtostat is shipped with four cables used to connect the Potentiostat Module to an electrochemical cell. All operating modes – potentiostatic, galvanostatic and ZRA – use the same connections.

#### **Working Electrode**

The Working Electrode cable has an SMC coaxial connector on one end and a green banana plug on the other end. The cable is shipped with an alligator clip covering the banana plug.

The SMC connector screws into the Working connector on the Potentiostat module front panel. The banana plug end connects to the working electrode of an electrochemical cell. The working electrode is the specimen being tested.

In the potentiostat and ZRA modes this lead is the input to a virtual ground type current to voltage converter. In galvanostat mode, it is the virtual ground terminal of an op amp galvanostat. The potential at this point is thus kept near to FAS1's floating ground.

#### **Reference Electrode**

The Reference Electrode cable has an SMC coaxial connector on one end and a white pin jack on the other end. The cable is shipped with an alligator clip plugged into the pin jack.

The SMC connector screws into the Reference connector on the Potentiostat module front panel. The pin jack end connects to the reference electrode of an electrochemical cell.

This lead is usually connected to a standard reference electrode such as an SCE or Ag/AgCl electrode. In all operating modes, this lead is the input to a high input impedance electrometer. With the working electrode at a virtual ground, the output of this electrometer is the reference electrode potential versus the working electrode.

#### **Counter Electrode**

The Counter Electrode cable has an SMC coaxial connector on one end and a red banana plug on the other end. The cable is shipped with an alligator clip covering the banana plug.

The SMC connector screws into the Counter connector on the Potentiostat module front panel. The banana plug end connects to the counter electrode of an electrochemical cell.

The counter electrode (often called the auxiliary electrode) is usually a large inert metal or graphite electrode. The counter electrode terminal is the output of the FAS1's  $\pm$  30 mA at  $\pm$  12 volt control amplifier.

#### **Ground Connection**

The FAS1 is shipped with a lead suitable for most applications for the ground connector. One end connects to the Ground connector on the FAS1 front panel. The other end is equipped with a black alligator clip.

#### Chapter 3 -- Cell Lead Connections -- Cell Connections

The ground connector on the FAS1's front panel is connected to the FAS1's Floating Ground. This is the circuitry ground for the analog circuits in the FAS1.

When the FAS1 is used with a cell that is earth grounded, this terminal should be left disconnected. Earth grounded cells include most stress apparatus, autoclaves, and pipeline probes. In a typical pipeline probe, none of the electrodes (working, counter and reference) are connected to earth ground directly. However, the pipeline wall is effectively a fourth electrode which is earth grounded.

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 reduce noise in your data by connecting the FAS1's Floating Ground to a source of earth ground. A water pipe is often a suitable source of ground.

Caution: If any electrode is at earth ground, you <u>must not</u> connect the Floating Ground to earth. As mentioned above, autoclaves, stress apparatus, and field measurements often involve earth grounded electrodes.

Whenever you measure very small currents, you will need a metal enclosure completely surrounding your cell (a Faraday shield). This shield significantly lowers the current noise. The Faraday shield should be connected to the FAS1's Floating Ground. If your electrodes are all isolated from ground, you may also connect the shield to a source of earth ground.

## **Chapter 4 -- 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 already phase shifted feedback signal. 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.

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 FAS1 Femtostat's potentiostat is often 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 FAS1 has been tested for stability with cell capacitors between 10 pF and 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 FAS1 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 FAS1'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 FAS1 potentiostat/cell system. This list is not in any particular order. Any or all of these steps may help.

- Slow down the potentiostat. The FAS1 has 4 control amplifier speed settings, which can be selected in software. Slower settings are generally more stable.
- Increase the FAS1's I/E stability setting. The FAS1 includes 2 capacitors that can be paralleled 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 Lugin capillaries. If you do have a Lugin capillary, make sure that the capillaries'
  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 4-1. 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.

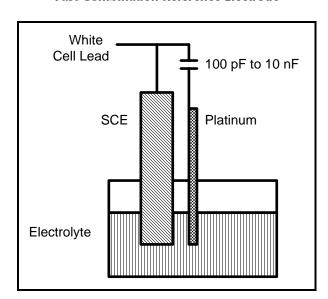
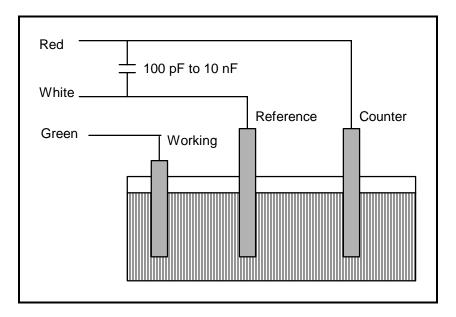


Figure 4-1
Fast Combination Reference Electrode

• 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 4-2. The capacitor value is generally determined by trial and error. One nanofarad is a good starting point.

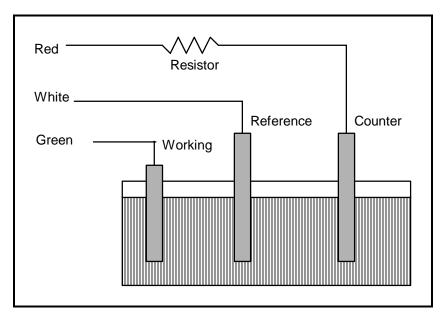
In a sense, this is another form of an AC coupled low impedance reference electrode. The counter electrode is the low impedance electrode, eliminating the need for an additional electrode in the solution.

Figure 4-2 High Frequency Shunt



• Add resistance to the counter electrode lead. See Figure 4-3. This change lowers the effective gain bandwidth product of the control amplifier. As a rule of thumb, the resistor should be selected 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.

Figure 4-3 Resistor Added for Stability



## **Chapter 5 -- Measurement of Small Signals**

#### **Overview**

The FAS1 operates near the limits of measurement science. It can resolve current changes as small as 1 femtoamp (10<sup>-15</sup> amps). To place this current in perspective, 1 fA represents the flow of about 6000 electrons per second!

The small currents measured by the FAS1 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 and fA currents. In many cases, the basic physics of the measurement must be considered.

This chapter will discuss the limiting factors controlling low current measurements. It will include hints on cell and system design. The emphasis will be on EIS (Electrochemical Impedance Spectroscopy), a major application for the FAS1.

### **Measurement System Model and Physical Limitations**

To get a feel for the physical limits implied by femtoamp measurements, consider the equivalent circuit shown in Figure 5-1. We are attempting to measure a cell impedance given by  $Z_{cell}$ .

This model is valid for analysis purposes even though the real FAS1 circuit topology differs significantly.

In Figure 5-1:

E<sub>s</sub> Is an ideal signal source

Z<sub>cell</sub> Is the unknown cell impedance

R<sub>m</sub> Is the current measurement circuit's current measurement resistance

 $R_{shunt}$  Is an unwanted resistance across the cell Is an unwanted capacitance across the cell

 $C_{in}$  Is the current measurement circuit's stray input capacitance  $R_{in}$  Is the current measurement circuit's stray input resistance

I<sub>in</sub> Is the measurement circuit's input current

In the ideal current measurement circuit  $R_{in}$  is infinite while  $C_{in}$  and  $I_{in}$  are zero. All the cell current,  $Z_{cell}$ , flows through  $R_{m}$ .

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

The voltage developed across  $R_m$  is measured by the meter as  $V_m$ . Given the idealities discussed above, one can use Kirchoff's and Ohms law to calculate  $Z_{cell}$ :

$$Z_{cell} = E_s * R_m / V_m$$

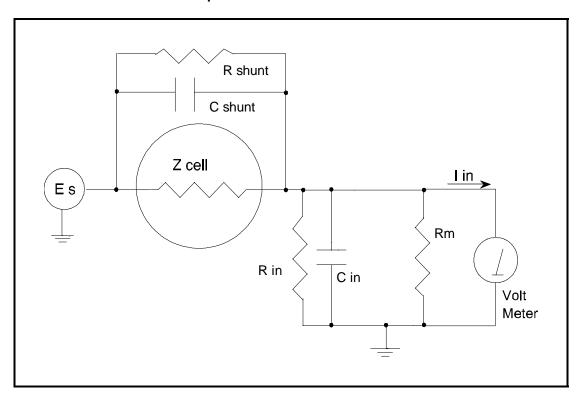


Figure 5-1
Equivalent Measurement Circuit

Unfortunately technology limits high impedance measurements because of:

- Current measurement circuits always have non-zero input capacitance, i.e.  $C_{in} > 0$
- Infinite R<sub>in</sub> 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<sub>shunt</sub> and a finite R<sub>shunt</sub>

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

## Johnson Noise in Z<sub>cell</sub>

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 equation.

$$E = (4 \text{ k T R } \delta F)^{1/2}$$

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

#### where:

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

 $T = temperature in {}^{\circ}K$ 

 $\delta F$  = noise bandwidth in Hz R = resistance in ohms.

For purposes of approximation, the Noise bandwidth,  $\delta F$ , is equal to the measurement frequency. Assume a  $10^{12}$  ohm resistor as  $Z_{cell}$ . At  $300^{\circ}K$  and a measurement frequency of 1 Hz this gives a voltage noise of  $129~\mu V$  rms. The peak to peak noise is about 5 times the rms noise. Under these conditions, you cannot make a voltage measurement of  $\pm$  10 mV across  $Z_{cell}$  without an error of up to  $\pm$  3%. 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 4  $\mu V$  rms.

Current noise on the same resistor under the same conditions is 0.129 fA. To place this number in perspective, a  $\pm$  10 mV signal across this same resistor will generate a current of  $\pm$  10 fA, or again an error of up to  $\pm$  3%. Again, reducing the bandwidth helps. At a noise bandwidth of 1 mHz, the current noise falls to 0.004 fA.

With  $E_s$  at 10 mV, an EIS system that measures  $10^{12}$  ohms at 1mHz is about 3 decades away from the Johnson noise limits. At 0.1 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 5-1 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°) can be calculated from:

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

Decreasing  $R_{\rm m}$  increases this frequency. However, large  $R_{\rm m}$  values are desirable to minimize voltage drift and voltage noise.

A reasonable value for  $C_{in}$  in a practical, computer controllable low current measurement circuit is 5 pF. For a 30 pA full scale current range, a practical estimate for  $R_m$  is  $10^{10}$  ohms.

$$f_{RC} = 1/6.28 (1x10^{10}) (5x10^{-12}) \approx 3 \text{ Hz}$$

In general, one should stay two decades below  $f_{RC}$  to keep phase shift below one degree. The uncorrected upper frequency limit on a 30 pA range is therefore around 30 mHz.

One can measure higher frequencies using the higher current ranges (i.e. lower impedance ranges) but this would reduce the total available signal below the resolution limits of the "voltmeter". This then forms one basis of statement that high frequency and high impedance measurements are mutually exclusive.

Software correction of the measured response can also be used to improve the useable bandwidth, but not by more than an order of magnitude in frequency.

### **Leakage Currents and Input Impedance**

In Figure 5-1, both  $R_{in}$  and  $I_{in}$  affect the accuracy of current measurements. The magnitude error due to  $R_{in}$  is calculated by:

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

For an  $R_m$  of  $10^{10}$  ohms, an error < 1% demands that  $R_{in}$  must be >  $10^{12}$  ohms. 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  $l_{in}$  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  $10^{12}$  ohm resistance between +15 volts 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 rule of thumb, the DC leakage should not exceed the measured signal by more than a factor of 10.

The FAS1 uses an input amplifier with an input current of around 50 fA. Other circuit components may also contribute leakage currents. You therefore cannot make absolute current measurements of low fA currents with the FAS1. In practice, the input current is approximately constant, so current differences or AC current levels of a few fA can 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 circuit. 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 1  $\mu$ F. This could represent a passive layer on a metal specimen. The impedance of this electrode, assuming ideal capacitive behavior, is given by

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

At sixty Hertz, the impedance value is about 2.5 k $\Omega$ .

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 frequency.

Assume a reasonable noise voltage,  $V_n$ , of 10  $\mu V$ . Further, assume that this noise voltage is at the US power line frequency of 60 Hz. It will create a current across the cell capacitance:

$$I = V_n/Z \approx 4 \text{ nA}$$

This rather large noise current will prevent accurate DC current measurement in the pA ranges.

In an Electrochemical Impedance measurement, you apply an AC excitation voltage that is much bigger than the typical noise voltage, so this is not a factor.

#### **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 either metal 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 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 30 pF capacitance, 10 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 (one with infinite resistance). Even Teflon<sup>TM</sup>, which is one of the best insulators known, has a bulk resistivity of about  $10^{14}$  ohms. 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 FAS1 Potentiostat Mode specifications in Appendix A contain equivalent values for the potentiostat's  $R_{shunt}$  and  $C_{shunt}$ . 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 <u>mandatory</u> 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 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.

If possible, place the FAS1 potentiostat module within the Faraday shield. Your computer should be kept outside of the shield. The opening in the shield should be just slightly larger than the cable between the computer and FAS1 potentiostat module.

The shield must be electrically connected to the FAS1's ground terminal.

An additional connection of both the shield and the FAS1 ground to an earth ground may also prove helpful.

NOTE: Only connect the FAS1 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. The one exception is the FAS1 potentiostat module itself.

### **Cell Lead Length and Construction**

The FAS1 has three true cell leads. They are the leads for the Working Electrode, Reference Electrode, and Counter Electrode. The cell leads connect to the FAS1 potentiostat module via SMC miniature coaxial connectors.

The cell leads are made from coaxial cable with a virgin Teflon dielectric.

The FAS1 is shipped with short cell leads. Use of longer cell leads may cause significant problems. These include potentiostat oscillation, loss of bandwidth, EIS phase shifts and additional noise.

The FAS1 cell leads are also built for very good isolation between the inner conductor and the shield. Both commercial and home built SMC cables are unlikely to offer as good an isolation resistance.

None of the above discussion applies to the Ground connection of the FAS1. In most cases, any type of cable may be used in this connector. Lead length is generally not a problem. The only exception occurs in floating operation on an earth grounded cell, where any connections made to the ground terminal should be very well insulated from earth ground. Teflon insulated wire is preferred if a connection to the ground is required at all when floating.

#### **Lead Placement**

Many experiments with the FAS1 involve cells with very small capacitances, the value of which may be important.

In these cases, the capacitance between the FAS1's leads can result in an error. The FAS1 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 the working electrode lead.
- 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 center conductor to shield isolation.

The cell leads must not be moved during an experiment which measures 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  $10^{10}$  ohms cannot be used to measure  $10^{13}$  ohm impedances. In general, glass and Teflon are the preferred cell construction materials. Even glass may be a problem if it is wet.

You also must worry about  $C_{\text{shunt}}$ . Make the "inactive" portion of your electrodes as small as possible. Avoid placing electrodes close together or parallel with each other.

#### **Reference Electrode**

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

Try to avoid:

- Narrow bore or Vycor tipped Lugin capillaries.
- Poorly conductive solutions especially in Lugin capillaries.
- Asbestos thread and double junction reference electrodes.

#### **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 1pA of AC current on top of 1 mA of DC current.

#### **EIS Speed**

In EIS, do not expect the FAS1 to measure 10<sup>12</sup> ohm 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, must be less than  $10^{10} \Omega Hz$  for good EIS measurements with an FAS1.

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

#### **Ancillary Apparatus**

Do not use the FAS1 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 FAS1 cell leads.

## **Floating Operation**

The FAS1 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 FAS1'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 FAS1 is operated in a floating mode. The instrument specifications only apply on isolated cells with the FAS1 earth ground referenced (not floating).

Special precautions must be taken with the cell connections when the FAS1 must float. Make sure that all the cell connections are isolated from earth ground. In this case, even the Ground terminal of the FAS1 must be kept isolated.

#### Chapter 5 -- Measurement of Small Signals -- Floating Operation

The case of the FAS1 must also be isolated from earth ground. Do not set the FAS1 potentiostat module down on a wet dirt surface and expect good floating measurements.

Finally, most ancillary apparatus connected to the cell of the FAS1 must be isolated. External voltmeters, ammeters, FRA's etc. must be isolated. This includes devices connected to the monitor connectors located on the FAS1 controller board minipanel.

# **Appendix A -- Specifications**

## **Control Amplifier**

Compliance Voltage	± 12 volts @ 30 mA
Output Current	± 30 mA
Unity Gain Bandwidth (software selectable)	>1 MHz, > 200 kHz, > 20 kHz
Slew Rate	>8 V/µsec

## **Electrometer - Fast Mode**

Input Impedance	$> 10~\text{G}\Omega$ in parallel with 20 pF
Input Current	< 50 pA
Bandwidth (-3dB)	> 5 MHz

### **Electrometer - Slow Mode**

Input Impedance	$>$ 1 T $\Omega$ in parallel with 8 pF
Input Current	< 100 fA
Bandwidth (-3dB)	> 250 kHz

## **Voltage Measurement**

Full Scale Ranges	$\pm$ 30V ( $\pm$ 12 V usable), $\pm$ 3V, $\pm$ 300 mV, $\pm$ 30 mV
Resolution	16 Bits
DC Accuracy	± 0.2% Range ± 1mV
Offset Range	± 12 V with 1.5 mV resolution

#### **Current Measurement - All**

Analog Full Scale Ranges	$\pm$ 30 pA to $\pm$ 30 mA in decades
Controller Board Gains	1, 10, 100
Resolution	16 bits
Offset Range	± 4X full scale (only 2X full scale is useful)

## **Current Measurement - High Speed**

DC Accuracy	± 0.2% range ± 1nA
Bandwidth (-3 dB)	1 MHz (30 μA30mA)
	100 kHz (3 μA)
	100 Hz (300 pA)

## **Current Measurement - Low Speed**

DC Accuracy	± 0.2% range ± 1 pA
Bandwidth (-3 dB)	250 kHz (30 μA30mA)
	25 kHz (3 μA)
	10 Hz (30 pA)

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

Range	± 3 volts differential
Bandwidth	20 Hz
Input Impedance	100 kΩ

## **Auxiliary D/A Output**

Range	± 5 volts or 0 to 10 volts
Resolution	2.5 mV

### **Environmental**

Operating Temperature	0-70 °C (inside computer)
Specification Temperature	25 °C

### **Potentiostat Mode - All**

Applied E Range	± 11 volts
Accuracy	$\pm$ 2 mV $\pm$ 0.3% of setting
DC Bias	± 8 V
Scan Ranges	$\pm$ 6.4 V, $\pm$ 1.6V, and $\pm$ 0.4 V
Resolution	200 μV/bit, 50 μV/bit, 12.5 μV/bit
Drift	< 30 μV/C
Noise and Ripple	< 20 μV rms (1Hz - 10 kHz)
Equivalent Cell Shunt - Resistance	$> 2 \times 10^{13} \Omega$
Equivalent Cell Shunt - Capacitance	< 500 fF

### **Galvanostat Mode**

Applied i range	± full scale current (no 30 pA range)
DC accuracy	± 0.3% full scale
Scan Ranges	± 2X full scale current

# **Current Interrupt**

Measurement Type	(sample 2 points on decay, extrapolate)
Cell Switching Time	$<$ 1 μsec (1 k $\Omega$ cell )
Minimum Interrupt Time	15 μsec
Maximum Interrupt Time	64 msec

# A/D converter

Resolution	16 bits
Accuracy	0.1% of full scale
Timing	50 μsec to 600 sec

# **I/O Connections**

Analog Outputs	Voltage, Potential
External Input (added to Applied E or I)	10 kΩ input Z
Digital	4 inputs, 4 outputs

# Other

Power	20 W maximum
	1.4A at 12 volts
	300 mA at +5 V
	80 mA at -5 V
	20 mA at -12 V
Leakage i (floating, earthed Working Electrode)	< 5 pA @ DC

### NOTES:

- All specifications subject to change without notice
   Offset specifications apply after software calibration

# **Appendix B -- Changing The Default FAS1 Settings**

#### Overview

Your FAS1 Femtostat can be configured for the specific needs of your computer system. A DIP switch on the FAS1 allow you to:

- Choose the I/O addresses used by the FAS1.
- Reconfigure an FAS1 as Pstat 1 through Pstat 4 in a multiple potentiostat system.

Whenever an FAS1 setting is changed, the software controlling that FAS1 must be made aware of the change.

Gamry Instruments' software reads the FAS1's settings from an initialization file called "GAMRY.INI". This file lists the hardware configuration for any Gamry potentiostats in the system.

The "GAMRY.INI" file does not merely record DIP switch settings. It also contains software information and information about software setup of hardware. This includes:

- Interrupt level
- Auxiliary D/A range
- Software calibration data

The "GAMRY.INI" file is located in the Windows directory. It is an ASCII text file that is divided into sections identified by a section name in square brackets (e.g. [EIS300]). Each section contains settings for a specific aspect of the system.

Turnkey systems are provided with a "GAMRY.INI" file appropriate for the items you have purchased with that system. The "GAMRY.INI" file in user installed systems is usually created by the Setup programs that come with Gamry Instruments' Windows based application software. Most configuration information should be changed by rerunning the Setup programs.

Use this appendix when you need to change the system configuration via changes to your DIP switch settings or via manual changes in your "GAMRY.INI" file.

#### About the "GAMRY.INI" File

Gamry Instruments' Windows based software is configured by means of the "GAMRY.INI" file. The information in this file is used to:

- Identify each Potentiostat in the system.
- Determine the I/O address and interrupt level used by the system's potentiostats.
- Authorize use of a specific potentiostat by specific software packages.
- Store calibration data for each potentiostat.
- Store scaling factors for system D/A and A/D converters.
- Store software configuration information.

The "GAMRY.INI" file is an ASCII file. You can modify the file using an ASCII editor or a word processor in a non-document mode (a mode with no formatting codes in the text). The Windows Notepad accessory is a convenient ASCII editor.

The copy of "GAMRY.INI" actually used by the software must be located in the Windows directory (normally C:\WINDOWS). The Setup programs provided with Gamry Instruments' software either install "GAMRY.INI" in the correct directory or modify an existing file in this location.

A portion of a typical "GAMRY.INI" file is shown in Figure B-1. Only some of the information required for FAS1 configuration is shown. A complete "GAMRY.INI" file is longer than this example.

In Figure B-1, the 1st line is called a section identifier. The name of the section is enclosed in square brackets, e.g. [Framework]. The [Framework] section extends to the next section identifier [InteruptList]. The [Framework] section contains configuration information for the Framework program. This section is required in all "GAMRY.INI" files that configure a Gamry Framework System.

The section labeled [DeviceList] contains a list of all the potentiostats in the system. Each entry in this section corresponds to a potentiostat in the system. The entry name is the name of the "GAMRY.INI" section that describes this potentiostat. In the sample file, the [DeviceList] contains only one entry (for Pstat0).

"GAMRY.INI" also contains potentiostat calibration information. Each potentiostat's data is in its section. You do not normally have to edit the calibration data which is automatically created and updated by the calibration routine built into the software.

The [Pstat#] section also contains the AUXDACRES field which you may need to change. See below for details.

# Figure B-1 Portion of a Typical "GAMRY.INI" File

[Framework]

LineFreq=60

PACKAGE0=EIS300

[InterruptList]

IRQ10=

[DeviceList]

Pstat0=

[Pstat0]

;This is a PC4 Femtostat Section

Label=Fstat1

CMSDriver=PC4.DLL

IRQLevel=10

BaseAddress=0x120

PstatClass=PC4

BoardNo=1

FraCurveClass=FRACURVE4

AuthEIS300=1234567890

# Changing "GAMRY.INI" using Setup

The Setup program that comes with Gamry Instruments' software can automatically make changes in the "GAMRY.INI" file. You can use this program to change the base address, the interrupt level, and FStat identifier information in "GAMRY.INI".

The Setup program for each Gamry Instruments' application software package can generally be used to alter the sections of the "GAMRY.INI" file that apply to that application.

# Using Notepad to alter "GAMRY.INI"

It is often convenient to use an ASCII editor to make small changes in "GAMRY.INI". The Notepad accessory included with Windows is a useful ASCII editor. Please read the Microsoft documentation for full instructions about using it.

### Adding a New FAS1 to an Existing System

To add a new FAS1 to a system that already contains one or more FAS1s, you need to do the following:

- Set the board number switch for the FAS1 Potentiostat to be added.
- Add the potentiostat card set to the computer.
- Add potentiostat information to "GAMRY.INI".

### Setting the Board Number Switch on an FAS1 Potentiostat

Each potentiostat in a multiple potentiostat system must be set for a unique board number. The board number is set using a DIP switch on the Controller Card.

To change the board number switch settings on an FAS1 proceed as follows:

- 1. Determine the desired board number for this FAS1. Board numbers should be sequential starting with board number 1. For example, a two potentiostat system has its cards configured as board numbers 1 and 2.
- 2. Locate S101, the four pole DIP switch in the middle right side of the Controller Card.
- 3. The ON position of the switch is marked on the body of the switch.
- 4. The order of the switches is S101-1 on the left, S101-4 on the right. This also is marked on the body of the switch.
- 5. Set the switches S101-1 and S101-2 from Table B-1. The switch settings are read across the row labeled with the desired board number. Do not change the settings of S101-3 and S101-4!
- 6. Double check that the change has been made correctly.
- 7. Label the outside of the metal bracket with the new board number.

Table B-1
Switch Setting for Different Board Numbers

Board	S10	01-
Number	1	2
1	ON	ON
2	OFF	ON
3	ON	OFF
4	OFF	OFF

### **Installing the FAS1 in the Computer**

Please follow the instructions in Chapter 2 to install the new card set in the computer. You will need two card slots for each additional FAS1 to be installed.

### Adding Potentiostat Information to "GAMRY.INI" using Setup

The easiest way to add the new information to "GAMRY.INI" is to rerun the Gamry Instruments Setup program. You can skip over the file copy portion of Setup. Enter the new information in the dialog box that asks for potentiostat information.

### Manually Adding Potentiostat Information to "GAMRY.INI"

Before you can add potentiostat information to "GAMRY.INI", you need to understand how potentiostats are identified in the file. We will use the sample "GAMRY.INI" file in Figure B-1 as an aid in our discussion.

Look at the [DeviceList] section in Figure C-1. The line "Pstat0=" declares that there is a potentiostat in the system that is further described in a [Pstat0] section. A field labeled "Pstat1=" would identify a second potentiostat, "Pstat2" the third potentiostat, and so on.

Suppose you are adding another FAS1 Potentiostat to this system. You must add the line:

Pstat1=

to the [DeviceList] section of your "GAMRY.INI" file.

You also must add a [Pstat1] section to the file. In general, you can copy the information in an existing section. Make sure that you change the BoardNo= field to match the setting on the board.

You may also have to add one or more authorization codes to the new [Pstat1] section of "GAMRY.INI". Each Gamry Instruments program requires a unique 10 digit authorization code before it will use a specific potentiostat. If the Framework does not find a valid authorization code, it will not take data. See the application's Installation Manual for more information on this topic.

Your original shipping documentation should contain all the authorization codes that you need to operate your system.

If you do use Notepad to alter "GAMRY.INI", you must restart Windows before you can be sure the change to "GAMRY.INI" is effective.

### Removing a Potentiostat from an Existing System

To remove a potentiostat from a system you need to do two things. The first is to physically remove the card set from the computer. The second is to remove the potentiostat's PstatX= field from the [DeviceList] section in the "GAMRY.INI" file (where X stands for the zero based board number of the potentiostat you are removing).

### **Interrupt Level Setting**

Most peripheral devices in an AT compatible computer coordinate I/O (input/output) operations with the microprocessor by means of hardware interrupts. An interrupt is a request by a device that the computer suspend the program it's currently running, and perform an I/O operation. The FAS1 Potentiostat generates an interrupt at the end of each data point.

An IBM AT compatible computer allows for 16 levels of hardware interrupts. In a Gamry Instruments system, all the potentiostats use the same interrupt level. This interrupt level cannot be used by other system functions or expansion cards.

Unfortunately, very few of the 16 interrupt levels are not used by AT compatible system functions or the "common" expansion cards (e.g. video cards, serial ports, disk controllers, etc.). The interrupt levels available to the FAS1 card set were chosen as those most likely to be free for FAS1 use in a normal computer

configuration. Table B-2 lists these levels along with any conflicts with levels assigned in the IBM AT Technical Reference Manual.

Table B-2
Interrupt Level Selection

Interrupt	Also
Level	used for
IRQ5	LPT2
IRQ10	
IRQ11	
IRQ15	

NOTE: All the Gamry potentiostats in a computer are generally set for the same interrupt level.

To change the interrupt level used by the potentiostats in your system, you simply change a setting in the "GAMRY.INI" software initialization file.

If you have any uncommon expansion cards in your computer, you should consult their documentation to determine if they can generate system interrupts. If they can, you should determine their interrupt level setting(s), and select an Interrupt Level for your FAS1(s) that does not conflict with these cards.

For example, if you determine that your computer contains an ethernet card that generates interrupts on level 10, the interrupt level you choose for your FAS1(s) cannot be 10. Assume that you choose interrupt level 11. Follow these instructions to change the level.

- 1. Edit "GAMRY.INI" in the Windows directory. Windows Notepad is a convenient ASCII editor.
- 2. In this file, locate the [InterruptList] section.
- 3. Add an entry for the new level (if its not already present). The entry has the form "IRQXX=" where XX is the level to be used. For the default IRQ setting the entry is "IRQ10=".
- 4. Make sure that every [PstatN] section in the "GAMRY.INI" file has its IRQLevel field set to the new value. In the default file, each [PstatN] section should contain the line "IRQLevel=10".
- 5. Save the edited file.

The information in the altered "GAMRY.INI" file is not effective until you exit and restart the Gamry Framework. If you have unsuccessfully attempted to run an experiment on the wrong level, you may have to power down and restart your computer before interrupts will occur normally.

### **Changing your I/O Register Address**

The FAS1 Femtostat card set, like virtually all IBM compatible expansion cards, has hardware registers that the computer must be able to access. These registers are located at a specific address in the I/O (input/output) address space of the computer's microprocessor. Addresses are expressed in the hexadecimal numbering system where the digits are 1,2,3,4...8,9,A,B,C,D,E,F. We will always precede a hexadecimal number with the prefix 0x, e.g. 0x22F.

The FAS1 requires 32 I/O register addresses. Unlike most expansion cards, which cannot share I/O addresses, all the FAS1's in a system are normally set up to use the same I/O register addresses. The board number

assigned to each PC3 prevents harmful address clashes among FAS1s. However, the I/O address range used by the FAS1(s) still must not overlap with the I/O addresses used by any other device in your computer.

The 32 I/O addresses of the FAS1 card can be selected to appear at a variety of locations in the I/O address space of the computer. Table B-3 is a list of the locations that can be set using the DIP switch on the Controller Card. Addresses are given as a base (starting) address of the 32 byte I/O address range.

Table B-3 I/O Address Selection

Base	S101-	S101	Address Ra	nge
Address	3	4	hex	decimal
0x220	ON	ON	0x220-0x23F	544-575
0x120	ON	OFF	0x120-0x13F	288-319
0x240	OFF	ON	0x240-0x25F	576-607
0x140	OFF	OFF	0x140-0x15F	320-351

If you have any uncommon expansion cards in your computer, you should consult their documentation to determine the location of their I/O registers. If their register locations conflict with the default address range of 0x120-0x13F, determine a new base address from Table B-3 that does not conflict with any of your cards.

For example, suppose you determine that your computer contains a tape backup controller that uses I/O registers at addresses 0x130-0x13F. These clash with the default FAS1 I/O addresses 0x120-0x13F. Therefore, the FAS1 base I/O address must be changed. Assume that you choose a new base I/O address of 0x140. This base I/O address level and its associated S101 switch settings should be entered into the FAS1 DIP switch and the "GAMRY.INI" file.

To change the I/O Address on your FAS1s, proceed as follows:

- 1. Determine the desired I/O address. See above discussion.
- 2. Locate S101, the four pole DIP switch in the middle right side of the Controller Card.
- 3. The ON position of the switch is marked on the body of the switch.
- 4. The order of the switches is S101-1 on the left, S101-4 on the right. This also is marked on the body of the switch.
- 5. Set the switches S101-3 and S101-4 from Table B-3. The switch settings are read across the row labeled with the desired I/O address. Do not change the settings of S101-1 and S101-2!
- 6. Double check that the change has been made correctly.
- 7. Repeat 1-6 for any other FAS1s Potentiostats in your system.

The procedure for manually changing "GAMRY.INI" to reflect the new I/O address setting is:

- 1. Edit "GAMRY.INI" in the Windows directory. Windows Notepad is a convenient ASCII editor.
- 2. In each [PstatN] section, locate the line BaseAddress=0xYYY, where YYY is the old I/O base address. For the default "GAMRY.INI" YYY is 120.
- 3. Enter the new base address in place of the YYY in this line. For example, if the new base address is 0x140, the line should read BaseAddress=0x140.
- 4. Save the edited file.

The information in the altered "GAMRY.INI" file is not effective until you exit and restart the Gamry Framework.

# **Changing the Auxiliary Analog Output Scaling**

A setting in the "GAMRY.INI" file allows you to change the scaling of the D/A converter used to generate the auxiliary analog output.

The default setting configures this D/A converter for a bipolar output of  $\pm$  5 volts with a bit resolution of 2.5 mV/bit. You can switch to a unipolar output of 0 to 10 volts, still with a bit resolution of 2.5 mV/bit.

The D/A scaling is controlled by a field in the [PStat#] section of the "GAMRY.INI" file. This field has the form:

The final 0 indicates that the scaling is bipolar. If this digit was a 1, the scaling would be unipolar.

# Appendix C -- I/O Connections for the FAS1

The FAS1 Femtostat has a number of connectors that allow it to communicate electronically with the world outside of the computer. This appendix describes these connectors and the signals available on their pins.

#### **Grounds and the FAS1 Femtostat**

The FAS1 has been specially designed for operation with cells in which one of the electrodes is connected to earth ground. Earthed electrodes often occur in field experiments, because metal pipelines and structures are generally earth grounded. In the lab, experiments involving either autoclaves or stress apparatus often have earth grounded electrodes. Conventional potentiostats do not work properly or safely in these experiments. In the typical glass or plastic test cell, none of the electrodes are earthed, so no grounding problems arise.

The FAS1 analog circuits are electrically isolated from the computer's chassis which is at earth ground. Another name for circuits that are isolated is "floating". The isolation is accomplished by means of optical isolators and transformers.

Extraordinary measures were taken in the design of the FAS1 to maximize the degree of isolation. However, you can still measure higher impedances (smaller currents) on cells that are not earth grounded than you can on earth grounded cells.

If you are working with earthed electrodes, ground connections for your potentiostat are critical. You must be careful that the floating ground connection on all FAS1 connectors does not get connected to earth ground.

We strongly recommend that you use an earth grounded Faraday shield whenever you are measuring small currents. See Chapter 5 for a discussion of Faraday shields.

### **The Cell Connections**

Basic cell connections are described in some detail in Chapter 3. That information will not be repeated here.

In a few cases, you may find that you need cell connection cables that differ from those shipped with your FAS1. By far the easiest changes involve modifying a standard cable. We can sell you an extra cable for this purpose.

If you need to modify a cable or make a completely new one, the following information may be helpful.

- SMC connectors are available from several manufacturers.
- SMC adapters to other cable types including BNC cables are also generally available.
- We recommend that you use shielded coax cables for all the cell connections.
- Connect the shield of the coax at the SMC connector end of the cable. Leave the shield open on the cell end.
- Do not ground any shields!
- Use good quality cable and keep the termination clean. You want at least 1000 G $\Omega$  of isolation between the shield and the inner conductor.
- Longer cables can dramatically slow the FAS1, especially on sensitive current ranges. This may cause potentiostat stability problems.

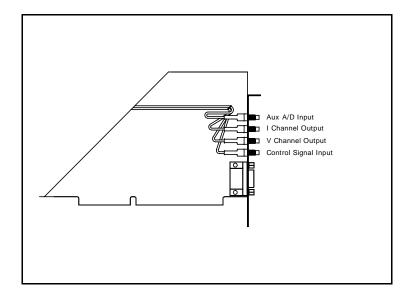
### **Control Signal Input**

The Control Signal Input allows you to inject a signal into the FAS1's potential or current control circuits. One use for this input is modulation of the applied voltage or current.

This input is the lowest SMC connector on the Controller Card minipanel. See Figure C-1 for the identity of all the SMC connectors on this minipanel.

Note that the shell of this BNC connector is connected to the FAS1's floating ground. Connecting an earth ground referenced signal source to this input will cause problems if you are using the FAS1 with a cell that has an earth grounded electrode.

Figure C-1
SMC Connectors on the FAS1 Controller Card Minipanel



In controlled potential mode (and ZRA mode), the potential applied to the cell is the sum of the applied potential and the control input voltage. For example, if the programmed voltage is +2 volts, and +1 volt is applied to the control input, the cell voltage ( $E_{work}$  -  $E_{ref}$ ) will be +3 volt. The input impedance of this input is  $10 \text{ k}\Omega$ . Adding a control resistor,  $R_{ext}$ , in series with the input allows you to alter the scaling factors. The equation describing the relationship is:

$$V_{cell} = V_{sig} \times 10 \text{ k}\Omega/(R_{ext} + 10 \text{ k}\Omega)$$

 $V_{sig}$  is the signal applied to the resistor and  $V_{cell}$  is the resulting cell voltage. If 90 k $\Omega$  is added in series, a 1 volt signal will be attenuated to cause only a 100 mV cell voltage.

In controlled current mode, you will get full scale current for 3 volts applied to this connector. The current will vary with the current range. For example, on the 30 mA range, 1.5 volts will give you 15 mA of cell current. The sign is such that a positive input gives you a cathodic current.

### Aux A/D Input

This input allows you to measure an externally generated voltage signal. The Aux A/D input is the upper of the SMC connectors on the FAS1 Controller Card's minipanel. See Figure C-1 for the identity of all the SMC connectors on this minipanel.

Uses of this input include the measurement of temperature, strain, or other non-electrochemical parameters. This input is fully differential, with about 80 dB of common mode rejection. Be careful though, the allowed

common mode voltage range is only  $\pm$  11 volts with respect to the floating ground. Voltages outside this range should not damage the instrument but they cannot be measured.

The input impedance is 25 k $\Omega$  on each leg of the differential input. You may have to take this into account if the signal source connected to this input has a significant output impedance.

The scaling on this signal is  $\pm$  3 volts full scale, resulting in  $\pm$  30,000 counts on the A/D converter.

### **V Channel Output**

This output reflects the cell voltage signal as seen by the FAS1 A/D converter. The V Channel output is the lower-middle SMC connector on the FAS1 Controller Card's minipanel. See Figure C-1 for the identity of all the SMC connectors on this minipanel.

The signal on this connector has been through a long and complex analog signal processing chain. It may have been filtered, offset by a DC voltage, and gained. All of these functions are under computer control. The meaning of the signal on this connector is therefore highly dependent on the program controlling the FAS1 and cannot be described simply here. The one thing that can be said concerns polarity. If this signal becomes more positive as a result of changes in the cell potential, the cell voltage has become more anodic.

Note that the shell of this SMC connector is connected to the FAS1's floating ground. Connecting an earth ground referenced measurement device to this output can cause problems if you are using the FAS1 with a cell that has an earth grounded electrode.

### **I Channel Output**

This output reflects the cell current signal as seen by the FAS1 A/D converter. The I Channel output is the upper-middle SMC connector on the FAS1 Controller Card's minipanel. See Figure C-1 for the identity of all the SMC connectors on this minipanel.

The signal on this connector has been through a long and complex analog signal processing chain. It may have been filtered, offset by a DC voltage, and gained. All of these functions are under computer control. The meaning of the signal on this connector is therefore highly dependent on the program controlling the FAS1 and cannot be described simply here. The one thing that can be said concerns polarity. If this signal becomes more positive as a result of changes in the cell potential, the cell current has become more cathodic.

Note that the shell of this SMC connector is connected to the FAS1's floating ground. Connecting an earth ground referenced measurement device to this output can cause problems if you are using the FAS1 with a cell that has an earth grounded electrode.

# Miscellaneous I/O Connector

This connector contains a number of chassis ground related signals. It is the miniature 15 pin female D shaped connector on the FAS1 Controller Card. Be careful, the ground on this connector is not the FAS1 floating ground. Connecting the two grounds may lead to problems if you are using the FAS1 in a floating mode.

The auxiliary analog output, derived from a D/A converter, is on this connector. The scaling is normally 2.5 mV per bit, for a  $\pm 5$  volt full scale range. These ranges can be altered via the AUXDACRES field in the "GAMRY.INI" file.

The pinout of this connector is shown in Table C-3.

Table C-3 Miscellaneous I/O Connector

Pin	Name	Use
1	Analog Output Low	The auxiliary output ground connection.
2	Analog Output High	The auxiliary output signal.
3	no connection	
4	Start of Point	A TTL pulse before the start of a data point
5	End Of Point	A 1 μsec TTL pulse at the end of each data point
6	Ground	Digital ground
7	Digital Out 0	A CMOS digital output- 330 $\Omega$ output impedance
8	Digital Out 1	A CMOS digital output- $330\Omega$ output impedance
9	Digital Out 2	A CMOS digital output- $330\Omega$ output impedance
10	Digital Out 3	A CMOS digital output- $330\Omega$ output impedance
11	Digital In 0	A TTL digital input- 2.2 kΩ input impedance
12	Digital In 1	A TTL digital input- 2.2 kΩ input impedance
13	Digital In 2	A TTL digital input- 2.2 kΩ input impedance
14	Digital In 3	A TTL digital input- 2.2 kΩ input impedance
15	+5 Volts	Power- 100 mA maximum current

Appendix C -- I/O Connections for the FAS1 -- Miscellaneous I/O Connector

# Appendix D – Auxiliary A/D Input Characteristics

### **Overview**

The Controller Card used in the Gamry Instruments PC4 Potentiostat and the FAS1 Femtostat has undergone a number of changes over its lifetime.

One of the changes was a major modification of the input circuitry used for the Aux A/D function. Early revisions of the Controller Card only allowed connection of low impedance, quiet signals to this input. Revision F (and higher) cards allow for connection of higher impedance signals and for filtering of noisy input signals prior to A/D conversion. Three new jumpers added to the board control access to these features.

### **Identifying Your Controller Card Revision**

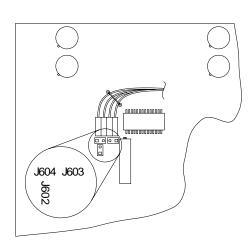
A label on the lower left side of your Controller Card will identify the revision of the card. The revision generally consists of a capital letter followed by a number. The letter identifies the printed circuit board revision. For example, revision F1 uses a revision F printed circuit board. The bulk of the descriptions in this document do not apply if you have a board revision lower than Rev F.

#### **Revision E and Lower – Characteristics**

Revision E (and lower) boards have an Aux A/D input with fixed characteristics. The input impedence is 25 k $\Omega$  and the bandwidth is not limited by a filter. If your board is earlier than revision F, disregard the rest of this appendix because it does not apply to your instrument.

### **Jumper Identification**

The three jumpers that configure the Aux A/D input are in a cluster located at the left side of the Controller Card. The four coax cables that route analog I/O to the card enter the card just above these jumpers. J603 and J604 are located immediately below the cables. J602 is lower and perpendicular to J603 and J604. See the figure to the right for jumper locations.



### **Input Impedance Selection**

Two jumpers are associated with the input impedance – J603 and J604. With J603 and J604 installed, the Aux A/D inputs have 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 SMC connector.

With both J603 and J604 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 these jumpers, <u>do not</u> calibrate the potentiostat unless you have a cable connecting both Aux A/D inputs to floating ground.

### **Filter Selection**

J602 controls filtering of the Aux A/D input. With J602 removed, there is no filtering on this input. With J602 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 $\Omega$  will 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 will be less than 0.25 Hz.

# **Revision F or Higher- Aux A/D Specifications**

These specifications for the Aux A/D apply to revision F or higher boards:

Range	±3.276 volts	
Input Impedance	100 k $\Omega$ (approx) or	J603 & J604 installed
	10 G $\Omega$ (typical)	J603 & J604 removed
Input Bias Current	< 10 nA	J603 & J604 removed
Filter Cutoff	20 Hz ± 20%	J602 in place with Zsource $<$ 1 $k\Omega$

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