VersaSTAT LC

Low Current Interface (LCI)

User’s Guide Manual

VersaSTAT LC Low Current Interface

Safety Instructions and Symbols

This manual contains up to three levels of safety instructions that must be observed in order to avoid personal injury and/or damage to equipment or other property. These are:

DANGER Indicates a hazard that could result in death or serious bodily harm if the safety instruction is not observed.

WARNING Indicates a hazard that could result in bodily harm if the safety instruction is not observed.

CAUTION Indicates a hazard that could result in property damage if the safety instruction is not observed.

Please read all safety instructions carefully and make sure you understand them fully before attempting to use this product.

Cleaning Instructions

WARNING: Using this instrument in a manner not specified by the manufacturer may impair the protection provided by the instrument.

To clean the instrument exterior:

- Unplug the instrument from all voltage sources.
- Remove loose dust on the outside of the instrument with a lint-free cloth.
- Remove remaining dirt with a lint-free cloth dampened in a general-purpose detergent and water solution. Do not use abrasive cleaners.

CAUTION: To prevent moisture inside of the instrument during external cleaning, use only enough liquid to dampen the cloth or applicator.

- Allow the instrument to dry before reconnecting the power cord.

1. INTRODUCTION

1.1. About This Manual

This manual covers the installation and use of the VersaSTAT LC Low Current Interface (Figure 1) option for the VersaSTAT® series of potentiostats/galvanostats. The VersaSTAT LC is also referred to as the LCI option.

This manual does not discuss software installation or operation outside of the calibration procedure (Section 2.2). For software operation, please refer to the VersaStudio™ operation manual.

Chapter 2 describes how to properly install, calibrate, and test the LCI option.

Chapter 3 discusses the measurement of small current signals with recommendations on shielding and cell design.

Chapter 4 gives the physical and electrical specifications of the LCI.

Figure 1. VersaSTAT LC assembly.

1.2. About the Low Current Interface (LCI)

The Low Current Interface (LCI) is a plug-in, research grade option for the VersaSTAT® Series of potentiostat/galvanostat systems (Table 1) that has been designed to enhance the measurement resolution and accuracy of extremely small current signals.

PAR Potentiostat/Galvanostat models that support the VersaSTAT LC Low Current Interface
VersaSTAT 3
VersaSTAT 4
VersaSTAT 3F*
VersaSTAT MC

Table 1.

*V3F does not operate in float mode with LCI attached.

The LCI consists of:

1) Interface cable that connects the cell cable interface of the potentiostat/galvanostat to the LCI main body.
2) LCI main body.
3) Four (4) coaxial cell leads that connect from the LCI main body to the electrodes and/or cell under test, as well as a ground lead.

1.2.1. LCI Interface Cable

The LCI-potentiostat interface cable (part number 223763, shown below) is a 1m long cable designed specifically for the LCI. The cable is bi-directional, meaning that both ends are identical and may be connected to either the LCI or potentiostat interfaces.

CAUTION: This cable is not to be modified from its original configuration.

1.2.2. LCI Main Body

The main body of the LCI is 19cm X 15cm X 4.5cm assembly. The cell cable panel (shown below-left) has the coaxial connections for the four cell cables, as well as a ground connection. Also, pin-jack connections for the cell cables are provided that connect across a built-in 10Mohm (10E7) resistor that is to be used to provide feedback when the LCI does not have a cell to connect to, and/or it can be used as a “dummy cell” for QA/QC or diagnostic purposes. The potentiostat interface panel has the interface connection and power LED.

LCI Cell Cable Panel (left) and Potentiostat Interface Panel (right)


LCI with cell cables connected across internal feedback (dummy cell) resistor. The WE cable connects to the W pin jack, the SE cable connects to the S pin jack, the RE cable connects to the R pin jack and the CE cable connects to the C pin jack.

1.2.3. LCI Cell Cables

The cell cables (WE,SE,RE, and CE) and ground leads that connect to the LCI are approximately 40cm in length, The cell cable terminate at the LCI end with a coaxial (BNC) connection, while the cell connection end terminates in a standard pin-jack that can accept various connectors such as the included alligator clips shown below.

NOTE: The SE (working-sense) and RE (reference) leads terminate in connectors that will accept an additional pin input to make it easier to run three electrode (WE shorted into SE) experiments and two electrode (WE shorted into SE, and CE shorted into RE) experiments.

1.3. Inspecting Your New Instrument

As soon as you receive your new LCI, inspect it for shipping damage. If any damage is noted, immediately notify Princeton Applied Research and file a claim with the carrier. Save the shipping container for possible inspection by the carrier.

WARNING: If your instrument has been damaged, its protective grounding might not work. Do not operate damaged equipment! Tag it to indicate to a potential user that it is unsafe to operate.

1.4. Maintenance, Service, and Support

The LCI has been designed for optimum reliability and requires no periodic maintenance. **There are no user-serviceable parts in this instrument. Breaking the seal by opening the cover will void your warranty!** Contact the factory service department or the affiliate in your area if your unit needs service (see the Warranty in Section 5 for more information).

Remember that our worldwide staff continues to support you after you have purchased your instrument. We provide top quality service, applications support, and a variety of helpful information in the form of application notes, technical notes, and training materials. For more information, visit our website at www.princetonappliedresearch.com.

2. INSTALLATION

This chapter details how to install, calibrate, and test the LCI.

2.1. Installation

2.1.1. Connect Interface Cable to LCI Main Body

Connect the interface cable (section 1.2.1) to the LCI main body (section 1.2.2), and secure the cable to the connector by tightening the screws until snug (do not over tighten!).

2.1.2. Connect Interface Cable to Potentiostat Cell Interface

NOTE: Power off the potentiostat before connecting the LCI. Connecting the LCI with power-on will not allow the potentiostat to recognize the LCI. Only at boot-up (cycling the power on the potentiostat) will the LCI be recognized.

Connect the opposite end of the interface cable to the potentiostat front panel cell cable connector, and secure the cable to the connector by tightening the screws until snug (do not over tighten!). The LCI replaces the standard cell cable supplied with the potentiostat/galvanostat.

NOTE: Once the LCI is connected and power applied to the system, a calibration will be required, and the user will be prompted when the VersaStudio software is launched to perform the calibration prior to use. Please see section 2.2 below for calibration instructions.

2.1.3. Connect Cell Cables to LCI Front Panel

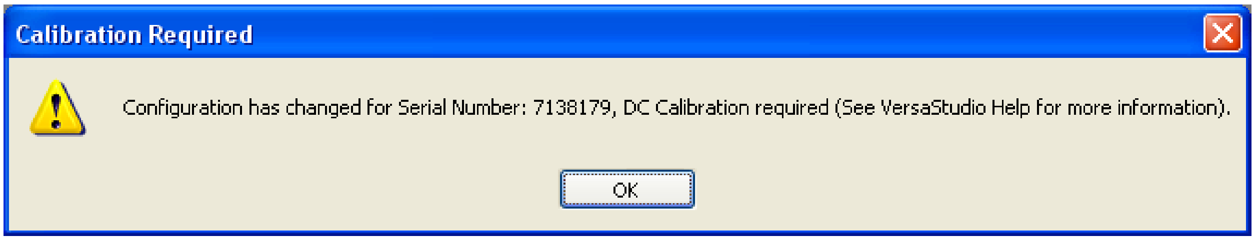
Connect the four cell cables to the cell interface panel. The cables and panel on the LCI are labeled WE (working lead), SE (working-sense lead), RE (reference lead), and CE (counter lead). The terminating colors on the cell leads match those of the standard cell cable (WE=green, SE=gray, RE=white, and CE=red).

2.2. LCI Calibration

The VersaSTAT LC Low Current Interface is designed to provide ultra-low current measurements beyond the capabilities and specifications of the potentiostat to which it is attached. As such, calibration of the potentiostat-LCI combination is required each time it is connected, and it is highly recommended that a calibration be performed prior to each use to provide the best accuracy with each use.

2.2.1 Calibration

The VersaSTAT system was designed to auto-detect a configuration change, so after adding the LCI option and launching the software, the following message will appear immediately upon starting the software:

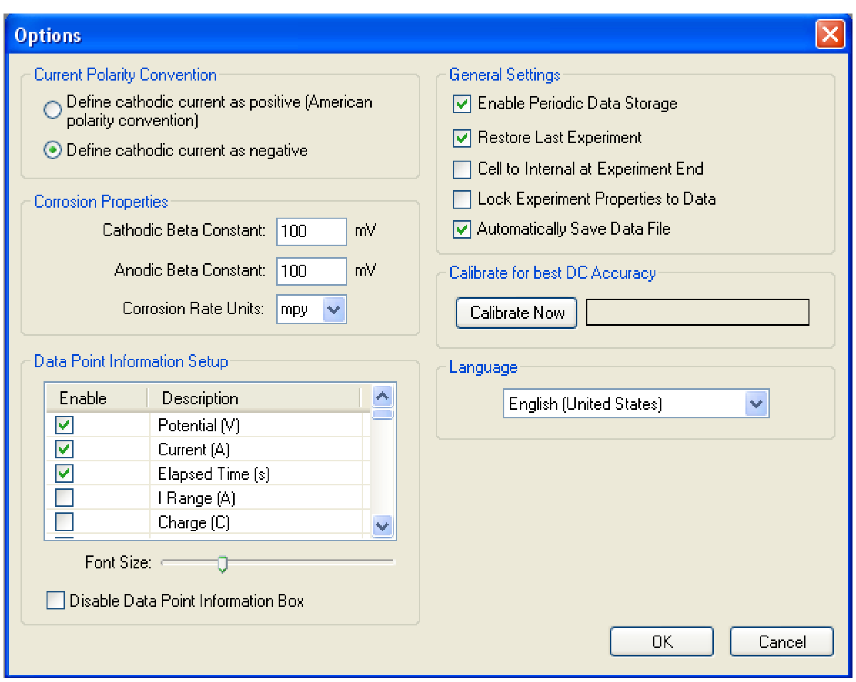


This message will appear after adding the LCI option to a system, as well as after removing the LCI option.

NOTE: It is very important that the calibration be performed in the exact same manner as the experimental settings. The lowest current ranges and current measurements are affected by temperature fluctuations, vibrations, and environmental sources of electromagnetic fields. Always calibrate the system in the same experimental setup as your experiment requires.

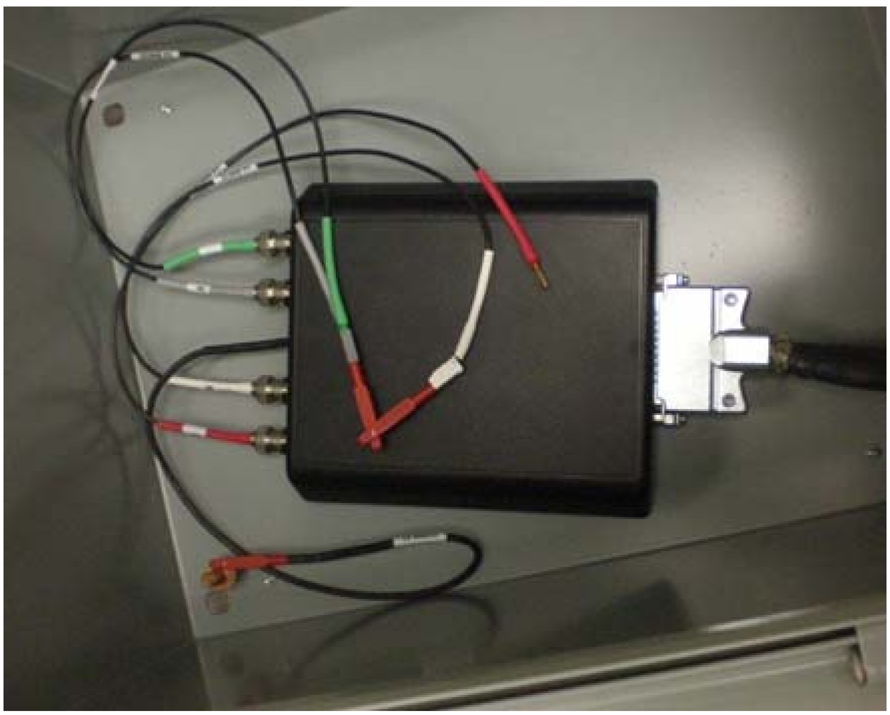
Also, it is very important that the LCI and VersaSTAT be allowed warm up and reach temperature equilibrium. After power-on, wait a minimum of 20 minutes BEFORE performing the calibration.

The calibration procedure is located in the Tools>Options… menu within VersaStudio. Once the LCI is properly connected and configured for the experimental conditions, select the “Calibrate Now” button.

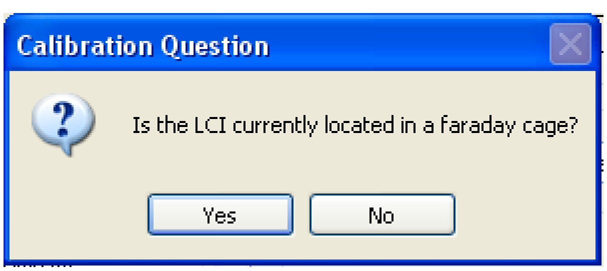


After a few minutes of adjusting some offsets, the following window will appear:

This configuration is shown below where the WE,SE, and RE are connected, but the CE is not. Once connected, select “OK” to continue.

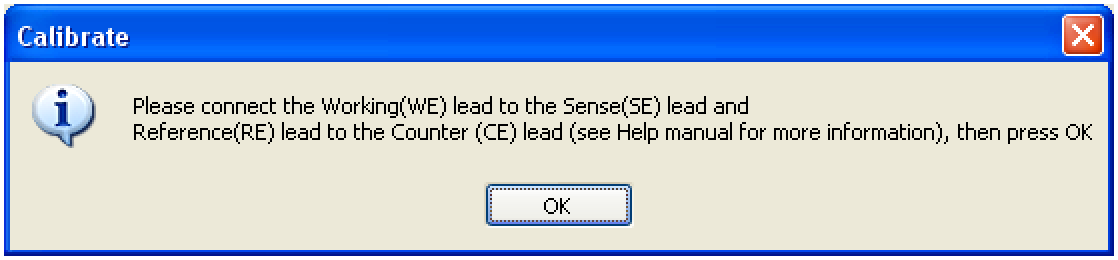


After a few more minutes of adjusting offsets, the following wondow will appear:

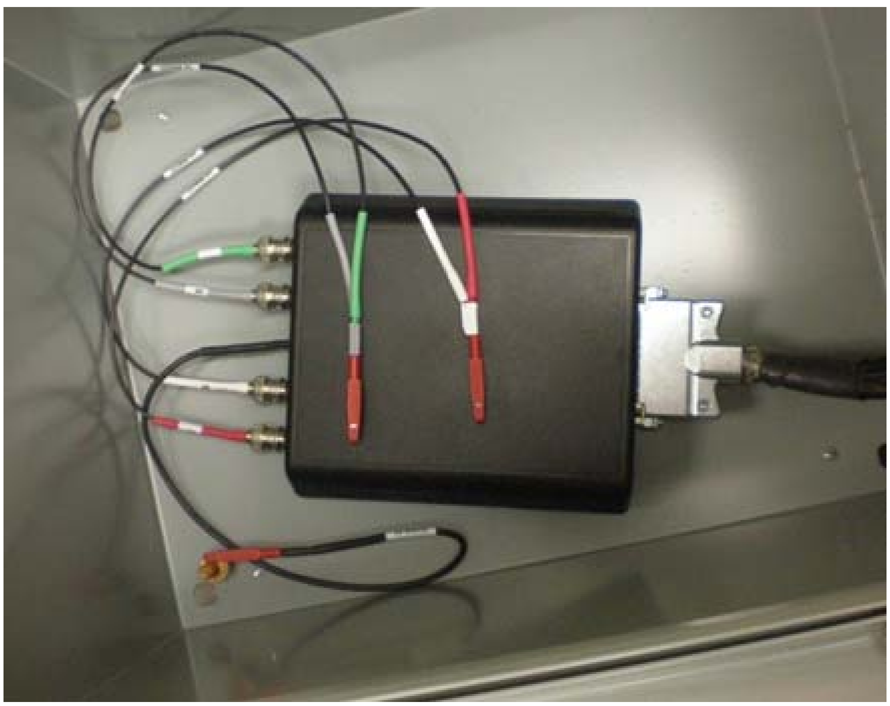


NOTE: As the LCI is inteneded to measure ultra low currents, it is highly recommended that all experiments (and therefore, calibrations) be conducted in a shielded environment, such as a Faraday cage. Any experiments where nanoamps and lower are to be measured, a shielded environment must be used to block the electromagnetic fields that are prevelent in most all laboratory settings.

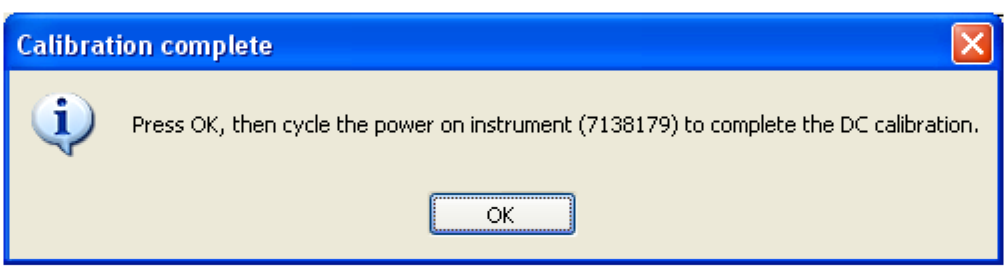
After indicating the presence of a Faraday cage or not, the following window will appear:



The photo below shows these connections. Note that this photo has the LCI inside the Princeton Applied Research Faraday box, part number K0269B (door open on the K0269B for viewing). The ground lead is also connected between the LCI and K0269B to properly ground the Faraday box to the same chassis ground as the LCI and VersaSTAT.



After completing the calibration, the following window will appear:



The calibration will not take effect until the system is re-booted by cycling the power on the potentiostat.

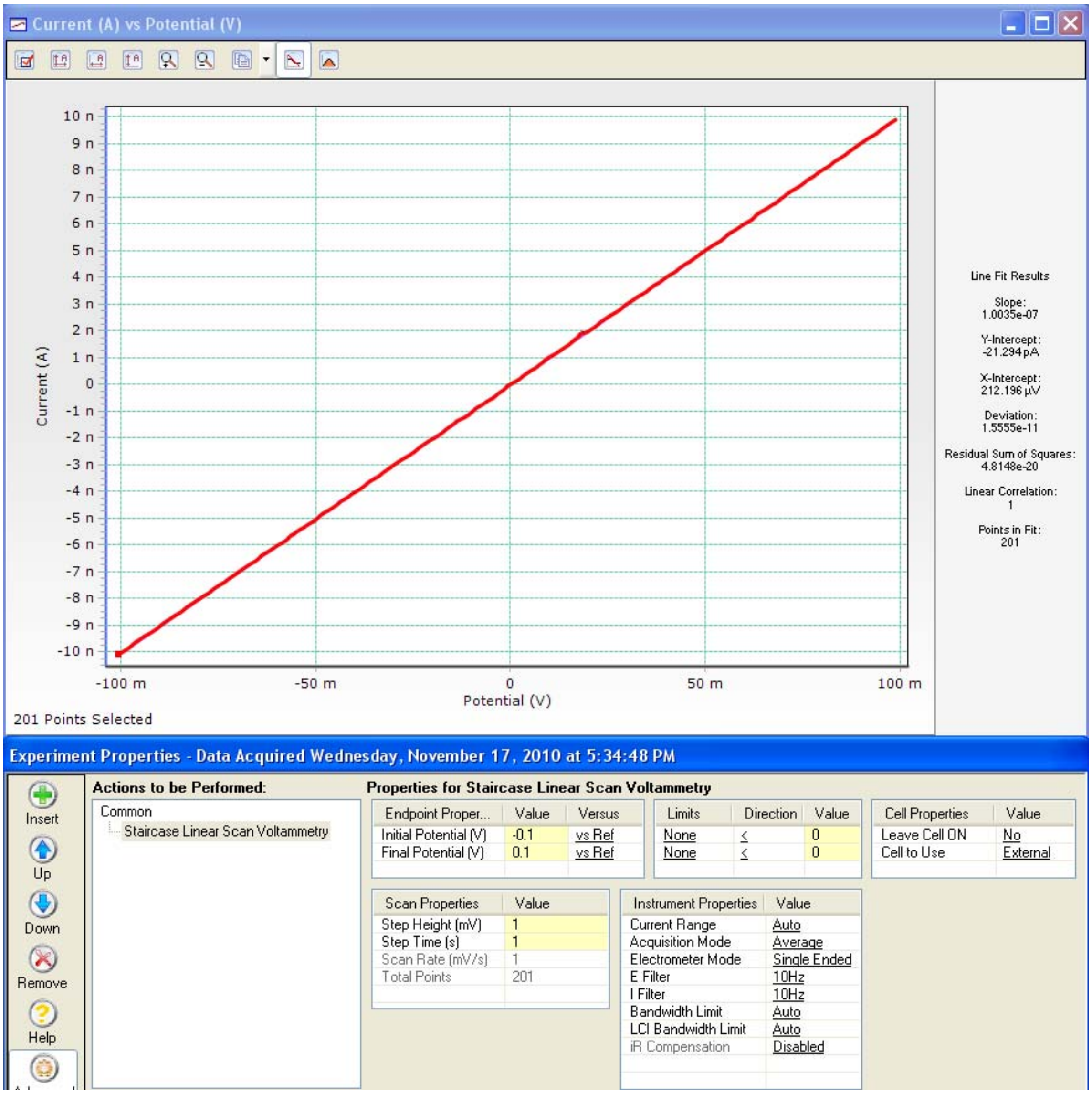
Note: If "Failed Calibration" is reported by the software, double-check connections, let the system stabilize for 30min, then try again.

2.3. LCI Checkout Procedure

As shown in section 1.2.2, the VersaSTAT LC is equipped with a built-in 10Mohm resistor that can be used as a "dummy cell" to perform a limited test of the system for proper operation.

To test the LCI with a dc voltage scan:

1. Connect the cell leads to the built-in 10Mohm dummy cell (The WE cable connects to the W pin jack, the SE cable connects to the S pin jack, the RE cable connects to the R pin jack and the CE cable connects to the C pin jack).
2. Select "New Experiment" in VersaStudio, and choose the Staircase Linear Scan Voltammetry action. Set the Initial Potential = -100mV, Final Potential = +100mV, Step Height=1mV, Step Time=1s, Acquisition Mode=Average, Electrometer=Single Ended, E and I Filters = 10Hz (as shown below). Once setup, select "Run" to acquire the data.
3. Once the data collection has finished, select all of the data points (Data>Select All) and perform a Line Fit by selecting the Line Fit button at the top of the graph.
4. The vertex currents should be +/-10nA, and the slope should be 1.0E-7 (the inverse of which is 10Mohm).


Current (A) vs Potential (V)
10 n
9 n
8 n
7 n
6 n
5 n
4 n
3 n
2 n
1 n
0
-1 n
-2 n
-3 n
-4 n
-5 n
-6 n
-7 n
-8 n
-9 n
-10 n
Current (A)
-100 m
-50 m
0
50 m
100 m
Potential (V)
201 Points Selected
Line Fit Results
Slope: 1.0035e-07
Y-Intercept: -21.294 pA
X-Intercept: 212.196 µV
Deviation: 1.5555e-11
Residual Sum of Squares: 4.8148e-20
Linear Correlation: 1
Points in Fit: 201
Experiment Properties - Data Acquired Wednesday, November 17, 2010 at 5:34:48 PM
Insert
Up
Down
Remove
Help
Actions to be Performed:
Common
Staircase Linear Scan Voltammetry
Properties for Staircase Linear Scan Voltammetry
Endpoint Proper... Value Versus
Initial Potential (V) -0.1 vs Ref
Final Potential (V) 0.1 vs Ref
Limits Direction Value
None ≤ 0
None ≤ 0
Cell Properties Value
Leave Cell ON No
Cell to Use External
Scan Properties Value
Step Height (mV) 1
Step Time (s) 1
Scan Rate (mV/s) 1
Total Points 201
Instrument Properties Value
Current Range Auto
Acquisition Mode Average
Electrometer Mode Single Ended
E Filter 10Hz
I Filter 10Hz
Bandwidth Limit Auto
LCI Bandwidth Limit Auto
iR Compensation Disabled

3. MEASUREMENT OF SMALL CURRENT SIGNALS

This chapter describes the issues surrounding the measurement of small current signals to include proper shielding and cell design recommendations.

3.1. Faraday Shield

The LCI and corresponding potentiostat system operate near the limits of current measurement. The LCI system can resolve current in the attoamp (10^{-18}) range. For perspective, 100aA represents the flow of around 600 electrons per second!

A Faraday shield for low current measurements is mandatory. A Faraday shield is a conductive enclosure that surrounds the cell. The materials can vary from a metal box or fine wire mesh, to a plastic case covered in conductive paint. An effective Faraday shield can be as simple and inexpensive as a cardboard box completely covered in aluminum foil (although copper tape is preferred). Regardless, the conductive coating should be continuous on all sides of the cell (including the bottom), and all parts operate as a single, conductive unit.

For the LCI, the best shielding will occur if the LCI main body and the cell leads are all placed inside the Faraday shield, as the **shield must be connected to the LCI ground**. Note the photos below of the Princeton Applied Research K0269B Faraday box. The box is made of steel with few and small openings for cell cables or other leads. The box can be used in a vertical or horizontal position. In the photo to the right, the LCI Main body has been attached to the wall of the K0269B using Velcro tape (not supplied). Also note in the upper left corner above the cell is a grounding point to connect the ground lead from the LCI to the Faraday shield (not connected in photo). Note: The ring-stand and cell are not included with the K0269B Faraday box.

3.2. Avoiding External Noise Sources

Always try to keep any electrochemical cell away from noise sources, regardless of current levels, but especially for low current measurements. Some common sources of noise are:

- Electrical outlets and power strips
- Computers and their monitors
- Electrical motors
- Heating mantles or other heating/chilling equipment
- Magnetic stirring devices
- Fluorescent lights

Always place the cell as far from these devices as possible, and never place any of them inside the Faraday shield.

3.3. Cell Design

3.3.1. Lead and Electrode Placement

The cell leads must not be moved during an experiment which is measuring small currents. Depending on how sensitive the measurement, this includes eliminating even the smallest vibrations caused by the cooling fan inside the potentiostat. Both microphonic and triboelectric effects can create false results when the cable is moved during a measurement.

3.3.2. Cell Construction

Always insure that your cell material and electrode materials are appropriate for the intended measurements. A cell with impedance between the electrodes of 10^{10} cannot be used to make measurements between electrodes at impedances of 10^{12}.

3.3.3. Reference Electrode

One of the most common problems with "cell noise" in electrochemical experiments in general is high impedance reference electrodes and assemblies. High impedance reference electrodes can cause potentiostat instability and excessive voltage noise generation. Always try to avoid:

Double junction Lugins (or bridge tubes)
Poorly conductive solutions inside Lugin

3.4. Experimental Properties

3.4.1. Auto Current Ranging

Although the VersaSTAT LC allows full auto-current ranging over the entire range from 20mA range down to 4pA range, this can lead to some data points that are suspect due to the long time constant associated with the lower current ranges. Any time the hardware "changes" during a data point, the VersaStudio software flags that data point with "Code 2" in the Data View window under the Comments section. As an example, the time constant associated with the 20pA current range is 7s, so if a range change occurred to go from the 200pA to the 20pA current range, it would take around 7s for that range to measure the current accurately and allow the I/E converter to adjust properly. If the experiment was collecting data at a rate of 1 point/second, there would be 6 points (provided the experiment collect more than 6 points on the new, lower 20pA range) that would be suspect and labeled as "Code 2" in the Data View. Below is a table of time constants associated with each range where the TC>500ms.

Current Range	Time Constant
20nA	900ms
2nA	2.1s
200pA	5s
20pA	7s
4pA	7s

3.4.2. LCI Bandwidth Limits

All potentiostats can become unstable and oscillate when connected to capacitive cells, and unfortunately most electrochemical cells are capacitive. Although designed to minimize the onset of these oscillations, they can still occur in some experimental conditions, and are most often manifested as excessive noise or "jumps" in the data and graphical output.

If an oscillation is suspected, one can use the LCI Bandwidth Limits in the VersaStudio software to try and eliminate the oscillation. The "limits" are a range of damping filters (Normal, Slow, and Very Slow) within the control loop of the VersaSTAT LC Low Current Interface that can help prevent oscillations when performing experiments on capacitive cells. There is no recommended setting other than Auto (which is "Normal"), and if oscillations occur, try stepping to "Slow" first, then "Very Slow" as a last resort.

4. SPECIFICATIONS

4.1. Electronic Specifications

With the VersaSTAT LC option connected to the appropriate potentiostat, the following specifications are in order. All other specifications not listed default to the connected potentiostat.

4.1.1 System Performance

- **Minimum Current Range** 4pA (4×10^{-12} A)
- **Minimum Current Resolution** 122 aA (122×10^{-18} A)

4.1.2. Power Amplifier

Maximum Current: ± 200mA

4.1.3. Differential Electrometer

Input Bias Current < 200 fA at 25°C
Maximum Voltage Range ± 10 V maximum
Input Voltage Differential ± 10 V
Bandwidth 700kHz (-3 dB)
Common Mode Rejection >60dB @ 100Hz, >50dB @100kHz
Input Impedance > 10^{14} Ω in parallel with < 200 fF, typical

4.1.4. Current Measurement

Ranges 12 decades, 200mA to 4pA
Accuracy (dc)

- 2 µA to 200 mA < 0.2% full scale
- 20 nA and 200nA ranges < 0.5% full scale
- 200pA – 4pA ranges < 1.0% full scale ± 500fA full scale

4.1.5. Current Control

Applied Current Range ± full scale per range
Applied Current Resolution ± 1/32,000 x full scale
Applied Current Accuracy ± 0.5% of range , ± 0.5% of reading
Maximum Current Range/Resolution ± 200mA /10 uA
Minimum Current Range/Resolution ± 4 pA / 122aA

4.2. Physical Specifications

Weight 500g (1.1 lbs), not including cable.

Dimensions 14.6 cm W x 19.1 cm D x 4.54 cm H (5.75 in x 7.5 in x 1.75 in)

4.3. Standard Environmental Conditions

This equipment is designed to meet or exceed the requirements of the following standards:

- LVD: EN61010-1:1993, Amendment 2
- EMC: EN61326: 1998 Emissions
- EN55011:1991, Group 1, Class A

Immunity:

- IEC 61000-4-2:1995, ESD
- IEC 61000-4-3:1995, EM field
- IEC 61000-4-4:1995, Burst
- IEC 61000-4-5:1995, Surge
- IEC 61000-4-6:1995, Conducted RF

Operating Temperature: 10^{o} C to 50^{o} C
Humidity: maximum 80% non-condensing
Protection against ingress of water, IPX0.

5. Advanced Measurement Technology, Inc.

a/k/a Princeton Applied Research, a subsidiary of AMETEK®, Inc.

WARRANTY

Princeton Applied Research* warrants each instrument of its own manufacture to be free of defects in material and workmanship. Obligations under this Warranty shall be limited to replacing, repairing or giving credit for the purchase price, at our option, of any instrument returned, shipment prepaid, to our Service Department for that purpose within ONE year of delivery to the original purchaser, provided prior authorization for such return has been given by an authorized representative of Princeton Applied Research.

This Warranty shall not apply to any instrument, which our inspection shall disclose to our satisfaction, to have become defective or unworkable due to abuse, mishandling, misuse, accident, alteration, negligence, improper installation, or other causes beyond our control. This Warranty shall not apply to any instrument or component not manufactured by Princeton Applied Research. When products manufactured by others are included in Princeton Applied Research equipment, the original manufacturer's warranty is extended to Princeton Applied Research customers.

Princeton Applied Research reserves the right to make changes in design at any time without incurring any obligation to install same on units previously purchased.

THERE ARE NO WARRANTIES THAT EXTEND BEYOND THE DESCRIPTION ON THE FACE HEREOF. THIS WARRANTY IS IN LIEU OF, AND EXCLUDES ANY AND ALL OTHER WARRANTIES OR REPRESENTATIONS, EXPRESSED, IMPLIED OR STATUTORY, INCLUDING MERCHANTABILITY AND FITNESS, AS WELL AS ANY AND ALL OTHER OBLIGATIONS OR LIABILITIES OF PRINCETON APPLIED RESEARCH, INCLUDING, BUT NOT LIMITED TO, SPECIAL OR CONSEQUENTIAL DAMAGES. NO PERSON, FIRM OR CORPORATION IS AUTHORIZED TO ASSUME FOR PRINCETON APPLIED RESEARCH ANY ADDITIONAL OBLIGATION OR LIABILITY NOT EXPRESSLY PROVIDED FOR HEREIN EXCEPT IN WRITING DULY EXECUTED BY AN OFFICER OF PRINCETON APPLIED RESEARCH.

SHOULD YOUR EQUIPMENT REQUIRE SERVICE

A. Contact the Customer Service Department (865-482-4411) or your local representative to discuss the problem. In many cases it will be possible to expedite servicing by localizing the problem.

B. If it is necessary to send any equipment back for service, we need the following information.

1. Model number and serial number.
2. Your name (instrument user).
3. Your address.
4. Address to which the instrument should be returned.
5. Your telephone number and extension.
6. Symptoms (in detail, including control settings).
7. Your purchase order number for repair charges (does not apply to repairs in warranty).
8. Shipping instructions (if you wish to authorize shipment by any method other than normal surface transportation).

C. U.S. CUSTOMERS — Ship the equipment being returned to:

Advanced Measurement Technology, Inc. PHONE: 865-482-4411
801 S. Illinois Avenue FAX: 865-483-2133
Oak Ridge, TN 37831
ATTN: Customer Service

D. CUSTOMERS OUTSIDE OF U.S.A. — To avoid delay in customs clearance of equipment being returned, please contact the factory or the nearest factory distributor for complete shipping information.