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LC-3C/D/E “Petit Ampère”

February, 2006

MF-9029 INSTRUCTION MANUAL

Battery-Powered Potentiostat
for LCEC and Amperometric Biosensors

Bioanalytical
Systems, Inc

2701 Kent Avenue
West Lafayette
Indiana 47906

www.bioanalytical.com

MANUFACTURER'S NOTE

This instrument, either wholly or in part, is manufactured for research purposes only.
Use for medical diagnosis is not intended, implied, or recommended by the manufacturer.
Use for this purpose and accountability for the same rests entirely with the user.

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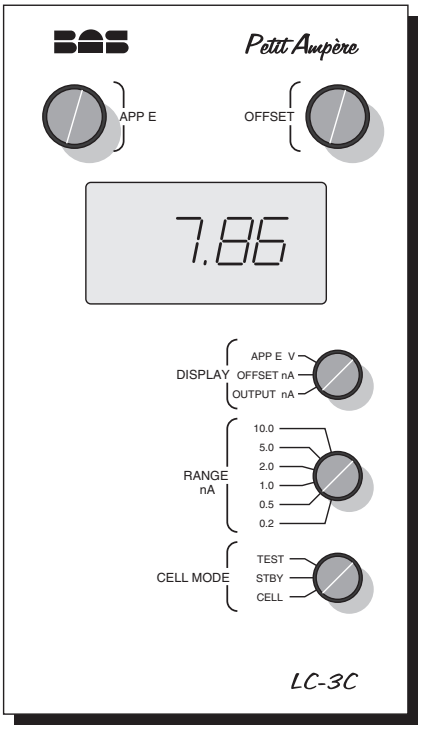
Section 1. Preface

This manual covers operation and service information for the BASi Petit Ampère Amperometric Controllers (LC-3C/D/E).

These electrochemical instruments were specifically developed for liquid chromatography/ electrochemistry (LCEC) and amperometric sensors such as those based on oxidase enzymes. The instruments are designed so that they can be used in parallel, with a single reference and auxiliary electrode, to form bipotentiostats, tripotentiostats, etc. They also can provide an additional channel for an existing LC-4C electrochemical detector.

The LC-3C/D/E was named in honor of the French physicist André Marie Ampère (1775–1836); we refer to it as the Petit Ampère (see Figure 1.1). It is currently available in three models, differing only in the current gains available (the Model LC-3C covers from 0.2 to 10 nA/V, the Model LC-3D extends from 5 to 200 nA/V, and the Model LC-3E ranges from 5 to 200 µA/V). Customized units are also available.

Figure 1.1. The Petit Ampère, Model LC-3C.



The Petit Ampère is designed to be used as part of a bipotentiostat, tripotentiostat, etc. for independent potential control of multiple working electrodes (see Figure 1.2). In this manner, it can also be used in conjunction with an LC-4C amperometric controller to provide additional channels (see Figure 1.3). Those acquainted with other BASi amperometric controllers will find the Petit Ampère to be very familiar and user-friendly.

Figure 1.2. Additional units can be slaved to a Petit Ampère to create any number of channels with independent potential control. Use cable EW-8116 for each additional channel.

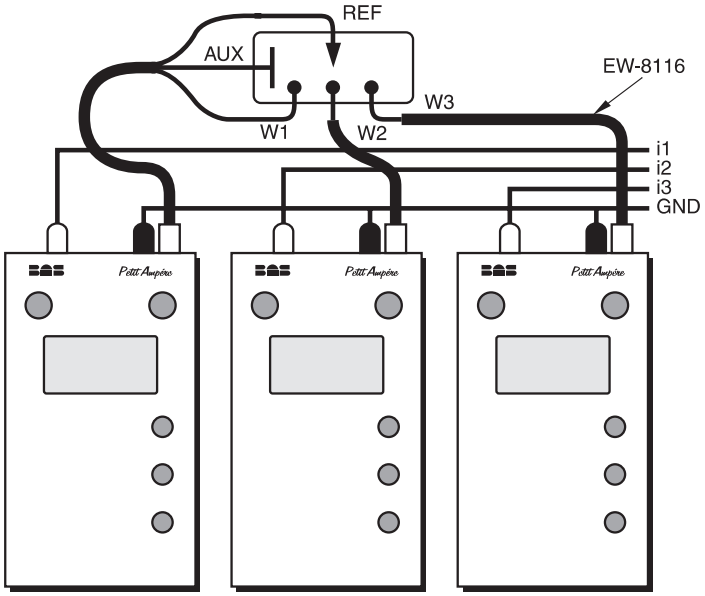
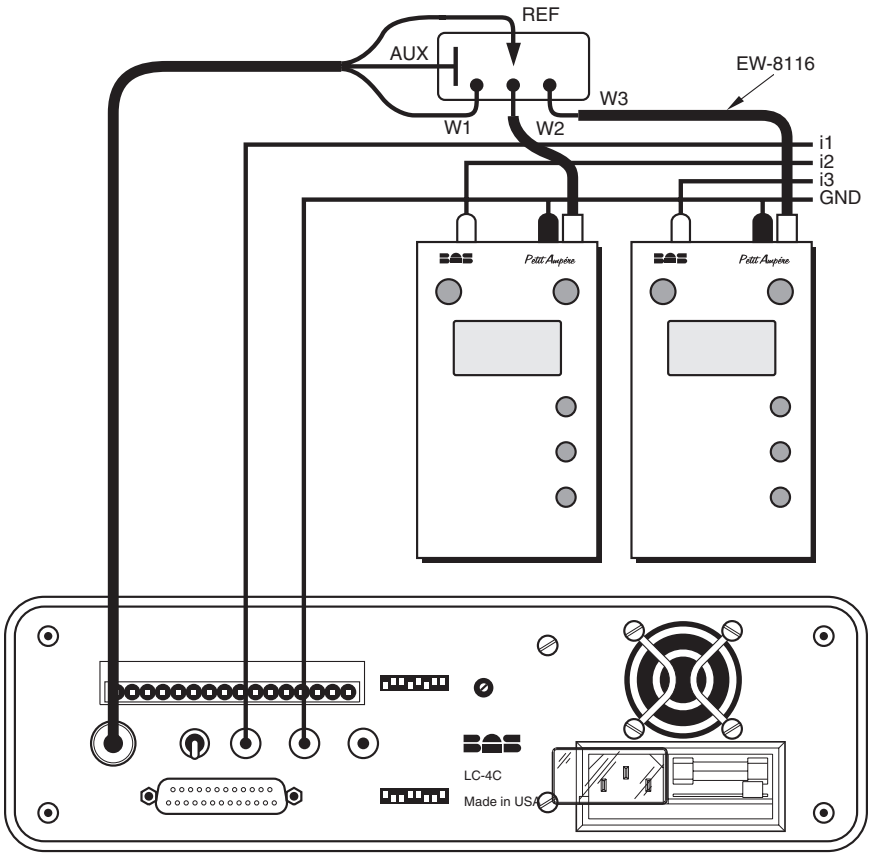


Figure 1.3. The Petit Ampère can be used in conjunction with an LC-4C amperometric controller. Use cable EW-8116 for each additional channel.



Section 2. Support Policy

2.1 User Updates

To activate your warranty and to receive product update information news and valuable information related to this and other BASi products, fill out and return the Warranty Enrollment Card that was shipped with the instrument.

2.2 Damaged Shipments

Breakage of any part of this instrument during shipping should be reported immediately to BASi Customer Service. It will be necessary to retain the original packing box and contents for inspection by the freight handler. BASi will replace any new instrument damaged in shipping with an identical product as soon as possible after the claim filing date. Claims not filed within 30 days of the shipping date will be invalid.

Do not return damaged goods to BASi without first contacting Customer Service for a Return Authorization Number (RA#). When a defective part is returned to BASi, the RA# immediately identifies you as the sender and describes the item being returned. BASi refuses all unauthorized return shipments.

2.3 Product Warranty

Bioanalytical Systems, Inc. (BASi) products are fully warranted against defects in material and workmanship. The LC-3C/D/E electronic controller is unconditionally warranted for one year from date of shipment, except when failure is due to obvious abuse or neglect, unauthorized tampering, procedures not described in manuals, or improper connection of electronic units to other components. Electrochemical cells are warranted for 60 days from date of shipment under the same exclusions. Chromatographic columns and injection valves are warranted for 30 days. The following consumable items are not covered under any warranty: carbon paste, activated aluminum oxide, lamps, panel lights, fuses, pump seals, valve seals, reference electrodes, membranes.

For any product expressly covered under this warranty, BASi is liable only to the extent of replacement of defective items. BASi shall not be liable for any personal injury, property damage, or consequential damages of any kind whatsoever. The foregoing warranty is in lieu of all other warranties of merchantability and fitness for a particular purpose.

2.4 Service Information

BASi provides a skilled service staff to solve your technical equipment problems. For further details, call customer service personnel (1-800-845-4246) who will route your problem to the correct individual. Following discussion of your specific difficulties, an appropriate course of action will be described and the problem resolved accordingly. Do not return any products for service until a Return Authorization Number (RA#) has been obtained. The RA# identifies you as the sender and describes your problem in full detail. Turnaround time on service can be quoted to you at the time your RA# is issued, although we cannot determine the actual amount of service required until we have received your unit and diagnosed the problem. All correspondence and shipments should be sent to:

RA# [insert your assigned number], Service Department
Bioanalytical Systems, Inc.
2701 Kent Avenue
West Lafayette, IN 47906

Section 3. Installation

3.1 Inspection of Your Shipment

After carefully unpacking the instrument, check the contents of the packages and inspect for breakage. If you ordered them, electrodes and flow cell components are packaged in a separate box. Assembly of these various parts is outlined in other manuals.

BASi publishes a variety of manuals and application notes. If you don't have the information you need for your application, don't hesitate to contact the factory (1-800-845-4246, FAX: 765-497-1102) or your local representative for assistance.

If there are any discrepancies, retain the packing slip and contact BASi Customer Service.

Please retain the shipping box and packing material until you have fully tested the unit to be certain that no damage was incurred during shipping.

3.2 Local Environment

Amperometric detection is a highly sensitive technique. The currents measured typically fall in the picoampere or nanoampere range. Hence, smooth operation can easily be influenced by electrical disturbances in the environment. Also, since detection is due to a chemical reaction, its response (and baseline drift) is temperature-dependent; this is very noticeable at high detector gain.

When selecting a location for the detector, follow these guidelines:

1. Connect the entire system (pump, detector, recorder, etc.) to the same grounded power line. This ensures adequate ground to all components of the system and eliminates the possibility of a ground loop, which could produce baseline noise. Also, select a power line that is lightly used. Other laboratory instruments, such as ovens, vortex mixers, centrifuges, and large motors may cause spikes in the power line.

2. Locate the instrument on a stable bench. Vibrations can hamper the performance of any sensitive instrument.

3. Select a room where the temperature remains stable throughout the day. Avoid installing the instrument near drafty areas, windows, air ducts, ovens, or refrigerators.

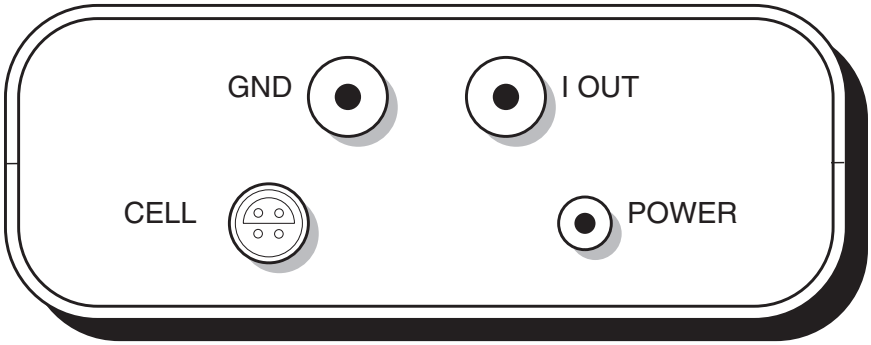
4. Place the instrument away from busy, congested areas. Remote, isolated areas are best for high-sensitivity work.

5. Avoid very dry areas and areas that are carpeted. Static electricity can affect instrument performance. Anti-static floormats and benchmats are useful if spiking caused by static charge is a problem.

6. Avoid areas where radio frequency interferences are likely. Beeper-type paging devices can be a problem in some installations.

3.3 LC-3C/D/E Connections

Figure 3.1. Connections to the LC-3C.



IOUT/GND

The IOUT/GND banana jacks (see Figure 3.1) feed the analog signal from the LC-3C/D/E to a recording device. The IOUT jack provides a signal of 1 volt for a full scale deflection in any range. The IOUT jack will provide up to a □5 V signal, thereby providing a five-times overrange capability at the output jacks.

The I OUT output drive capability from the LC-3X is limited to keep battery life acceptable when used without the AC adapter. This is fine when driving high input impedance strip chart recorders, but the output may need to be buffered when driving unbuffered A/Ds. Connection to unbuffered A/Ds can cause the output to oscillate, thus giving unreliable measurements.

NOTE: The LC-3C/D/E power supply is a floating supply. This means that it is not referenced to Earth ground. For quiet, stable operation, *THE GND CONNECTION MUST BE TIED TO EARTH GROUND AT ONE POINT.* Most strip charts, integrators, and data acquisition boards provide differential inputs (where the "–" input is not tied to Earth ground) and so will require a jumper from their negative terminals to Earth ground for proper operation. Simply connect a small wire between "–" and "GND" to accomplish this.

When connecting controllers for use with multiple working electrodes in the same cell, connect all ground lines together (see Figure 1.2).

CELL

The cell lead cable is attached to the CELL connector (see Figure 3.1). To make the connection, insert the cell lead cable connector into this socket until the two components snap into place. Do not force this connection; make certain the two components are properly aligned before making the connection.

CELL LEAD CONNECTION

The cell lead with LC-3C/D ends in connectors for the BASi thin-layer flow cell. The LC-3E lead ends in alligator clips. All leads are color coded with Black to the working electrode, White to the reference electrode, and Red to the auxiliary electrode. If one wants to work with a two-electrode system, the black is connected to the working electrode and both the red and white leads must be connected to the counter electrode.

NOTE: When using multiple detectors, the W1 (master) cell lead will be gray and have reference (white), auxiliary (red), working (black), and shield connections (EW-8112); all other (W2, W3, etc.) detectors (slaves) will have only the working (black) leads (EW-8116).

POWER

The plug of the wall-mount power supply is attached to the POWER connector (see Figure 3.1). The wall-mount power supply provides 12 V DC unregulated at 10 mA to the LC-3C/D/E. The power supply plug is a 5.5-mm outside, 2.1-mm inside plug. The outside is ground and the inside is positive.

NOTE: The LC-3C/D/E will run off any 10 V to 16 V unregulated power supply, such as a 12 V lantern battery or a 13.8 V automotive power source. If a different, unregulated power supply is to be used, make sure to measure its no-load voltage. Most 9 V DC unregulated power supplies provide around 12 V DC at no-load (or at the 5 mA to 10 mA load of the LC-3C/D/E). Most 12 V DC unregulated power supplies may be 18 V to 20 V DC at no-load, which is too high for the LC-3C/D/E.

CAUTION!! NEVER operate the LC-3C/D/E without the internal NiCad battery installed. See page 14.

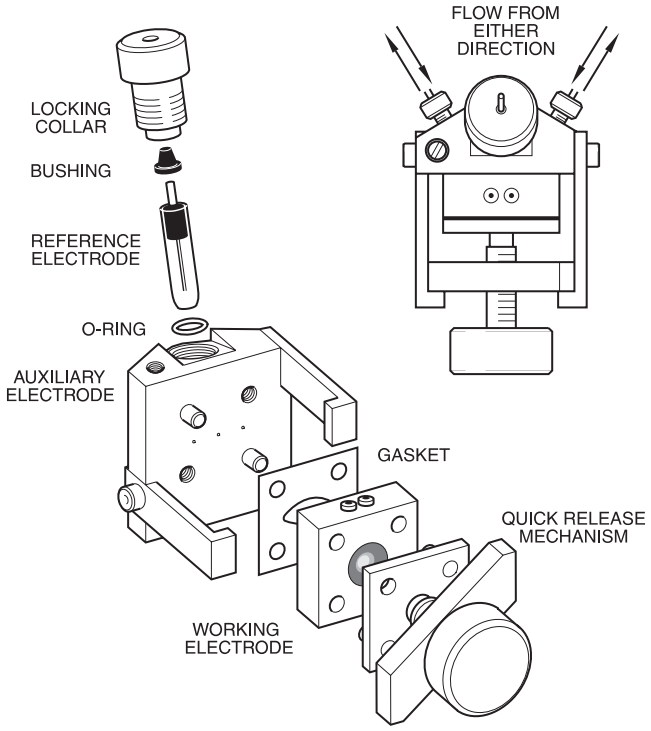
3.4 Electrochemical Flowcell

The electrochemical flowcell is typically arranged first in line following the column. Several different flowcells are available, so only a general description of detector connection can be presented here. Connect the flowcell after the column has been equilibrated with mobile phase.

Installation is as follows:

1. Attach the working electrode and gasket to the auxiliary electrode, using the clamp and backing plate as shown in Figure 3.2. Tighten securely.

Figure 3.2 Cross-flow thin-layer amperometric cell design.



2. Connect the column outlet to the detector inlet tube. A reusable plastic fitting is provided. *Avoid using metal ferrules because they become permanently attached.*

   Pull the plastic fitting back so that at least 5 mm of steel tubing is exposed at the tip. Insert the tubing into the column as far as it will go. Hold the tube in this position and finger-tighten the plastic fitting. Do the same at the detector end of the tubing.

3. Start the pump. The reference electrode well in the auxiliary electrode will fill up. Siphon off this fluid several times, until there are no bubbles visible in the well.

4. Stop the pump. Insert the reference electrode o-ring into the well, and push it down so it sits flush on the ledge inside the well.

5. Siphon off excess fluid to the level of the o-ring.

6. Carefully insert the reference electrode through the o-ring, so no bubbles are trapped under it.

7. Dry the area on top of the o-ring, and any fluid that squirted out behind the auxiliary electrode. Place the bushing on top of the reference electrode, and screw down the locking collar, finger tight.

8. Route the detector outlet either to a second detector (if used) or to waste. Then start the pump and check for leaks.

9. Connect the black lead labeled “W1” to the electrode. Connect the white female lead to the reference electrode and the red female lead to the auxiliary electrode. If you are connecting more than one Petite Ampère, see Figure 1.2 for details.

Section 4. Specifications and Controls

This section describes the functions of each control knob on the LC-3C/D/E. Refer to Figures 4.1 and 4.2 for their locations.

Figure 4.1. Top panel of LC-3C.

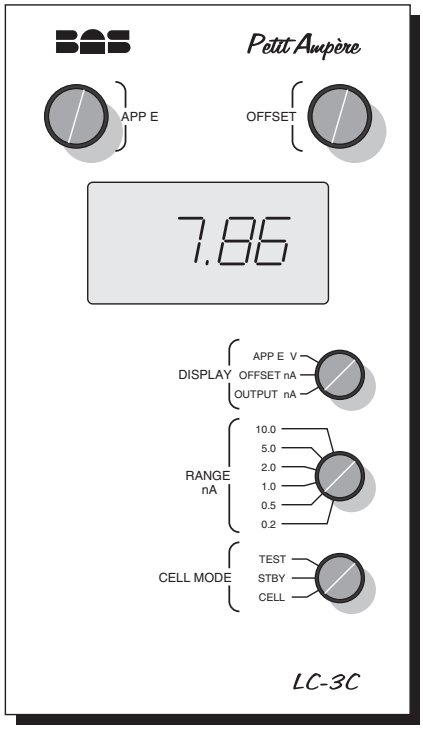
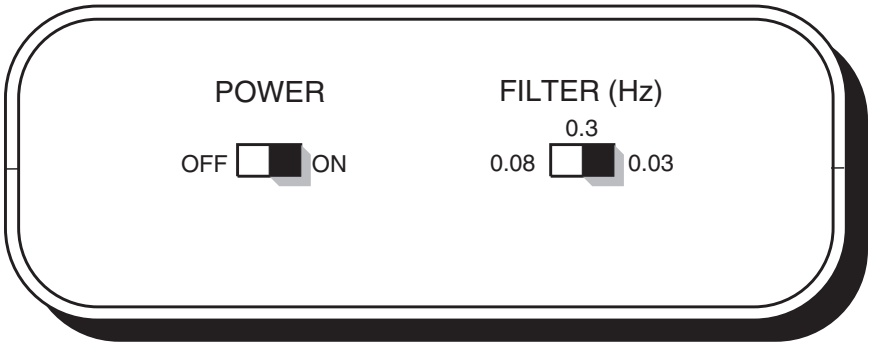


Figure 4.2. End panel of LC-3C.



DISPLAY

This switch selects the function to be monitored by the internal 3½-digit LCD voltmeter. The selections are:

APP E V displays the applied potential in volts.

+1.500 V to –1.500 V

OFFSET nA displays the offset current in nA or µA.

LC-3C: +19.99 nA to –19.99 nA

LC-3D: +199.9 nA to –199.9 nA

LC-3E: +199.9 µA to –199.9 µA

OUTPUT nA/µA displays the output current in nA or µA.

LC-3C: +1.999 nA to –1.999 nA for the 0.2, 0.5, 1.0 nA ranges; +19.99 nA to –19.99 nA for the 2.0, 5.0, 10 nA ranges

LC-3D: +19.99 nA to –19.99 nA for the 5, 10, 20 nA ranges; +199.9 nA to –199.9 nA for the 50, 100, 200 nA ranges

LC-3E: +19.99 µA to –19.99 µA for the 5, 10, 20 µA ranges; +199.9 µA to –199.9 µA for the 50, 100, 200 µA ranges

RANGE nA/µA

This switch selects one of six possible output (gain) ranges in nanoamps (or microamps) per volt. For example, a setting of 10 nA with a cell current of 5 nA would produce an output of 0.5 V. The output is capable of swinging +5 V to –5 V, which provides a five-times overrange capability at the output jacks.

LC-3C: 0.2, 0.5, 1.0, 2, 5, 10 nA/V

NOTE: If the current exceeds 50 nA on the 0.2, 0.5, or 1.0 nA ranges, or if the current exceeds 500 nA on the 2.0, 5, or 10 nA ranges, potential control may be lost.

LC-3D: 5, 10, 20, 50, 100, 200 nA/V

NOTE: If the current exceeds 500 nA on the 5, 10, or 20 nA ranges, or if the current exceeds 5 µA on the 50, 100, or 200 nA ranges, potential control may be lost.

LC-3E: 5, 10, 20, 50, 100, 200 µA/V

NOTE: If the current exceeds 500 µA on the 5, 10, or 20 µA ranges, or if the current exceeds 5 mA on the 50, 100, or 200 µA ranges, potential control may be lost.

CELL MODE

This switch controls the application of potential to the cell. In the STBY mode, the working, reference, and auxiliary are disconnected from the cell. In the CELL mode, all electrodes are connected and the selected potential is applied. The TEST mode is used for trouble-shooting the controller for electronic problems and connects to an internal dummy cell. The LC-3C dummy cell (100 M ohm) provides 10 nA/V applied E, the LC-3D dummy cell (10 M ohm) provides 100 nA/V applied E, and the LC-3E dummy cell (10 K ohm) provides 100 µA/V applied E.

NOTE: The CELL MODE should always be set to STBY before handling the cell lead connections, as static that has accumulated may discharge into the cell lead connections, damaging your unit.

APP E

The APP E knob adjusts the applied potential. This is a 10-turn adjustment with –1.5 V at full counterclockwise, and +1.5 V at full clockwise. The DISPLAY switch must be set to APP E V when the APP E adjustment is made.

OFFSET

The OFFSET knob provides a 10-turn adjustment of offset current, to zero output for a steady-state background current.

LC-3C: The offset range is –19.99 nA at full counterclockwise to +19.99 nA at full clockwise.

LC-3D: The offset range is –199.9 nA at full counterclockwise to +199.9 nA at full clockwise.

LC-3E: The offset range is –199.9 µA at full counterclockwise to +199.9 µA at full clockwise.

POWER

The POWER switch (see Figure 4.2) turns the electronics OFF and ON. The modular supply is powered as long as it is plugged in. The CELL MODE switch should be placed in STBY before the POWER switch is turned to ON. If the LC-3C/D/E electronics are turned off, the battery will fully charge in 12 hours; if the unit is on, it will take 24 hours for a full charge. With a full charge on the battery, the unit will run 20–24 hours. The battery is a 7.2 V 100 mA/hr NiCad in a standard 9 V battery form. This type of battery is readily available from retail merchants and hardware stores, or BASi (P/N ER-9701).

WARNING: VERY IMPORTANT! Use only NiCad or NiMetal Hydride batteries! Use of other batteries (alkaline or Ph acid) may cause acid leakage or explosion, and will definitely void the warranty. Please dispose of old batteries in an environmentally responsible manner.

The unit must NEVER be operated without the battery in place, or damage to the instrument *will* occur. The battery needs to be replaced every year even if the unit is always run from AC power.

FILTER (Hz)

The FILTER switch (see Figure 4.2) controls the filtering of the output. There are three settings: 0.03, 0.08, and 0.3 Hz. These settings correspond to the 3 dB point of the frequency roll-off. The 0.03 Hz is the most heavily filtered output and corresponds approximately to a five-second RC time constant, or an 11-second rise time. The 0.08 Hz setting is a medium filtered output and corresponds approximately to a two-second RC time constant, or a 4.4-second rise time. The 0.3 Hz setting is the least filtered output and corresponds approximately to a 0.5-second RC time constant, or a 1.1-second rise time. Most LCEC peaks are 10 to 30 seconds wide and a setting of 0.08 Hz is ideal.

Section 5. Operation

This section describes the operation of the LC-3C/D/E. Refer to Figures 5.1 and 5.2 for control locations.

Figure 5.1. Top panel of LC-3C.

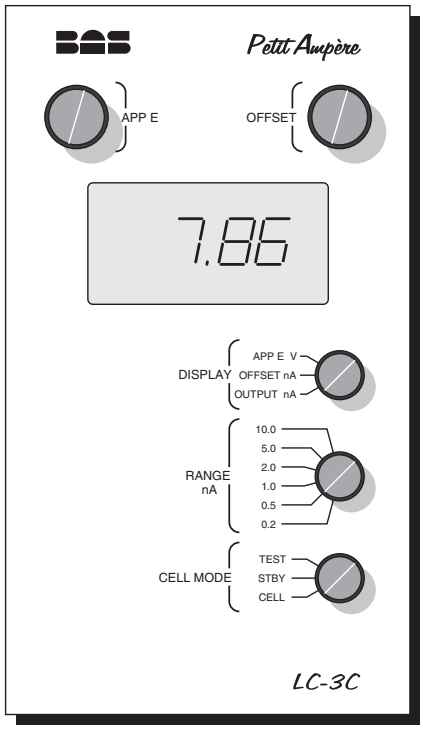
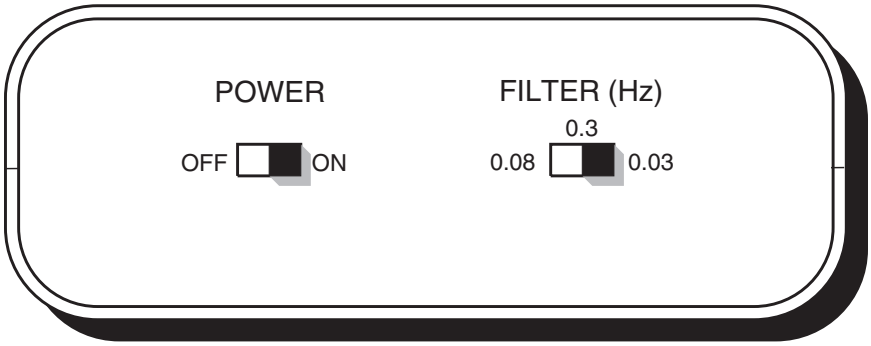


Figure 5.2. End panel of LC-3C.



1. Make sure that the chromatograph is hooked up and running properly. There should be no bubbles in the flowcell, and all connections are as previously described.

2. Zero the chart recorder or integrator with its internal zero control.

3. Make sure that the CELL MODE switch is in the STBY position (Figure 5.1).

4. Turn the POWER switch on (Figure 5.2).

5. Set the desired potential. Turn the DISPLAY knob to the APP E V position, then turn the APP E adjusting knob until the correct potential is displayed.

6. Set the appropriate filter (Figure 5.2). For most chromatography, we recommend the 0.08 Hz setting.

7. Set the RANGE control (Figure 5.1) to its least sensitive (highest) setting.

8. Set the OFFSET (Figure 5.1) to a near-zero reading by changing the DISPLAY knob to OFFSET and turning the OFFSET adjusting knob. Then turn the DISPLAY knob to the OUTPUT position.

9. Turn the CELL MODE switch to the CELL position. This applies potential to (turns on) the working electrode.

10. When the electrode is first turned on, you should see a large current signal. This is the charging and transient background current, and will gradually decay to a steady-state background. For sensitive analyses, this may take from several hours to overnight.

11. When the background stabilizes, turn the OFFSET adjusting knob until the output is near zero. Then change to the desired RANGE and, if necessary, rezero with the OFFSET adjusting knob.

12. The system is now ready for standards and samples.

WARNING: The CELL MODE switch must be in the STBY position when the LC-3C is turned on or off, and for any electrode servicing. Disassembling a “live” cell can permanently damage the working electrode and the electronic circuits.

Section 6. Electronic Troubleshooting

Test Procedure, LC-3C/D/E

1. With the CELL MODE switch in the STBY position, turn the POWER on. The LCD display should be active. If the LCD does not come on, check the power supply and wall outlet. If the power supply and wall outlet are okay, replace the 7.2 V NiCad battery.

2. Set the DISPLAY switch to OFFSET nA and adjust the OFFSET control knob to zero.

3. Set the RANGE to 10 nA for the LC-3C, 200 nA for the LC-3D, or 200 µA for the LC-3E.

4. Set the FILTER to 0.3 Hz.

5. Plug the black probe of a digital voltmeter into the GND, and the red probe into the green 1 V output jack. Set the CELL MODE switch to the TEST position. Using the first two columns of Table 6.1 as a guide, set the POTENTIAL and RANGE as indicated, and compare output of the front panel display and the voltmeter reading with the table. Using the first entry from the table as an example, when APP E is set to 1.0 (V) and the RANGE is 10 nA, the output as read on the front panel display should be 10.0 nA and the corresponding output voltage should be 1 V. Check the remainder of the outputs (current and potential) for the applied potentials and ranges in the table. It may take 10 to 15 seconds for the voltmeter reading to stabilize when switching between certain ranges, due to the influence of the filter on the LC-3C/D/E. This is normal.

6. Change the display to Offset. Turn the OFFSET control knob until a reading of 1 nA is shown on the LC-3C display, or 10 nA on the LC-3D, or 10 µA on the LC-3E. The output reading on the voltmeter should be 0 - 150 mV. Again, voltmeter readings may take 10 - 15 seconds to stabilize.

7. If the instrument fails any step of this procedure, contact BASi customer service personnel.

Table 6.1. Expected readings for self-test.

	Applied E (V)	Range	Display Output	Back Panel Output (V)
LC-3C:	1.000	10 nA	9.80 - 10.20 nA	0.980 - 1.020
	1.000	5.0 nA	9.80 - 10.20 nA	1.960 - 2.040
	1.000	2.0 nA	9.80 - 10.20 nA	4.900 - 5.100
	0.100	1.0 nA	0.950 - 1.050 nA	0.950 - 1.050
	0.100	0.5 nA	0.950 - 1.050 nA	1.900 - 2.100
	0.100	0.2 nA	0.950 - 1.050 nA	4.750 - 5.250
LC-3D:	1.000	200 nA	98.0 - 102.0 nA	0.490 - 0.510
	1.000	100 nA	98.0 - 102.0 nA	0.980 - 10.20
	1.000	50 nA	98.0 - 102.0 nA	1.960 - 2.040
	0.100	20 nA	9.50 - 10.50 nA	0.475 - 0.525
	0.100	10 nA	9.50 - 10.50 nA	0.950 - 1.050
	0.100	5 nA	9.50 - 10.50 nA	1.900 - 2.510
LC-3E:	1.000	200 A	98.0 - 102.0 A	0.490 - 0.510
	1.000	100 A	98.0 - 102.0 A	0.980 - 10.20
	1.000	50 A	98.0 - 102.0 A	1.960 - 2.040
	0.100	20 A	9.50 - 10.50 A	0.475 - 0.525
	0.100	10 A	9.50 - 10.50 A	0.950 - 1.050
	0.100	5 A	9.50 - 10.50 A	1.900 - 2.510

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