

TEMPERATURE CONTROLLERS FOR  
LIQUID CHROMATOGRAPHY

OPERATOR'S MANUAL

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#### 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.

\* \* \* PREFACE \* \* \*

The Bioanalytical Systems LC-22A Temperature Controller and LC-23A and LC-23B Column Heating Compartments are versatile tools for the experimenter who desires precise, reproducible temperature control for liquid chromatography.

The LC-22A Temperature Controller is a general purpose instrument capable of regulating devices such as heating mantles, heat tapes, electrochemical cell preheaters, and column heating blocks. The temperature control point is set digitally, and the power applied to the heating unit is regulated by a temperature sensing device located in the device being controlled. In temperature heating devices of BAS manufacture, the temperature sensor is built into the instrument. To control other devices, a separate, universal probe (part no. EW-8116) is available for separate purchase.

The LC-23A Column Heating Compartment is used in conjunction with the LC-22A. This instrument is simple in design and extremely durable. It accepts most commercially available 15, 25 or 30 cm analytical columns. A solid aluminum inner compartment, thermally isolated from the outer cover, encloses the entire column. A preheater coil is included with each unit. The LC-23A can, in some instances, be altered to accommodate wider diameter columns. This alteration would involve an additional charge for customization. If you require modifications, please contact BAS customer service for a quotation.

The LC-23B Cartridge Column Heating Compartment is sold within the BAS CC-4 Chromatography Compartment, which is also standard in BAS 400 Liquid Chromatographs and Electrochemical Detectors. The user can add a Rheodyne injector to the CC-4 in a BAS detector or LC22A/23B cartridge heater and convert the compartment to a basic liquid chromatograph. The LC-23B is also sold without the CC-4 as an upgrade to BAS detectors or BAS 400 systems.

The Cell Preheater option for BAS electrochemical detectors is also controlled by an LC-22A controller. The preheater coil can accommodate the heating of stainless steel tubing between the column and cell inlet.

## A. POWER REQUIREMENTS

### LC-22A Temperature Controller

The instrument is normally supplied for either 120V/60Hz or 240V/50Hz. Unlike other BAS instruments, the power supply is not interconvertible on each unit. Do not attempt to operate on line voltages other than specified on the serial number label on the back panel of the instrument.

A 3-prong power cable on the LC-22A must be connected to a standard, grounded power outlet for safety. If the instrument is operated from a power outlet without a ground connection, an appropriate adapter should be used and the ground connection of this adapter should be securely fastened to an earth ground.

### LC-23A and LC-23B Heating Compartments

The LC-23A and LC-23B compartments are designed specifically for use with the LC-22A controller. Two cables (one for heater power and the other for the temperature sensor) connect to the LC-22A. If you have purchased a heating compartment for use with an older model LC-22A, you may be unable to make the connection directly to the controller without an additional adapter. A change in design converted this from a 3-prong plug to the current female plug. The adapter (EF-1575) is available from BAS at an additional charge.

## B. INSTALLATION

### LC-22A Controller

This section concerns stand-alone operation of the LC-22A with the optional universal probe. For operation of the other accessories, see subsequent sections.

1. Use the cable adapter (EF-1575) to convert the special safety receptacle on the back panel of the LC-22A to a standard 3-prong socket. Plug the device to be controlled (e.g. heating mantle, tape, etc.) into this socket. If you only wish to measure temperature and do not need to control it, you can skip this step.
2. Plug the cable leads from the stainless steel UNIVERSAL PROBE (EW-8116) into the red, yellow, and black jacks on the back panel of the LC-22A labeled "liquid probe". The black jack provides grounding for the instrument and must be connected for safety purposes.
3. Move the toggle switch on the rear panel to the "LIQUID PROBE" side of the panel.

4. The LC-22A must be calibrated to obtain accurate thermostatic control using the liquid probe. If accuracy of the set-point is not important for the application, the liquid probe is usually within  $1^{\circ}\text{C}$  of the actual set-point when shipped. The reproducibility or precision of the temperature set-point is plus or minus  $0.1^{\circ}\text{C}$  regardless of the actual temperature. To calibrate the probe, place it in an ice bath and connect an external voltmeter into the OUTPUT jacks on the rear panel of the LC-22A. Turn the set point to  $00.0^{\circ}\text{C}$  and turn the power ON. The voltmeter should read close to  $00.0$  volts. To adjust the instrument, place a small screw driver in the hole just to the left of the liquid probe area on the back panel. Turn the screw until the output voltage monitored by the voltmeter reads  $00.0$  volts. The LC-22A is now calibrated for the liquid probe being used.
5. Remove the probe from the ice bath and proceed.

#### LC-23A Column Heating Compartment

1. Place the LC-23A Heating Compartment in the desired location. Six holes with captive 6-32 threads are provided for secure mounting on your liquid chromatograph, if desired. The holes are positioned to fit early model (<1986) BAS liquid chromatography support stands.
2. Connect the LC-23A heater power cable to the power socket on the rear panel of the LC-22A controller.
3. Connect the red, black, and yellow plugs on the probe cable to the jacks marked LC-23A Probe on the LC-22A back panel. Flip the rear panel toggle switch to "LC-23A PROBE".
4. Remove the cover on the LC-23A Column Heater by unscrewing the 2 captive screws. Place the column in the recessed cavity and route the inlet and outlet tubing out of the top and bottom slots. A special preheater block (EW-8146) is also supplied. Under certain circumstances, dramatic band broadening may result if the preheater is not utilized. Typically this becomes obvious when the temperature differential between ambient and the desired set point exceeds  $20-25^{\circ}\text{C}$ .

If you plan to use several different styles of column endfittings, it is best to use removable plastic fittings. BAS recommends a 2-piece teflon ferrule/steel collet seal as provided in BAS part no. MR-4018 (this includes a nut, collet and ferrule). One piece plastic fittings tend to loosen at higher temperature.

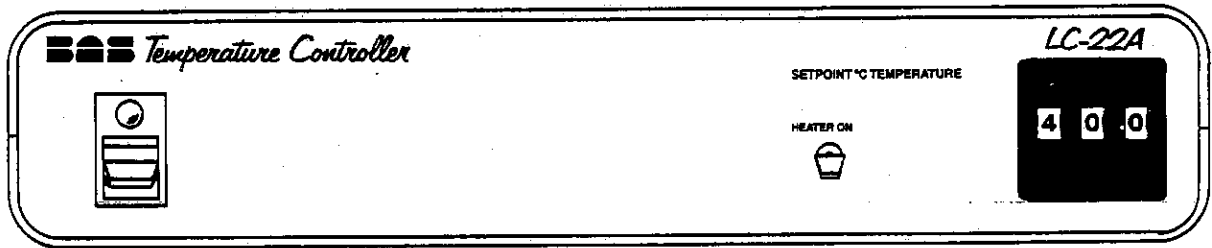


Figure 1. LC-22A Front Panel

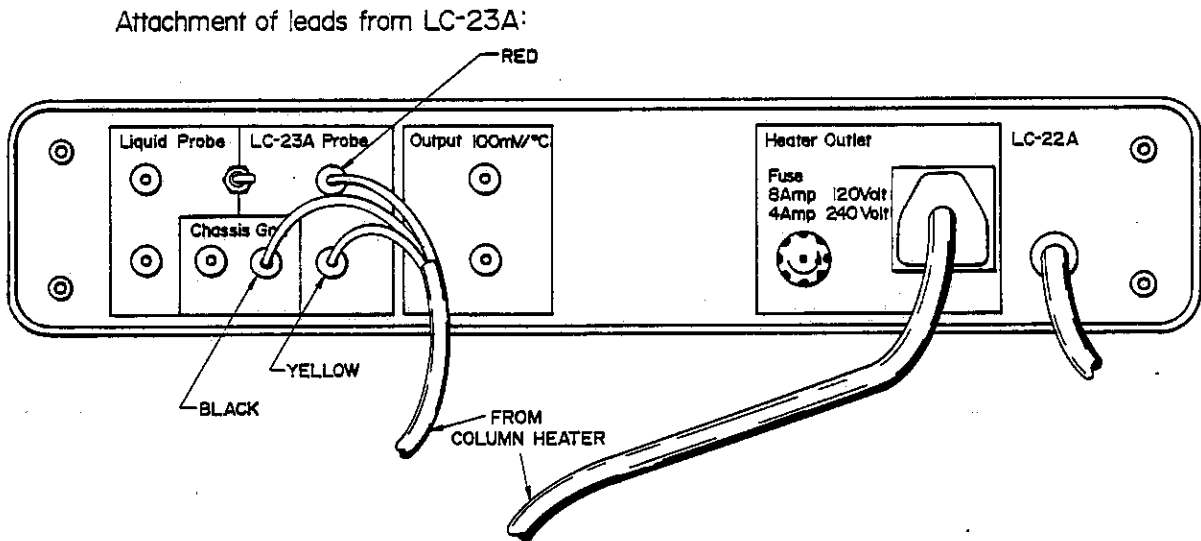


Figure 2. LC-22A Rear Panel

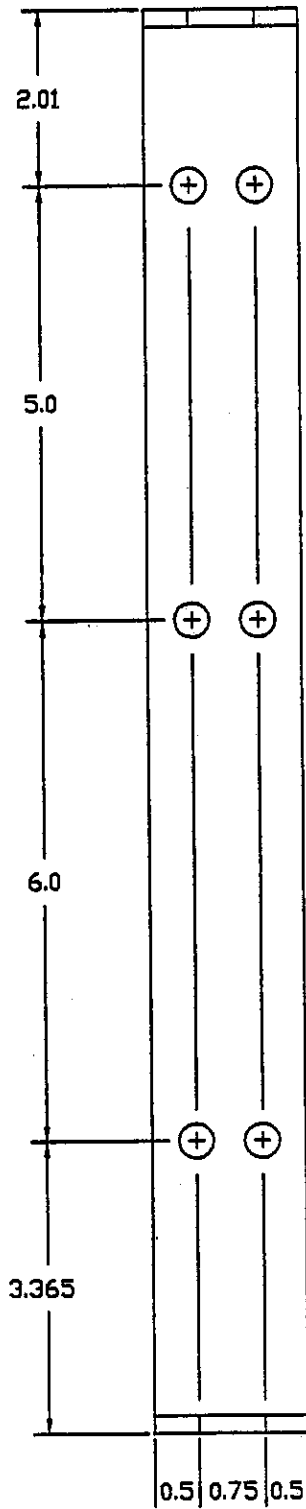


Figure 3. Position of Mounting Points on LC-23A Heater.

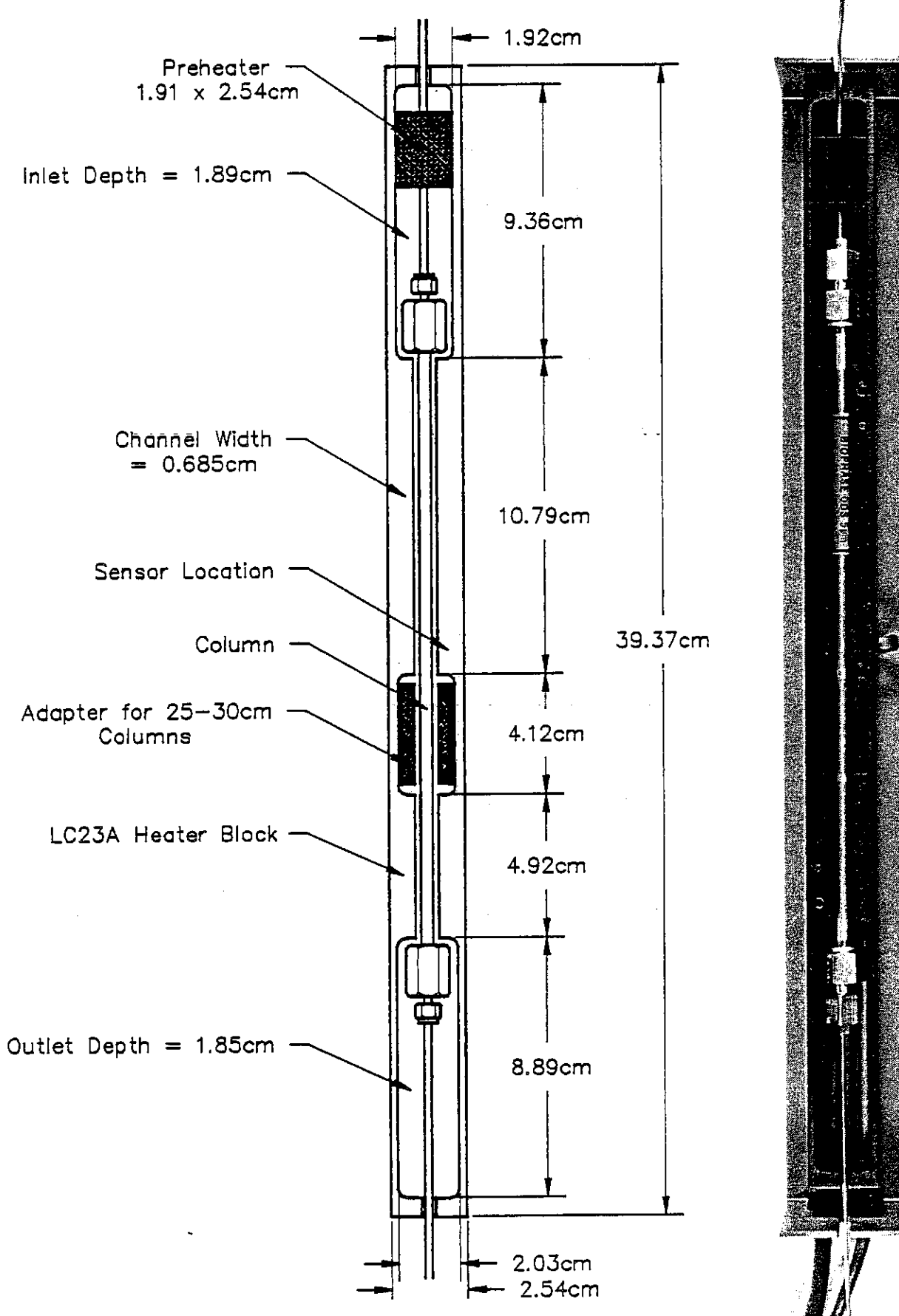


Figure 4. LC-23A Column Heater with LC-23PH Preheater Installation.

## LC-23B Column Heating Compartment

The LC-23B is specifically designed to fit within the BAS CC-4 Column Compartment. The CC-4 is pre-drilled to accept Rheodyne injection valves in the front panel. It has several grooves on both sides for convenient access to external detectors.

If you are installing the LC-23B as an upgrade to a BAS electrochemical detector or BAS 400 liquid chromatograph, and already have a cell preheater installed in the CC-4, you may wish to remove it. Routing the tubing from the LC-23B to the cell preheater and then to the detector is difficult due to space limitations. The LC-23B alone will provide satisfactory temperature control for the electrochemical cell if you keep the cover closed during operation.

Before installing the LC-23B, give some thought to the configuration of the column(s) you intend to use. Some configurations using compact BAS cartridge columns are diagrammed in Figure 5. You can also leave the analytical column outside the LC-23B (at room temperature) and heat a post-column reactor (tubing coil, packed bed) within the LC-23B.

### Installing the LC-23B in a CC-4 Compartment

1. Lower the CC-4 lid and remove the utility cover from the back panel of the CC-4 cabinet (4 screws).
2. On some models, pressed-in, threaded nuts will be located on the outside of the rear panel (Figure 6). These must be removed. DO NOT REMOVE IF THEY ARE ON THE INSIDE OF THE CABINET. To remove external nuts, screw a long machine screw (6-32 thread) into the nut from the rear until it protrudes through the nut. Use pliers for leverage and gently work the screw from side to side until the nut comes loose.
3. Remove the LC-23B cover and set aside. Push the heater block through the now open utility access in the CC-4 back panel. On some models, the 2 registration posts on the CC-4 inner panel will engage the LC-23B block for support.
4. Secure the LC-23B to the cabinet using 4 6-32 x 1/2" machine screws and 4 6-32 lock nuts. The lock nuts are not necessary if pressed-in nuts are available on the inside of the CC-4 cabinet. The mounting plate should now be flush with the outside of the rear panel on the CC-4 cabinet.

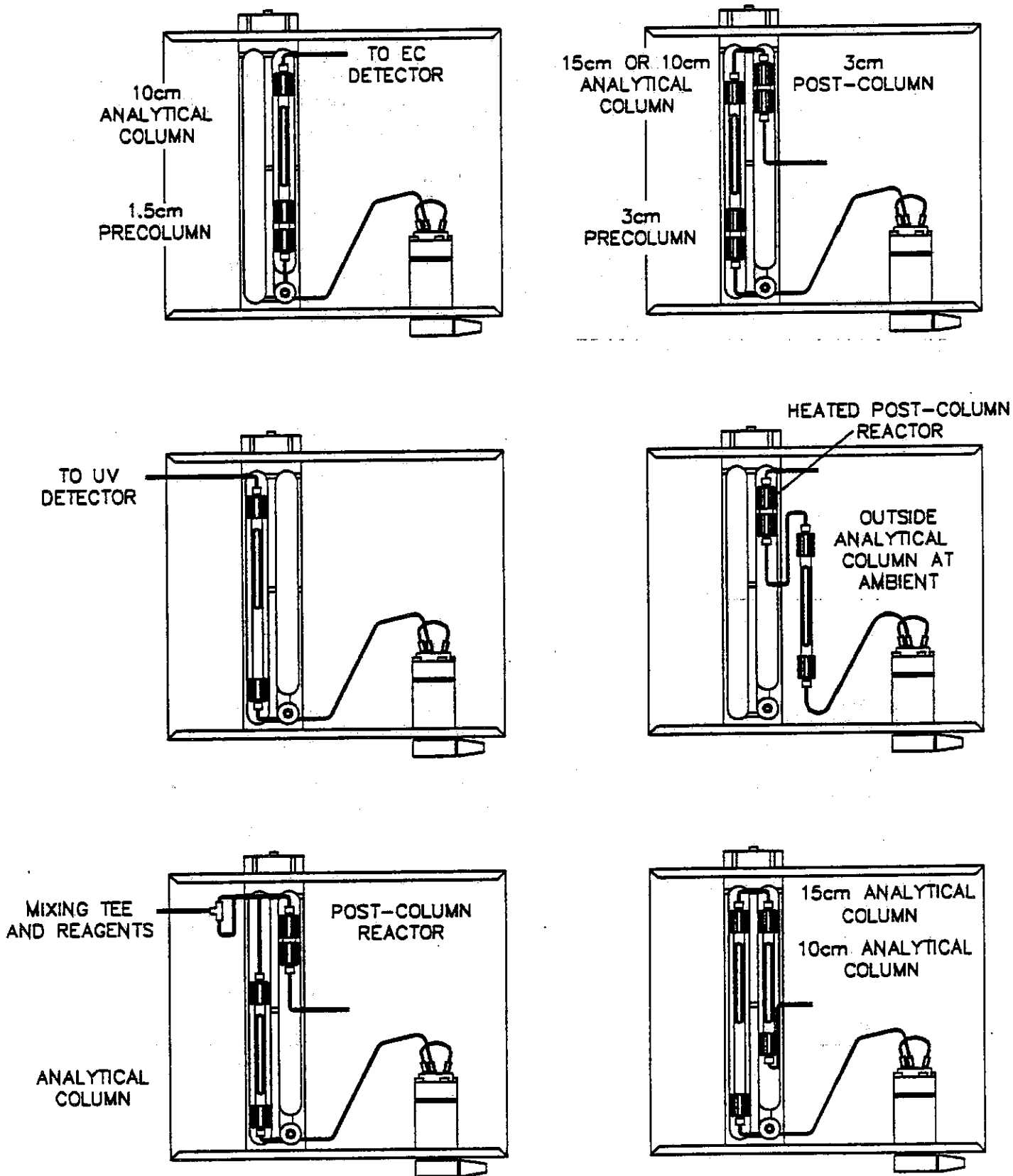


Figure 5. Combinations of column formats within the LC-23B.

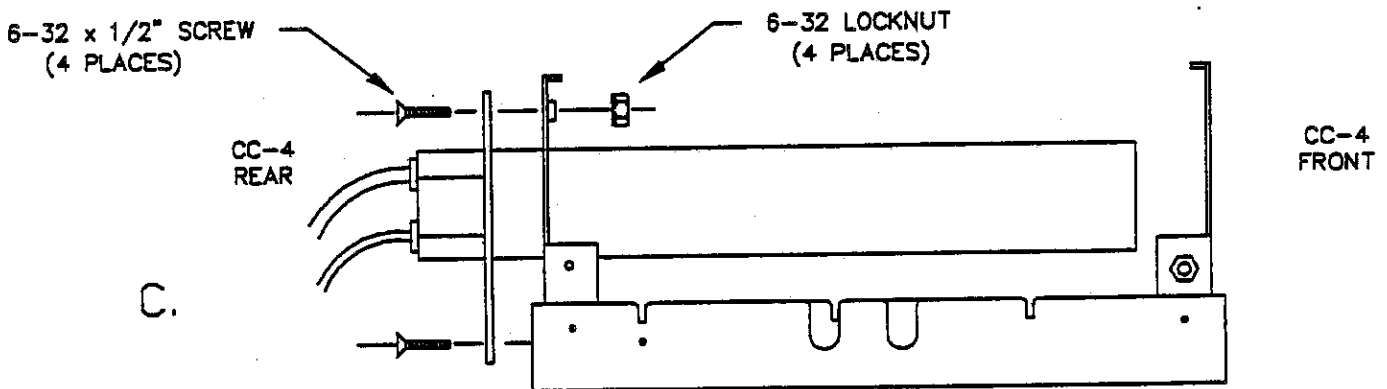
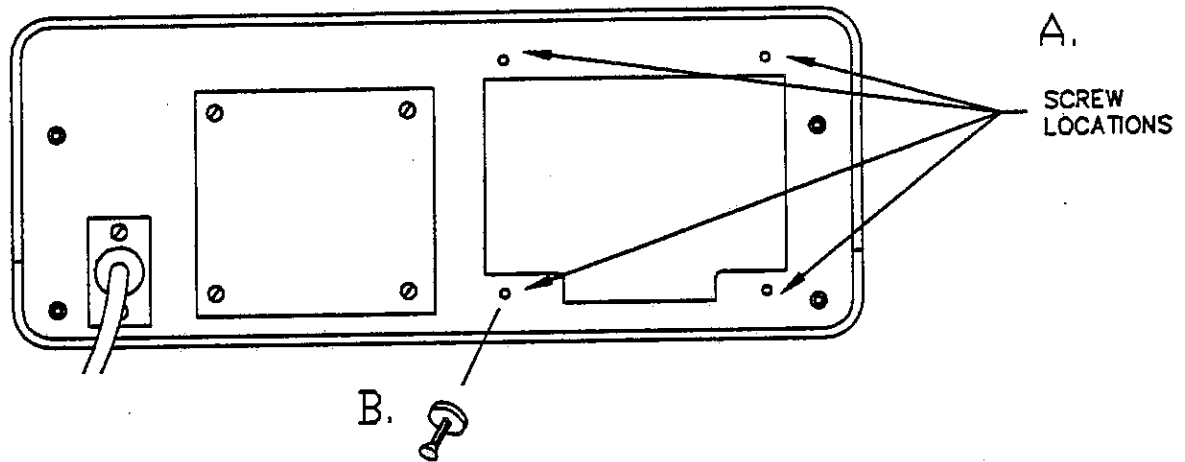


Figure 6. Installation of the LC-23B in a BAS 400 or LC4B/17A Electrochemical Detector. (A) Location of 4 screws (B) breaking off the pressed-in nuts (C) left side view of attachment hardware.

## Making Plumbing Connections

For simple arrangements, use the innermost trough of the LC-23B. This location minimizes detector and valve hookup tubing. If you are coupling columns together, the longer trough will be more useful.

A special coil of tubing is provided with each LC-23B (see Figure 7). At your option, it may be installed between the injection valve and column. The coil functions as a mobile phase preheater, so that large temperature gradients at the head of the column are minimized. The coil may be removed by unscrewing the central cap screw (5/32" hex).

It is advisable not to use a permanent ferrule on the outlet end of the preheater coil. A removable high-pressure fitting provides an equivalent seal without irreversible attachment. Use BAS part no. MR-4018.

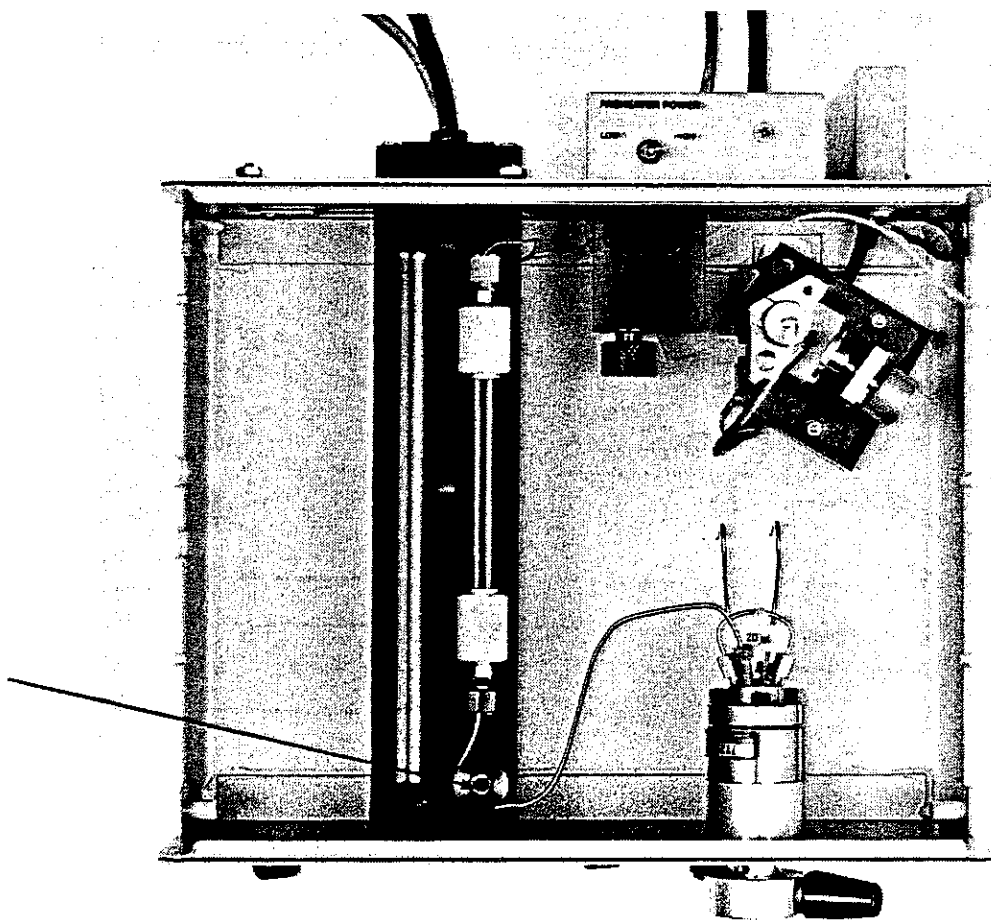


Figure 7. Column Preheater Coil installed in LC-23B. This unit is part of a BAS 400 Liquid Chromatography System with the Electrochemical Cell and Cell Preheater options installed.

## Leaks

Make sure that all fittings are leak-tight at the usual operating pressure before mounting the insulating top cover.

## Insulating Top Cover

The U-shaped top cover is insulated on 3 sides with a foam polymer. The cover is asymmetric; the notch in the cover goes along the right side.

Mount the cover at the rear first and then drop the front in place (Figure 8).

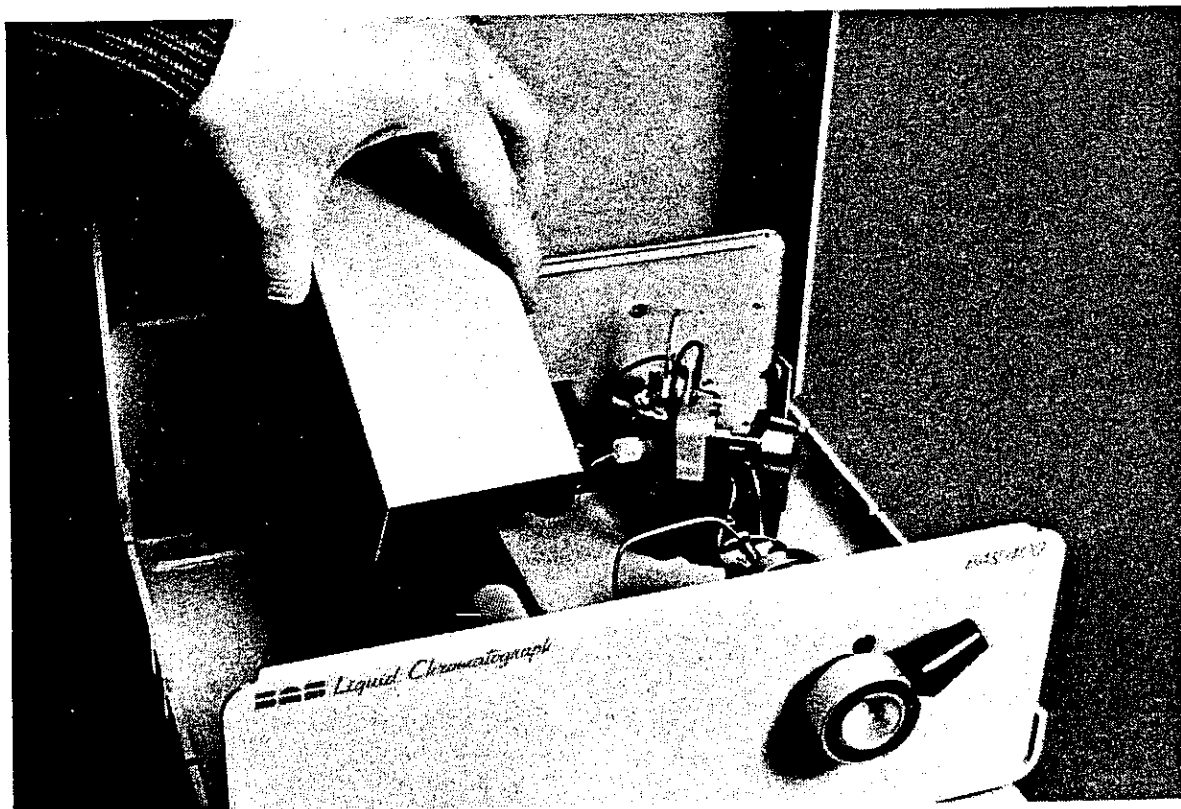


Figure 8. Mounting the top cover.

## C. OPERATION

### Temperature Control Mode

1. Set the digital switch to the desired control temperature. The value must be between ambient and 99.9° C. Initiate mobile phase flow if using the LC-23A or LC-23B Column Heating Compartments.
2. Turn on the main power for the LC-22A to initiate heating.
3. The LED indicator light will remain constantly illuminated until the temperature set point is reached, then it will blink intermittently to indicate maintenance at the desired temperature.

### Temperature Measurement/Control Mode: Universal Liquid Probe

1. Ensure that the stainless steel probe is plugged into the jacks marked LIQUID PROBE and observe the color code used in the jacks. Flip the toggle on the LC-22A back panel to LIQUID PROBE.
2. Turn on the main power to the LC-22A.
3. ~~The measured temperature is available at the jacks marked OUTPUT as an analog voltage signal. Each 100 mV (0.1 volt) is equivalent to 1° C. For example:~~

$$\begin{aligned} + 8.92 \text{ volts} &= + 89.2^{\circ} \text{ C} \\ - 0.36 \text{ volts} &= - 3.6^{\circ} \text{ C} \end{aligned}$$

This voltage may be recorded using a digital voltmeter or a strip chart recorder. Set the full scale input of the recorder to 10 Volts. Connect the "+" to the red jack and the "-" to the black jack.

4. To control the temperature while measuring it, first turn off the power to the LC-22A. Connect the power cable from the heating device into the LC-22A, using the adapter cable (EF-1575). Set the temperature on the LC-22A to the desired setpoint. Turn on the power to the LC-22A. The LED light will stay on continuously until the heater achieves the setpoint and then will flash intermittently thereafter while maintaining this temperature. The actual temperature (within the absolute accuracy of the probe) at any point in time will be displayed on the voltmeter or recording device.

### Temperature Measurement/Control Mode:

#### LC-22A or LC-23B Column Heaters and Electrochemical Cell Preheater

1. Ensure that the three leads from the column heaters or cell preheater are plugged into the jacks marked LC-23A PROBE and

observe the color code used in the jacks. Flip the toggle on the LC-22A back panel to LC-23A PROBE.

2. Connect the signal cable (EW-8107) from the OUTPUT jacks of the LC-22A back panel to the back panel of the LC-4B Amperometric Controller. Use the section labeled TEMPERATURE IN. If you do not have an LC-4B controller available, use a digital voltmeter.
3. Set the temperature of the LC-22A to desired point (at least  $10^{\circ}$  C above ambient). Turn on the LC-22A and LC-4B.
4. For users of the LC-4B Amperometric Controller, temperature display is shown on the front panel LED by rotating the MODE knob to read  $^{\circ}$ C.
5. For users without an LC-4B controller, the measured temperature is available at the jacks marked OUTPUT as an analog voltage signal. Each 100 mV (0.1 volt) is equivalent to  $1^{\circ}$  C. For example:

$$\begin{aligned} + 8.92 \text{ volts} &= + 89.2^{\circ} \text{ C} \\ - 0.36 \text{ volts} &= - 3.6^{\circ} \text{ C} \end{aligned}$$

This voltage may be recorded using a digital voltmeter or a strip chart recorder. Set the full scale input of the recorder to 10 Volts. Connect the "+" to the red jack and the "-" to the black jack.

6. The LED light will stay on continuously until the heater achieves the setpoint and then will flash intermittently thereafter while maintaining this temperature. The actual temperature (within the absolute accuracy of the probe) at any point in time will be displayed on the LC-4B, voltmeter, or recording device.

#### D. SAFETY AND MAINTENANCE CHECKS

The LC-22A, LC-23A, LC-23B and cell preheater are all essentially maintenance free instruments. BAS does advise that you attempt to keep these pieces in good condition by following these suggestions:

1. Avoid corrosive laboratory environments.
2. Don't stack heavy items on signal cables or allow them to become crimped during use or storage.
3. Clean up spills inside column heaters immediately.
4. Avoid contact of organic solvents with the insulating material inside the LC-23B cover.

The Universal Probe is based on a reliable solid state device capable of generating a predictable current related to the temperature of its environment. To check the integrity of this probe, apply the following test:

1. Expose probe to ambient temperature and disconnect any heating devices from the LC-22A.
2. Turn on the LC-22A power.
3. Set the temperature setpoint to a value greater than ambient (allow at least  $1^{\circ}$  C difference). The LED indicator on the LC-22A should be on continuously.
4. An open circuit anywhere in the probe, cable or across its input jacks will automatically shut down the heater circuit. This feature was incorporated to protect against thermal "runaway" and should be tested periodically. Perform the following check:

Plug in the probe, expose to ambient air temperature and turn on the power to the LC-22A.

Using a DC voltmeter, plug leads into the OUTPUT jacks. The measured potential (volts) should be 1/10 of the ambient temperature in  $^{\circ}$  C. This is normal operation in the temperature measurement mode.

Create an open circuit in the probe by unplugging one of its leads from the chassis jacks on the LC-22A. The measured potential at the OUTPUT jacks should now be approximately -13 volts and the HEATER ON lamp should be off.

Remove the probe entirely and place a jumper cable across the PROBE INPUT jacks to create a closed circuit. The OUTPUT jacks should now read +12 volts ( $\pm 1$  V) and the heater light should be off.

If your unit fails either step (3) or (4), please contact the BAS Service Coordinator to return both the LC-22A and the probe used to make these tests.