Spectra-Physics

SP8440 VARIABLE WAVELENGTH DETECTOR
Operating and Service Manual

Part Number A0099-440

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WARNING

The deuterium lamp, in the SP8440 detector, emits strongly in the ultraviolet range. The user should take precautions for the two main hazards:

1. The deuterium lamp should not be viewed directly by the eye during operation. Direct viewing can "sunburn" the retina. All personnel in the area should wear glasses, or safety glasses, if the SP8440 is operated with the top cover off, thus exposing the lamp to direct view.

2. All deuterium lamps generate ozone from atmospheric oyxgen. Ozone is a toxic gas and should be removed from the room by a forced air vent. The susceptibility of individuals to ozone varies greatly.

Notes, Cautions, Warnings

Throughout this manual, items appear in boxes which are of special importance to the operator.

NOTE

A NOTE specifies a special condition that may affect the data being collected.

CAUTION

A CAUTION specifies a special condition that may affect the instrument, usually in a harmful manner.

WARNING

A WARNING specifies a special condition that may prove harmful to the operator. Do not ignore a WARNING message.

1

OPERATION

Figure 1.1 The SP8440 Variable Wavelength Detector.

1.1 INTRODUCTION

The SP8440 is a variable wavelength liquid chromatography detector, operating between 190 nm (nanometers) and 600 nm in the UV/visible portion of the spectrum.

In addition to manual operation, the SP8440 detector can be controlled by remote programed commands. This is accomplished by appropriate optional interface circuitry.

1.2 POWER

1.2.1 Power Requirements

The SP8440 operates on either 50 or 60 Hertz power lines of from 90 to 135 volts, or from 180 to 270 volts. The power drawn is about 130 watts. In terms of current, 1.1 amperes at 120 volts, or 0.6 amperes at 240 volts.

The central pin in the power connector is the ground for the instrument. This pin must find (be connected to) a true earth ground (such as a cold water pipe) if the detector is to meet specifications.

1.2.2 Main Line Fuse

At the back of the unit is a power plug connector (Figure 1.2). If the power cord is removed, the plastic cover can be slid to the left, over the power pins, to reveal a fuse. The fuse lever (labeled FUSE PULL) may be pulled to remove the fuse. The fuse should be;

2.0 amp 250 volt slow-blow for 100 or 120 volts

1.0 amp 250 volt slow-blow for 220 or 240 volts

Be sure the correct fuse is installed.

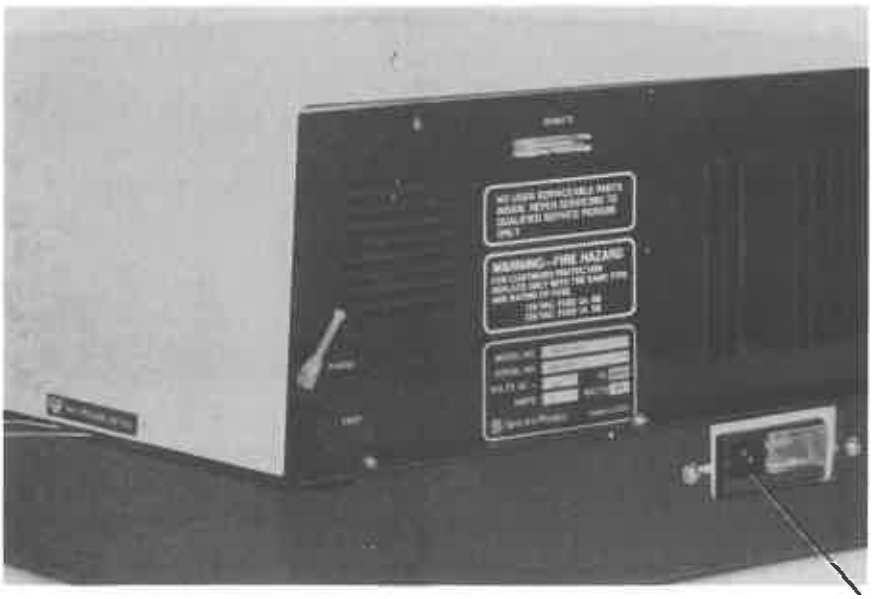


Figure 1.2 Power Connector

1.2.3 Voltage Selection

Just below the fuse is a small printed circuit board. This is labeled according to the power line voltage the unit is adapted to. If the visible labeling does NOT match the line voltage, remove the board, turn it around till the label will match, and re-insert the board into the connector. Slide the plastic cover back over the fuse and board (Figure 1.3).

1.2.3.1 Filament Transformer Fuse

When changing the voltage selection board (Figure 1.3) to match the line voltage, you must also verify the power line frequency (either 50Hz or 60Hz); and move the 5 ampere slo-blo fuse to position F5 or F6 on the power supply board (Figure 1.4). These two fuse locations are identified on the board as 50Hz or 60Hz. Install the fuse blank provided in the unused position (F5 or F6).

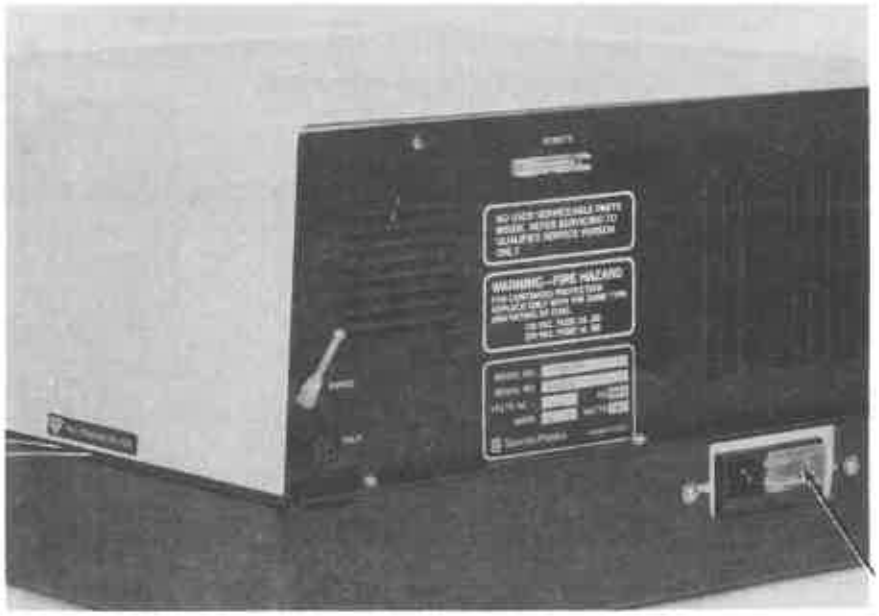


Figure 1.3 Voltage Selector.

1.2.4 Power Switch

The power ON/OFF switch is at the front, on the lower left side (Figure 1.4). This is a push-push type switch. That is, push once to turn the instrument ON, and push again for OFF. Note that the switch remains in when turned ON.

Figure 1.4 Power Switch.

Turn the unit OFF, attach the power cord, and plug it into a grounded power socket.

1.3 CONNECTIONS

1.3.1 Recorder/Integrator Cable

Locate the Recorder/Integrator output cable (A1463-010) in the Accessory kit. Plug it into the OUTPUT connector P901 at the back of the unit. (It will only go in one way.) Note the wires with spade lugs coming out the other end of the cable.

The black wire is the detector ground and also the return (low) line for both output signals. This wire should be connected to the low or minus terminal of the recorder or integrator.

The clear or white wire is the 10 mv Full Scale high output (positive) for the recorder. The output signal on this wire is corrected to the equivalent of a 1.0 cm flow cell optical path length. It comes from the Range switch on the front panel (Figure 1.5). The wire should be connected to the high (HI), or plus (+) terminal of a 10 mv recorder.

Figure 1.5 Range Switch.

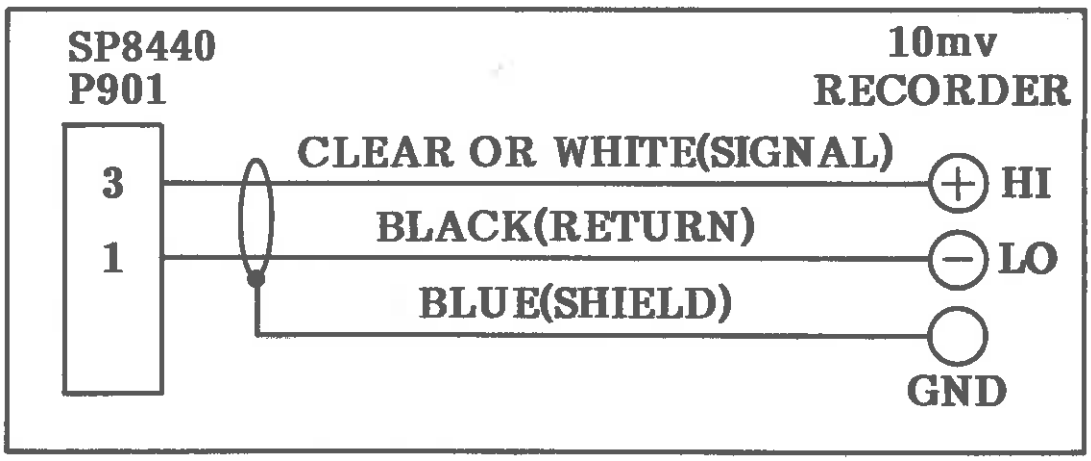


Figure 1.6 Recorder Wiring.

The red wire is a fixed 1.0 volt per 1.0 Absorbance Units (A.U.) for an integrator (with a 0.5 cm optical flow cell path). This wire should be connected to the high (HI), or plus (+) terminal of the integrator input (Figure 1.7).

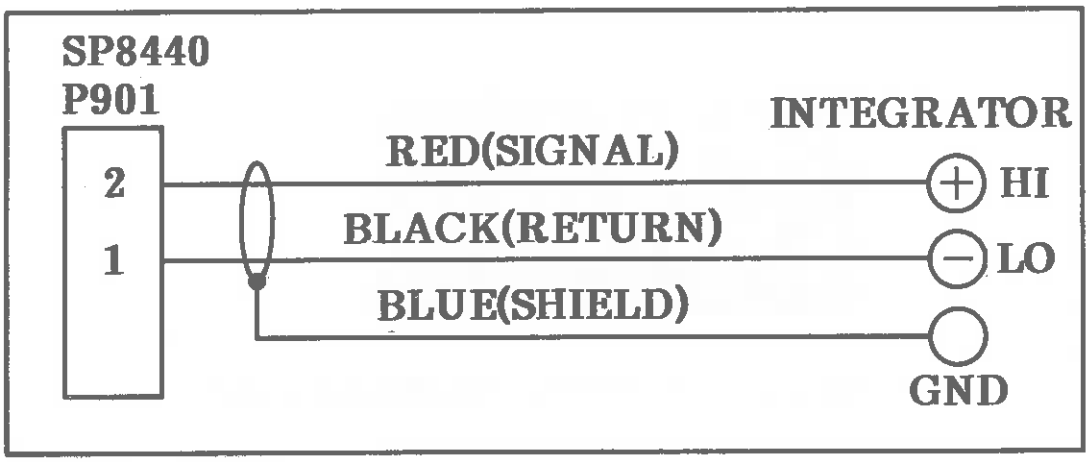


Figure 1.7 Integrator Wiring.

If the detectors output connection is to an integrator, the recorder should be connected to the integrator's attenuator output. (Figure 1.8).

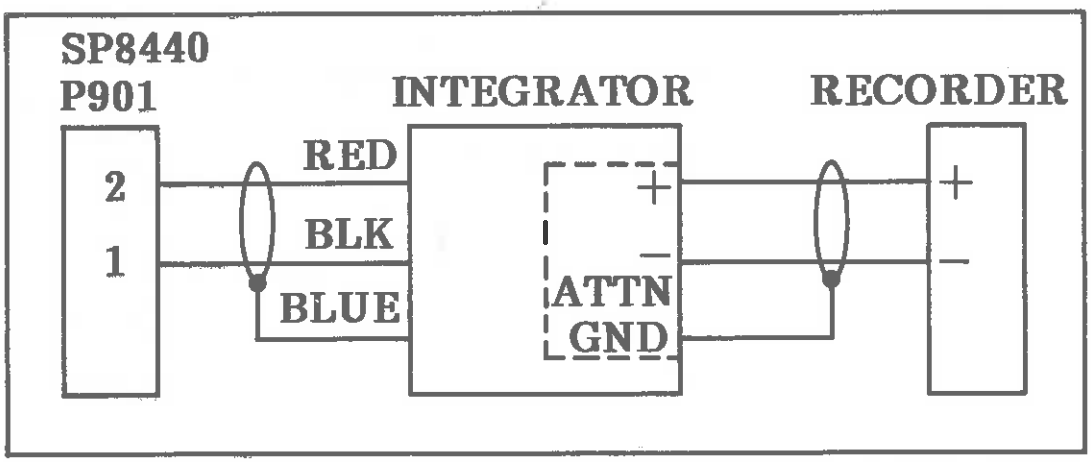


Figure 1.8 Wiring in Tandem.

Either connection (to a recorder or integrator) leaves one terminal free. Bend the unused wire back along the cable and tape it down. Also tape over the bare terminal lug to protect the detector's output (Figure 1.9).

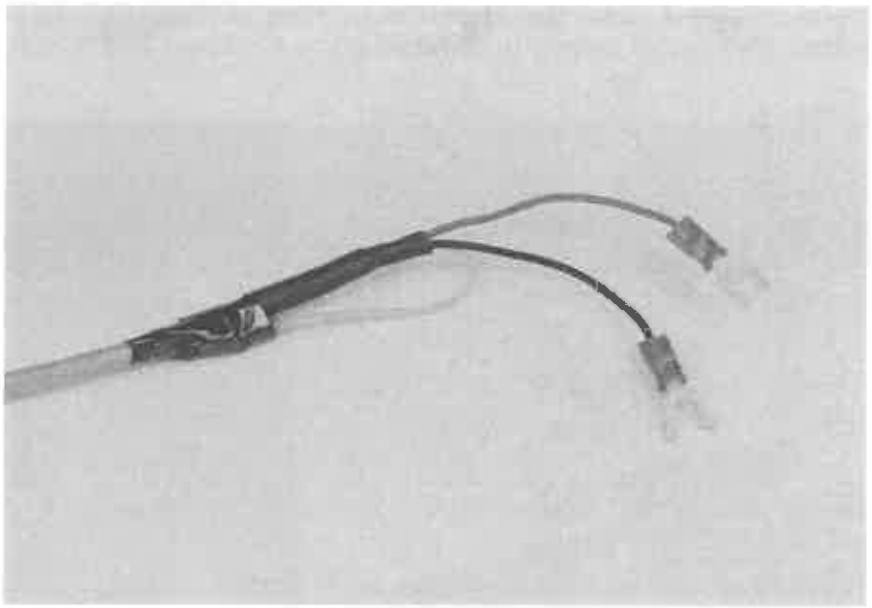


Figure 1.9 Terminated Cable.

If the integrator doesn't have an output for the recorder, the recorder may be connected in "parallel". That is, the recorder is connected between the clear (or white) and black wires, and the integrator is connected between the red and black wires (see Figure 1.10).

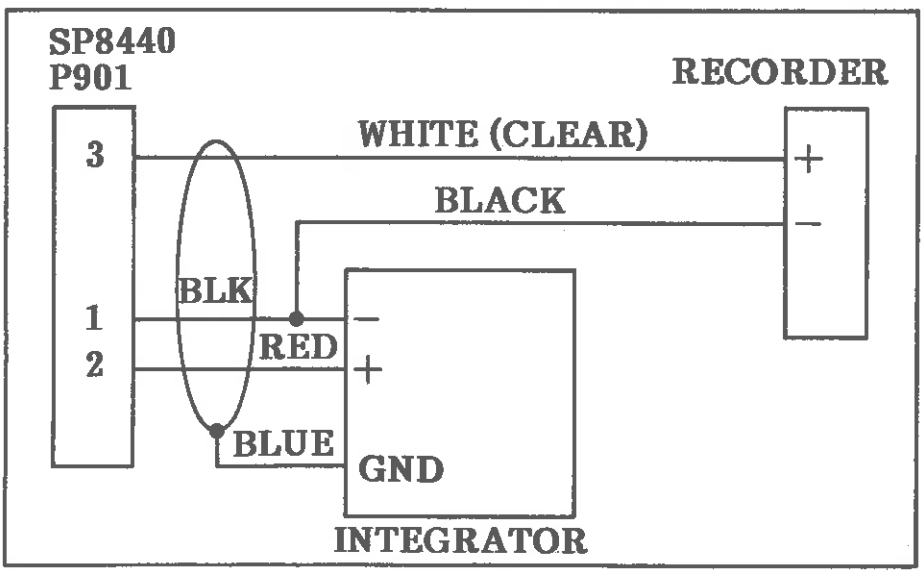


Figure 1.10 Wiring in Parallel.

1.3.2 Liquid Connections

The output of the chromatograph's column should be directly connected to the liquid INLET of the SP8440 detector. This connection is at the right side, when facing the detector. Note the direction of the connection (Figure 1.11). The direction of flow is important to reduce turbulence. The outlet connection should go to a solvent waste bottle.

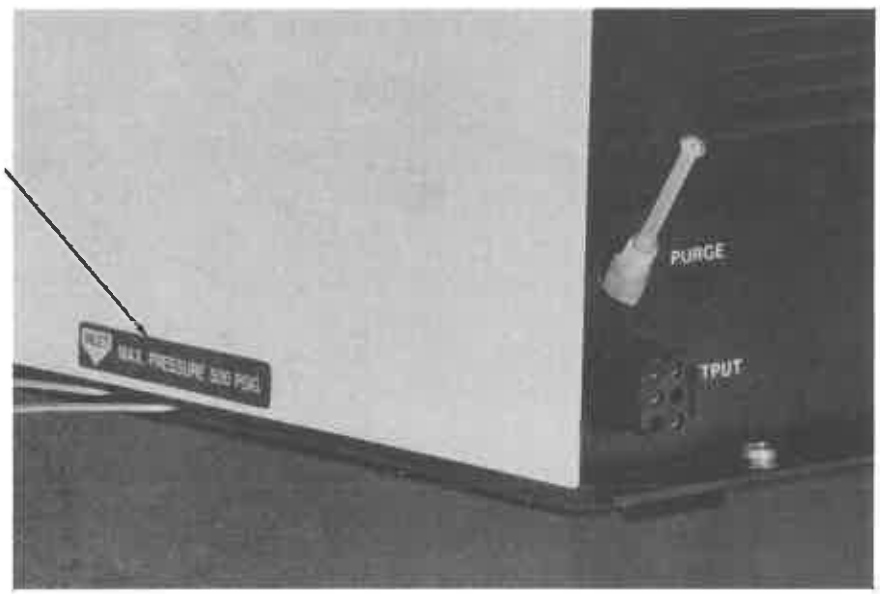


Figure 1.11 Liquid Connections.

The flow cell is made of stainless steel. A flow restrictor may be connected between the detector outlet and the waste bottle in order to maintain pressure and avoid bubble formation inside the flow cell. The pressure in the flow cell should not exceed 500 psig (34BAR).

1.3.3 Nitrogen Purge

At wavelengths below 260 nm, the ultraviolet light creates ozone. The ozone, in turn, absorbs the ultraviolet light, reducing its intensity. For this reason, to obtain optimum performance, the optic path should be purged with nitrogen to remove the oxygen. The plastic tube, and fitting, which extends through the rear of the cabinet is the nitrogen purge INLET line. (Figure 1.12).

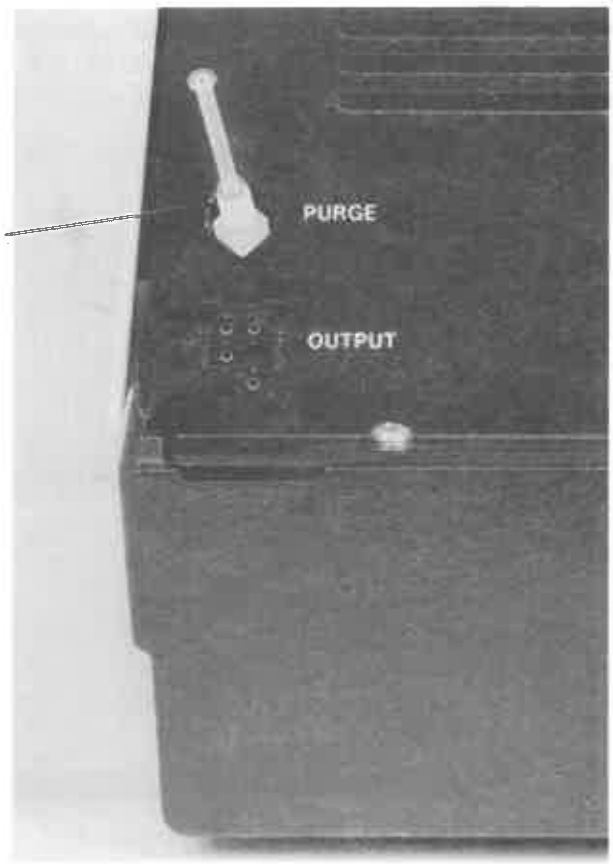


Figure 1.12 Nitrogen Purge Line.

It will be necessary to connect a needle valve at the output of the nitrogen bottle pressure valves. There should also be a flow meter to monitor the flow of nitrogen (such as Airco Model Number G-2130-4 or S-73). For minimum baseline noise the nitrogen flow should be kept between 15 and 20 cc/minute. This should be oil free, laboratory grade, nitrogen.

NOTE

The SP8440 is ordinarily shipped with the deuterium lamp in a separate container. It is necessary to mount and adjust the lamp before operating the instrument. Instructions are contained in Section 3.4.2 (Lamp Replacement), and 3.4.3 (Lamp Orientation).

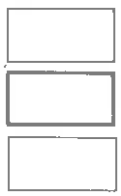
1.4 USING THE DETECTOR

Before using the detector, the following actions should be taken, preferably in sequence. Turn on the nitrogen purge for fifteen minutes if the wavelength is to be 260 nm or less. Turn on the electrical power, and allow the detector to warm up for thirty minutes. Start pumping degassed solvent after turning on the power. This will get any bubbles, or peaks retained on the column, out of the system, and give the column a chance to equilibrate.

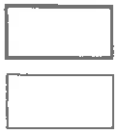
After the thirty minutes warm up, select a wavelength with the thumbwheel switches at the absorbtion maximum of the compounds of interest. If no value is known, a good first choice is 240 nm (Figure 1.13).

Figure 1.13 WAVELENGTH SELECT Thumbwheels.

Push the λ LOAD button. In this case λ = wavelength. So the button's label means "Wavelength Load".



When the LED display shows the selected value, wait for five seconds for the system to settle, then push the AUTO GAIN button.



Set the RANGE switch to SHUNT, and adjust the recorder zero upscale so the pen can move in either direction on the chart paper.

Start rotating the Range switch toward more sensitive positions a step at a time. Watch the recorder pen. If the pen moves off zero, bring it back with the Balance control. Continue moving to more sensitive ranges and adjusting the Balance control until reaching the .0025 A.U. range (Figure 1.14).

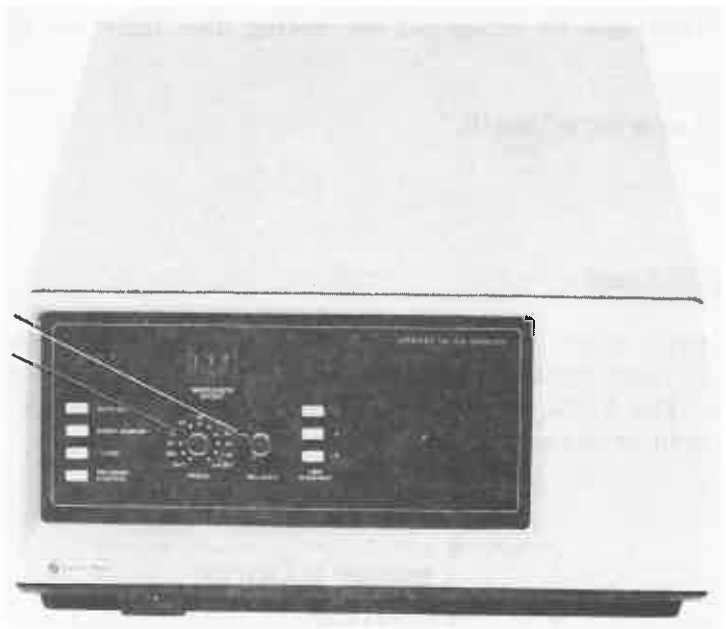


Figure 1.14 Range and Balance Controls.

When the Balance control is properly set, as above, return the Range control to Shunt. Adjust the recorder pen to zero on the chart paper. The Range switch may now be set to any desired position. The recorder pen should not move off zero.

The detector is now ready for a sample run.

1.5 CONTROLS

1.5.1 Power Up

Turn the SP8440 ON by pushing the power switch. Note the LED display will come up reading 150 nm. There will be a moments delay while the wavelength drive motor moves to 150 nm for calibration. Push λ Load. The motor will move to the value on the Wavelength Select thumbwheel switches. This can be observed by noting the LED display changing value.

Now select a new wavelength.

1.5.2 λ (Wavelength) Load

Note the LED's didn't change when the new wavelength was selected. Press the λ Load button, and the motor will drive to the selected wavelength. The LEDs should now agree with the thumbwheel switches. Any wavelength between 190 and 600 nm is usable.

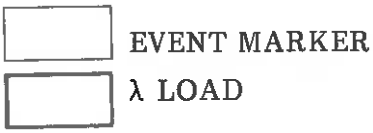
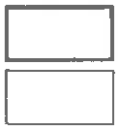


Figure 1.15 LED Display and Wavelength Select Switches.

1.5.3 Auto Gain

The Auto Gain control needs some explanation.



There is only a Sample side of the flow cell. The Reference signal is simulated by a fixed voltage.

At the start of a chromatogram, the baseline output of the sample detector should be made equal to the Reference voltage. This satisfies the initial conditions of the equation:

$$A.U. = \log_{10} \frac{I\ reference}{I\ sample}$$

Where I is the intensity of the light.

Pressing Auto Gain adjusts the output of an amplifier until the sample baseline equals the reference voltage. This brings the output of the logarithmic converter to within 400 A.U. of zero as seen on the .0025 A.U. range. The output of the logarithmic converter is then combined with the Balance control to set the recorder pen to zero on the chart scale.

Auto Gain should be done after selecting a new wavelength, and just before starting a chromatogram while pumping the mobile phase of interest.

1.5.4 Range

The Range control sets the level of sensitivity seen at the recorder output cable (white wire), but not the integrator output (red wire). The output is calibrated in Absorbance Units (A.U.) for a 10 mv full scale recorder, and a 1 cm flow cell optical path.

The Shunt position of the Range switch shorts the output to ground. It should be used to zero the recorder pen (Figure 1.16).

Auto Gain should be done at least 5 seconds AFTER selecting a new wavelength, and just before starting an analysis while pumping the mobile phase of interest.

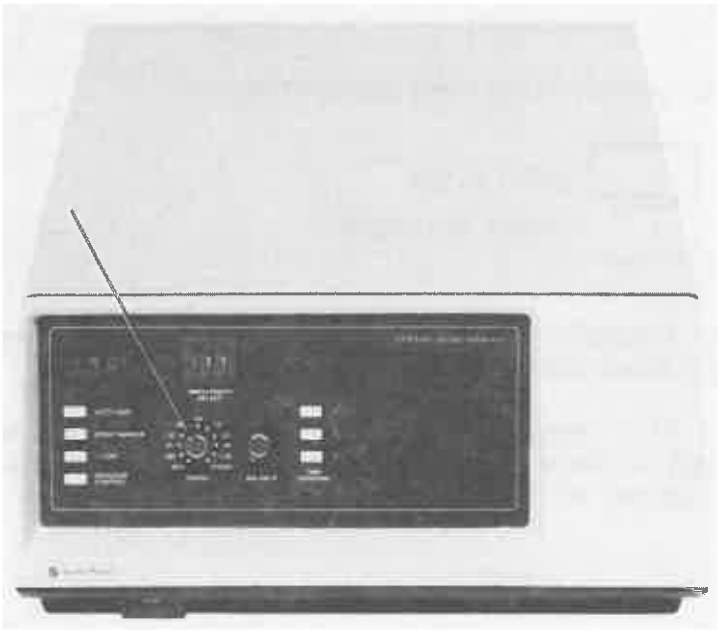


Figure 1.16 Range Rotary Switch.

1.5.5 Balance

As the Range switch is advanced to more sensitive settings, the Balance control is used to keep the recorder pen at zero. (This assumes AUTO GAIN has previously been done.) (See Figure 1.17.)

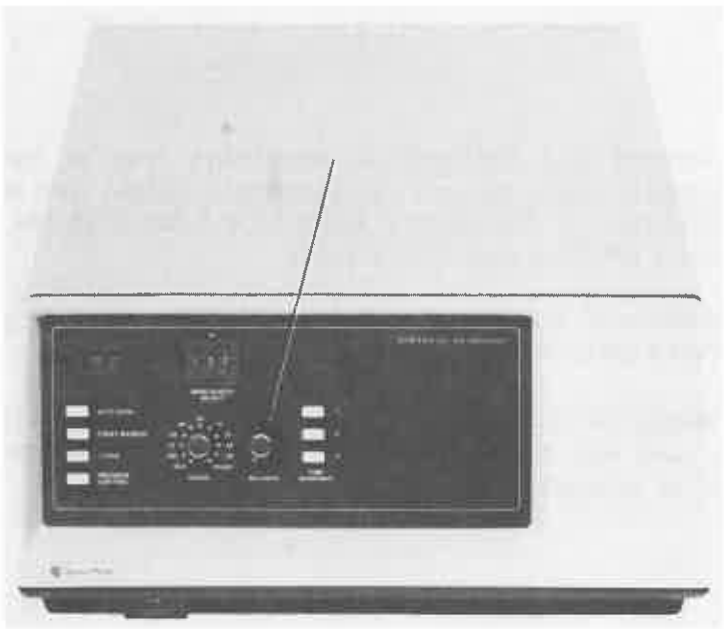


Figure 1.17 Balance Control.

1.5.6 Event Marker

Pressing the Event Marker button puts a negative (downward) going spike on the recorder. This is used to mark the start or ending of a chromatogram, or some other significant event.

1.5.7 Wavelength Select

Although any Wavelength between 000 and 799 may be set on the Wavelength Select thumbwheel switches, internal electronic limits will stop the travel at 150 and 600 nm. Because of the deuterium lamp spectrum, only 190 through 600 nm have sufficient intensity to be useful.

1.5.8 Time Constants

The time constant is a measure of the fastest response the detector will make to the rise and fall of a peak. The advantage of a greater time constant (slower response) lies in the elimination of baseline noise at highly sensitive Range settings. The SP8440 provides 3 time constant buttons for selecting any of 3 time constant periods, 0.1 second) button 1), 0.5 second) button 2), or 1 second) button 3). The buttons are interlocking. That is, when one is pressed in, the other two "pop out". However, it is possible to have all 3 pop out, if one is pressed only very lightly, in which case the 1 second time constant is active.

Selection of a time constant is dependent on the width of the fastest peak of interest (usually the first peak of interest in an analysis). To make intelligent use of the time constant feature, certain consequences of any time constant should be understood.

- The peak height will be slightly reduced. This effect is less than 1% if the half-height peak width is 20 times the time constant.
- The peak will be slightly delayed in apparent retention time. This is a function of the detector, not the column. The greater the time constant, the more the delay. This delay is about the magnitude of the time constant.
- The peak will be broadened. In other words, the peak width will become greater.
- The peak will tend to tail at the end, rather than come down sharply. This effect is not great unless the time constant approaches the value of the peak width.
- The peak area will be conserved. The loss in peak height is exactly corrected by the broadening of the peak. This certainly works in practice when a computing integrator is used as the data system. The old analog integrators might not perform as well because of the peak detection mechanism.

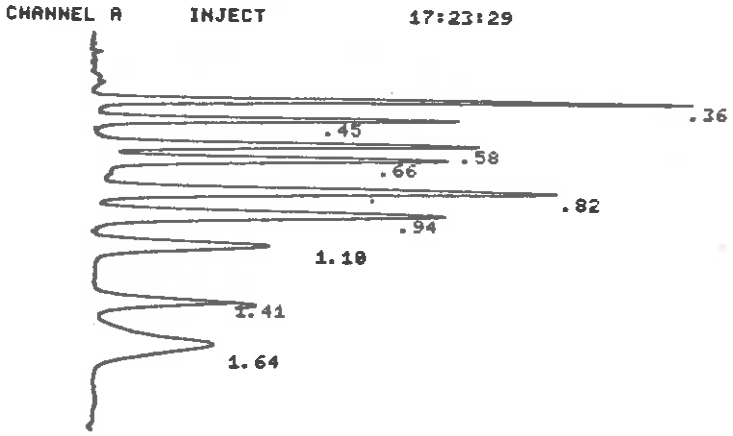


Figure 1.18 Analysis at 0.1 Second Time Constant.

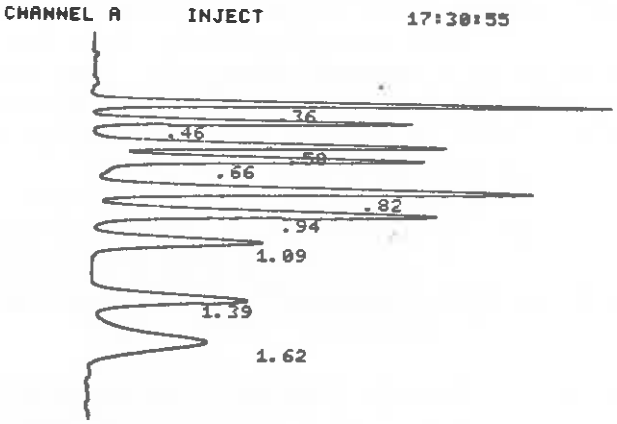


Figure 1.19 Analysis at 0.5 Second Time Constant.

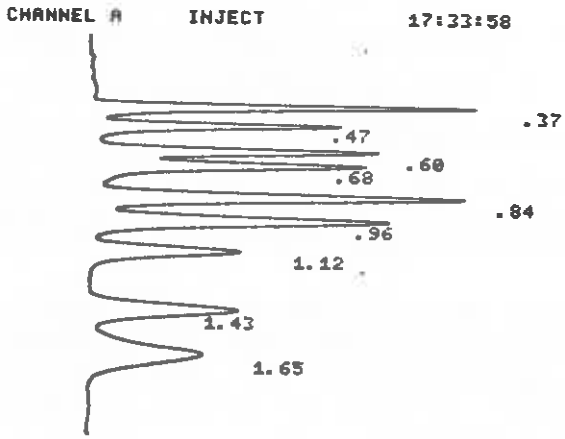


Figure 1.20 Analysis at 1.0 Second Time Constant.

For example: if the fastest peak of interest were 1.5 seconds wide at half-height, then a 0.1 second time constant would be acceptable. (One-tenth of 1.5 seconds is 0.15 seconds. The 0.1 second time constant is LESS than 0.15 seconds.) A 0.5 second or 1 second time constant, on the other hand, would be less acceptable, but probably satisfactory in most instances. This is illustrated by the examples in Figures 1.18, 1.19 and 1.20 in which the effects of time constant on the fast analyses of 9 aldehyde derevatives are compared. Only the time constant parameter has been changed from one figure to the next.

Note the reduction of the peak height of the early peaks, and in particular the first peak. Also, the separation of peaks 3 and 4 has deteriorated as a greater Time Constant is selected as shown by the valley between them moving up from baseline.

However, the retention times and the peak areas changed very little. Note that the later peaks are virtually unaffected by the changes in Time Constant. This is to be expected since these later peaks (7, 8, and 9) are much broader than the Time Constants.

Changes of time constant during an analysis should be done only during times when the baseline is stable and at zero. Otherwise, an upset will be seen by the data system. Usually the time constant is selected before the analysis, and not changed during the run.