Spectra-Physics

SP6040

Differential Refractometer
Operating and Service Manual

Part Number A0099–127

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LIST OF ILLUSTRATIONS

WARRANTY STATEMENT FOR AUTOLAB DIVISION PRODUCTS

WARRANTY COVERAGE

Spectra-Physics warrants that its products shall be free of defects in material and workmanship for 90 days from date of shipment, subject to the warranty coverage terms specified in Table 1. Spectra-Physics will, at its option, repair or replace defective instruments or components. The necessity of an on-site visit shall be solely at the discretion of Spectra-Physics. If on-site service work is performed by SP at the Customer's request but deemed unnecessary by Spectra-Physics, labor and travel expenses will be at the Customer's expense. Parts will be covered by warranty. Products that are installed by Spectra-Physics are warranted from the installation date, all others from the ship date.

If Customer schedules or delays installation by Spectra-Physics more than 30 days after delivery, then warranty period starts on the 31st day from date of shipment (60 and 61 days respectively for products shipped internationally).

SP warrants that its software and firmware designed by SP for use with an SP analytical instrument will execute its programming instructions when properly installed on that instrument. SP does not warrant that the operation of the instrument, or software, or firmware will be uninterrupted or error free.

After expiration of the applicable warranty period, Customer has the option to purchase a Customer Service Agreement or receive service at the then prevailing SP rates. Any obligation SP may have to provide service and/or parts for discontinued products shall cease five (5) years after the manufacture of such products has been discontinued.

Certain accessories, including some not manufactured by SP, may be warranted by SP for the specific items and periods expressed in writing on SP warranty statements, published price lists, and/or individual quotations. All such accessory warranties extended by SP are limited in accordance with all the terms, conditions, and other provisions stated in this warranty except as specifically extended by SP in writing. SP makes no warranty whatsoever concerning products or accessories not of its manufacture, except as noted above.

REPLACEMENTS AND MODIFICATIONS

In-warranty repaired or replacement parts or products are warranted only for the remaining unexpired portion of the original warranty period applicable to the repaired or replaced parts or products. Repair or replacement of products or parts under warranty does not extend the original warranty period.

The refractive index monitor is a widely used and universal detector for liquid chromatography. The SP6040 differential refractometer directly measures the deflection of a light beam due to the difference in refractive index between the sample and reference liquids.

The SP6040 is designed for the convenience of the user, with improved optical and temperature control systems.

Important features of this detector are:

1) Symmetrically designed detector.

2) Long focal length lens (150mm) for high sensitivity.

3) Small volume (10 microliter) flow cell.

4) Specially manufactured photo diode.

5) Heat exchanger that reduces temperature and flow effects by equilibrating the temperature of the sample and reference flows.

Full scale deflection is equivalent to 1×10^{-6} refractive index units.

Noise level is less than $\pm 1 \times 10^{-8}$ refractive index units under static conditions.

This manual provides the user with information necessary to the operation and maintenance of the detector. The manual should be read before operating the instrument. For further assistance, please contact your Spectra-Physics Representative.

NOTE
The detector is shipped with isopropanol in the lines.

1. Refractive Index Range	1.0 - 1.8 RIU.
2. Attenuator Range	1 to 512 x 10^{-6} RIU/full scale. (10mv) 10 binary steps.
3. Cell Volume	10 microliters
4. Cell Materials	316 S.S., PTFE, Quartz
5. Maximum Safe Cell Pressure	70 p.s.i. (4.9 Bars)
6. Cell Inlet	0.25mm I.D., 80 ul
7. Cell Outlet	0.6mm I.D., 170 ul
8. Focal Length	150mm
9. Noise	±1 x 10^{-8} RIU, Water in flow cell (static).
10. Drift	Less than 8 x 10^{-7} RIU/Hour, heater on after 90 min. equilibration. Less than 6 x 10^{-6} RIU/Hour with heater off and water in flow cell (static).
11. Linearity	Linear over all ranges
12. Temperature Control	Ambient - 60° C (Internally Adjustable)
13. Signal Output	10mV Recorder (Spade Lug) 0 - 10V Integrator (14 Pin Connector, adjustable).
14. Event Marker	1mV Fixed
15. Polarity Switch	+, -

16. General

Voltage: 100, 115, 230 VAC, ±10%, 50/60Hz Selectable; 135 VA
Weight: 18 kg

Dimensions:	Width	Depth	Height
Optical Unit	20cm	40cm	16cm
Control Unit	20cm	40cm	16cm

Figure 1. SP6040 Optical Unit, Front View

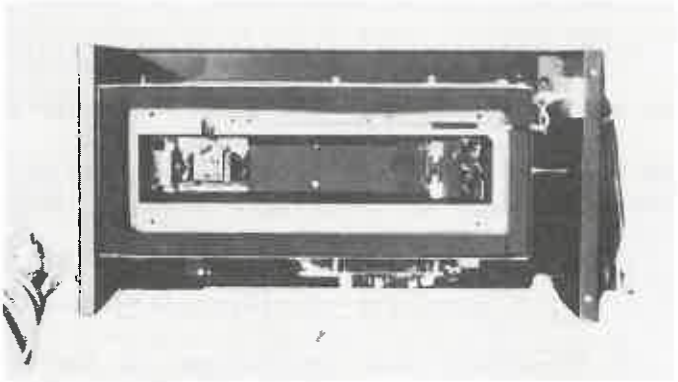


Figure 2. SP6040 Optical Unit, Interior View

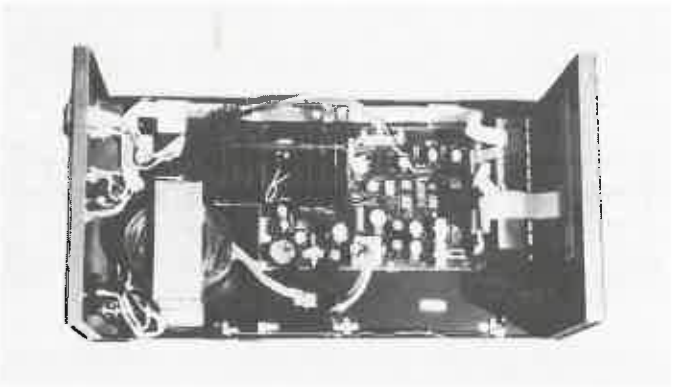


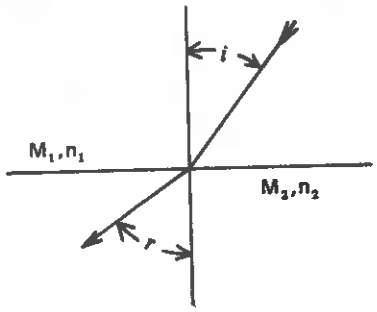
Figure 3. SP6040 Electrical Unit, Interior View

4.1 THEORY OF DEFLECTION REFRACTOMETRY

Refraction is a term applied to the bending of a light beam as it passes from one material into another. Deflection refractometry measures this effect.

Snell's law states that the ratio of the refractive indices equals the ratio of the sine of the angle of incidence to the sine of the angle of refraction.

A ray of light passing from n_1 and then obliquely into n_2 may be expressed mathematically in terms of the angles as follows:



$$\sin i/\sin r = n_2/n_1 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where i is the angle of incidence and r is the angle of refraction

The refractive index of the mixture equals the sum of the weighted products of each component by the simple mixture law, and is expressed as follows:

$$n \text{ soln} = C_1 n_1 + C_2 n_2 + \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

where C_1, C_2 etc. represent mole percentages of the component and n_1, n_2 etc. the corresponding refractive indices. To the extent that the mixtures deviate from ideal mixtures, this deviation will appear in the total refraction.

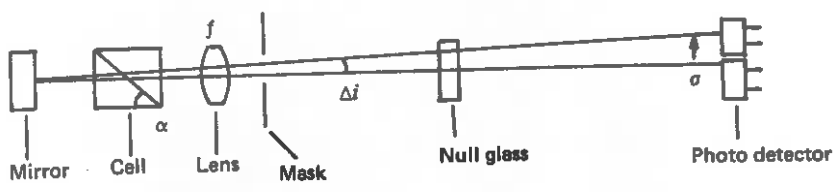


Figure 4. Construction of a Differential Refractometer

The deviation of the light beam depends upon the relative refractive indices of the sample and reference. When the difference in refractive index between the two media is small, the deviation of the light beam (δ), is given by Snell's law.

$$\delta = 2f\ (\tan\alpha).\ \triangle n \qquad (3)$$

where δ is the deviation of a light beam, f is the focal length of a lens, α is the cell angle and $\triangle n$ is $n_1 - n_2$

These equations interrelate the difference of refractive index between the two cells, the deflection of the beam, and the changes in density of the liquids in the cell.

4.2 CONSTRUCTION OF THE OPTICAL SYSTEM

To minimize the effect of fluctuations in ambient temperature, the main optical parts are mounted on a stainless steel optical bench (thickness: 10 mm), which is installed in an aluminum block functioning as a heat exchanger.

The aluminum block is maintained at a constant temperature by the thermistor controlled heater. Figure 5 shows the schematic design of the optical system.

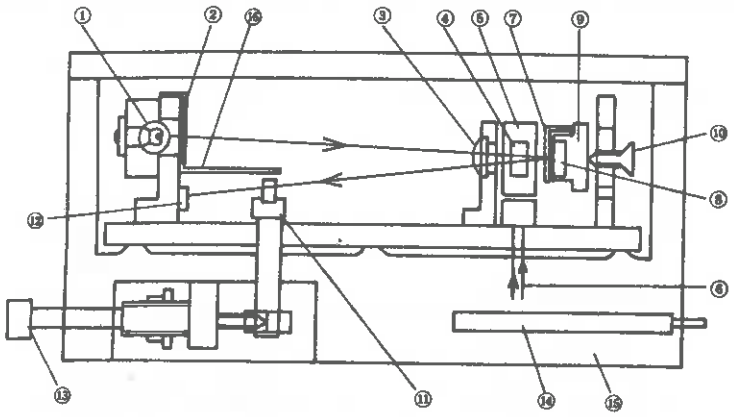


Figure 5. Schematic Diagram of Optical System

The light beam emitted by the tungsten lamp (1) passes through the slit (2) that confines the beam within the region of the flow cell.

A prismatic cell is held at a 45° angle in the cell holder (5), which has inlet and outlet tubes. The volume of the cell is 10ul and volume of the heat exchanger inlet tube is 80ul. The cell holder is positioned by the cell base plate mounted on the optical bench.

A lens (3) is attached to the optical bench to collimate the optical beam. The focal length of the lens is 150 mm.

This beam passes through the flow cell to the back mirror (8), which is covered by the mask (7).

The mirror is adjusted about the vertical and horizontal axes by four screws (10) and reflects the beam back through the flow cell to the lens, which then focuses it on the photo diode (12). At this point, the location of the light beam depends on the relative refractive index of the sample and the reference.

The photo diode is a specially manufactured silicon dual sensor (distance: 100 um) which accurately measures the position of the optical beam on the photo detector surface. An electrical signal is generated that is proportional to the difference between the positions of the two beams.

The aluminum block (15) is maintained at a constant temperature by the heater (14) and thermistor.

The heat exchanger (6) reduces the flow effects of the refractometer by equilibrating temperature of the sample and reference stream to that of the flow cell. The heat exchanger consists of the 1/16" O.D. (0.25 mm I.D.) stainless steel tube. The shading plate (16) is used to keep out stray light.

The optical zero glass (11) provides a method of deflecting the beam from side to side. This glass (see Figure 6) is a piece of BK7 (n = 1.5168) which is rotated around an axis through its center.

The function of this glass is to deflect the beam as the glass is rotated. The image on the detector moves horizontally as the glass is rotated, which provides an accurate means of adjusting the image location on the detector.

The deviation of the light beam (δ) is then given by

$$\delta = d.\sin\theta\left(1 - \frac{\cos\theta}{\sqrt{ng^2 - \sin^2}}\right) \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4)$$

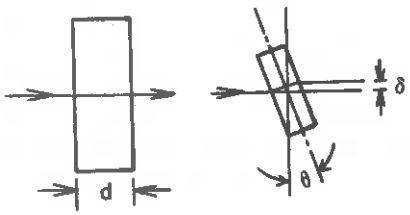


Figure 6. Schematic of Optical Zero Glass

where d = 3mm thickness of plate.

The optical zero glass is rotated with gears and a tangent bar mechanism by the balance knob (13). The balance control can be used as a secondary standard for calibration purposes. The deviation of light beam, δ , is given by equation (3). One turn of the balance control corresponds to a refractive index change of 3.4×10^{-5} RIU.

4.3 CONSTRUCTION OF THE ELECTRICAL CIRCUIT

The electronics provide a stable lamp supply voltage, to control temperature of the equipment, to amplify the signal generated by the photo detector, to amplify the signal of the difference and to display the optical balance.

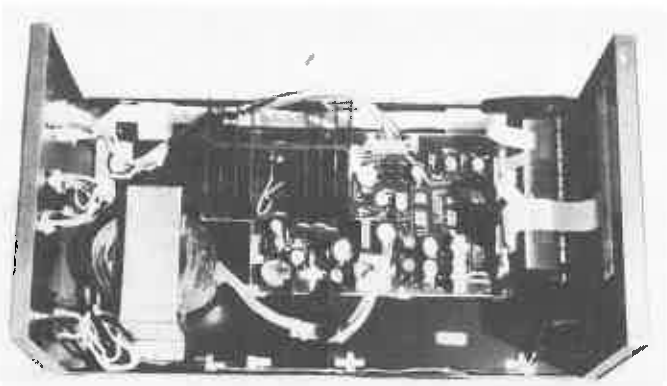


Figure 7. SP6040 Electronics Board

Figure 8 shows the schematic diagram of the lamp supply voltage regulator. The lamp supply voltage regulator is a switching regulator. The output voltage can be adjusted 2 to 5 volts by a variable resistance (5 kilohms VR). The standard rating of the lamp is 5 volts, 0.3 ampere. In this detector, normal operating condition is about 3 volts, ensuring extended lamplife.

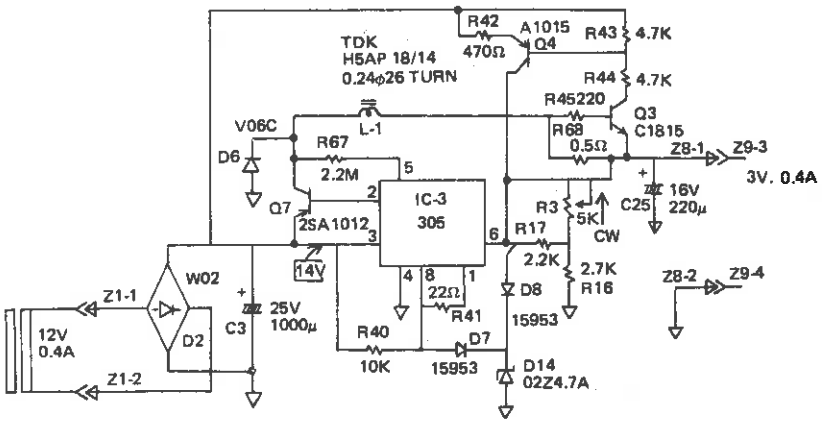


Figure 8. Schematic, Lamp Power Supply Voltage Regulator

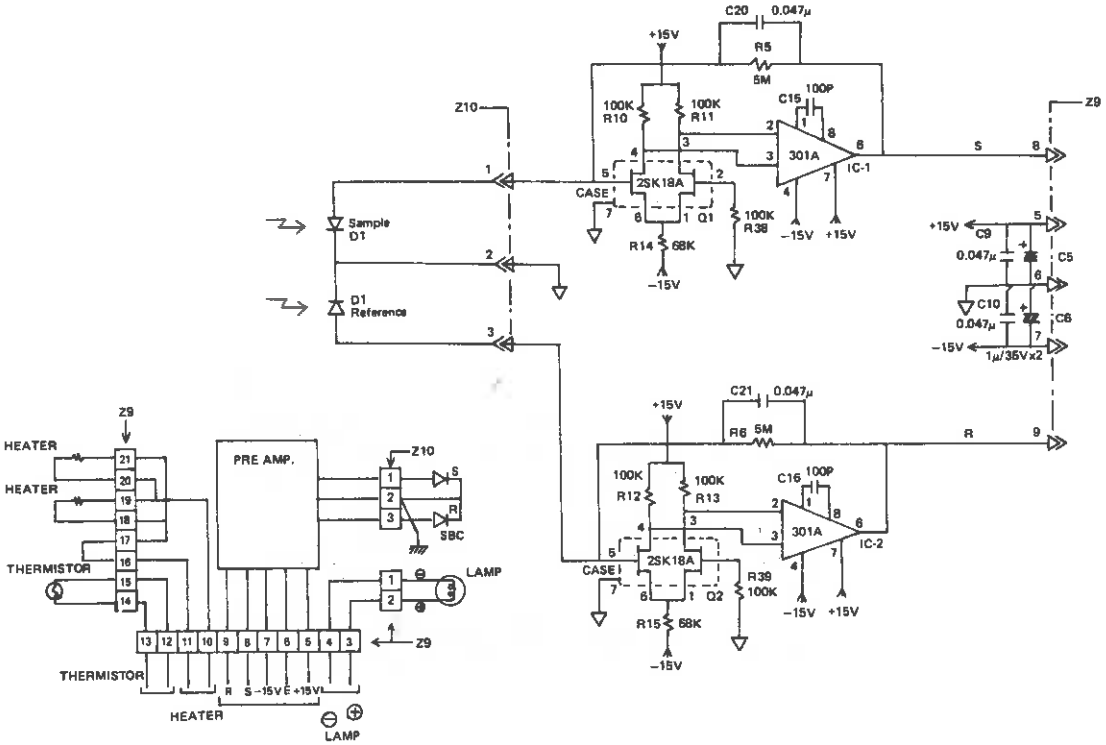


Figure 9. Schematic Diagram, Preamplifier

The Preamplifier consists of two operational amplifiers connected to the dual photo diode detector in the optics block. The amplifier is powered by the ±15V supply. Output of the preamplifier is -0.15V under optimum conditions.

The terminals on the preamp board are designated:

Z10; 1) Sample side photodiode 2) Ground
3) Reference side photodiode

Z9 1) Lamp out (-) 2) Lamp out (+) 3) Lamp in (+) 4) Lamp in (-)
5) +15V 6) Ground 7) -15V 8) Output (S)
9) Output (R) 10) Heater input 11) Heater input
12 through 15) Thermistor 16, 17) Heater Fuse
18 through 21) Heater outputs

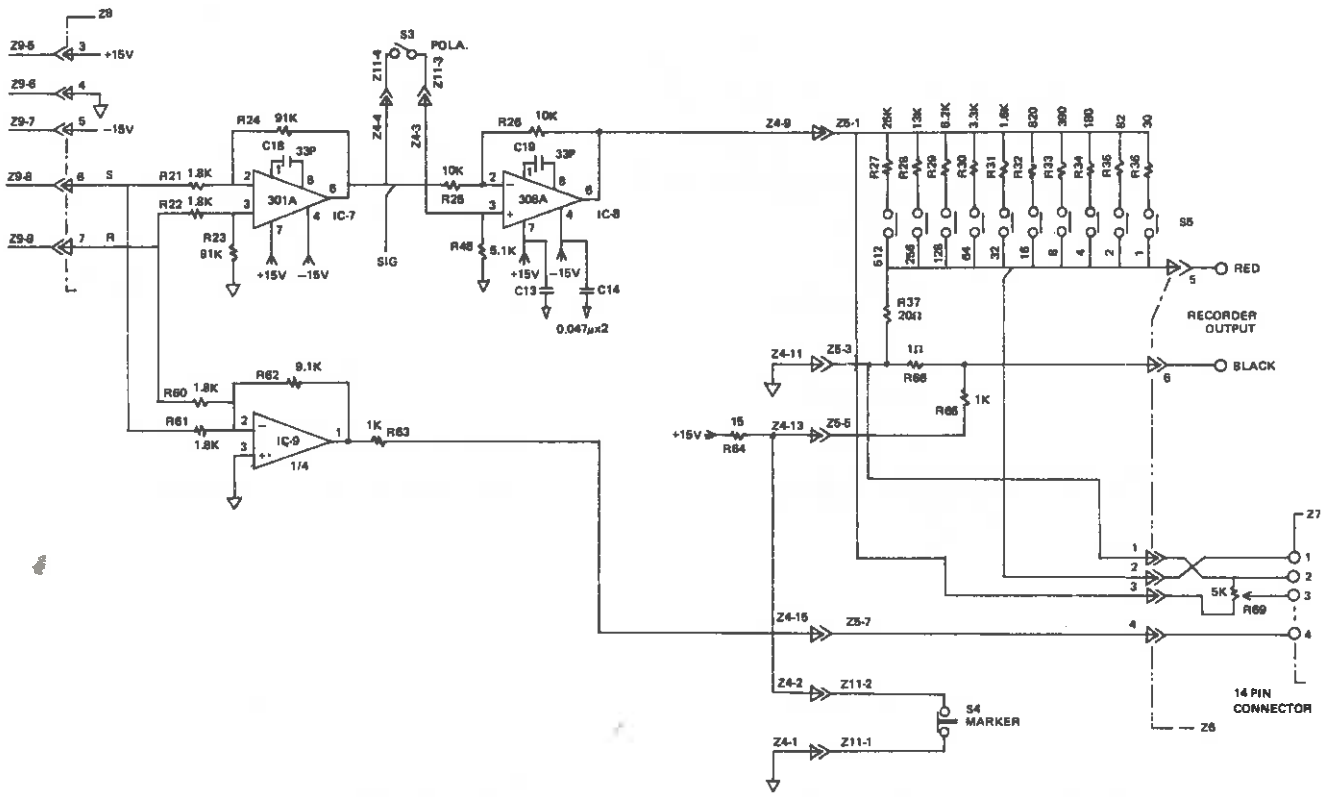


Figure 10. Schematic Diagram of Main Amplifier

The main amplifier amplifies the difference between the preamp signals. The output voltage of this comparator passes through the polarity amplifier and the input attenuator (front panel Range Control).

Two outputs are provided: 10mV for a recorder and 0-10V adjustable output for an integrator. The red terminal is plus and the black terminal is minus for the 10mV output. The Z7 connector on the back panel provides recorder, integrator and test outputs.

Z7, pin 3 output is the integrator output referenced to Z7 pin 2. Z7, pin 4 is a summed signal from the reference and sample preamp. It can be used for checking source lamp life or flow cell contamination.

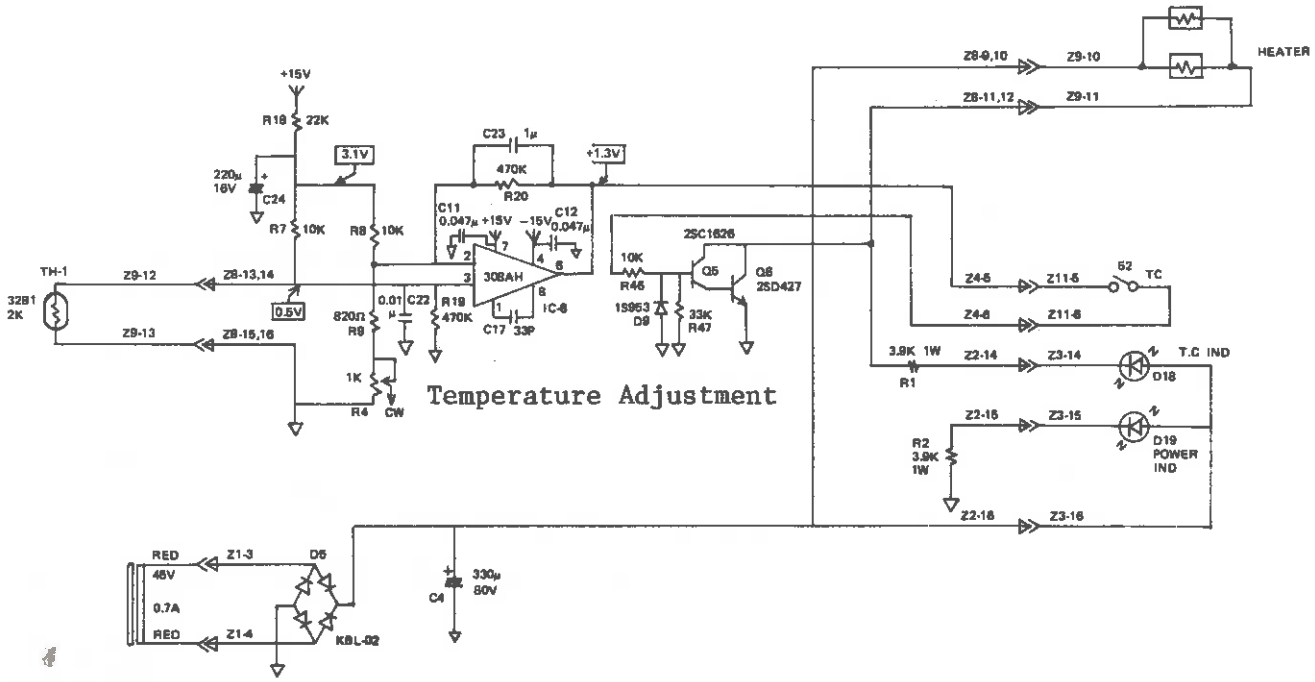


Figure 11. Schematic Diagram, Temperature Control Circuit

Temperature is controlled by monitoring a thermistor in a resistive bridge. Any negative temperature offset from set point will cause power transistor (2SP427) to conduct, turning on the heaters. The set temperature is adjusted approximately 10 to 35° above ambient by the 1 kilohm potentiometer on the electronics board.

Figure 12 shows the diagram of the power supply (±15 volt). This circuit provides the fundamental regulation against line voltage change, thus allowing the equipment to operate over a wide range of supply voltages.

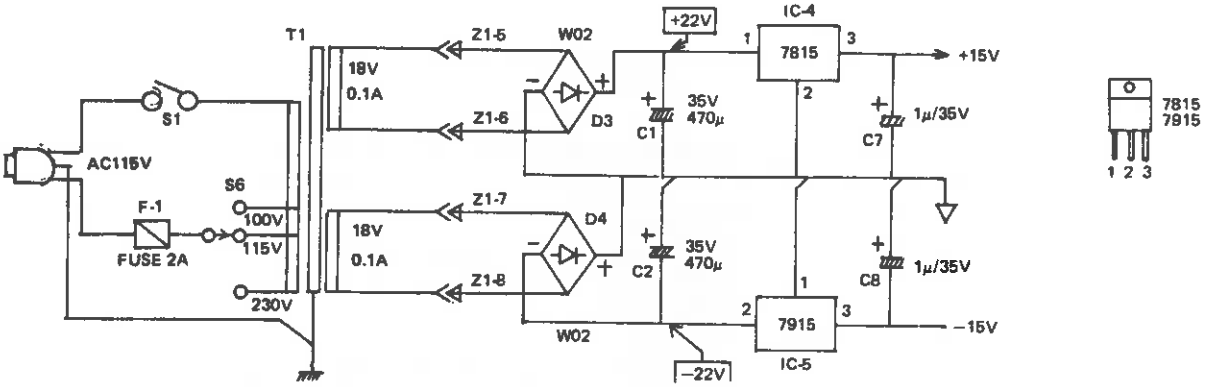


Figure 12. Power Supply Diagram.

Figure 13 shows the balance control circuit diagram. The balance signal is taken from the main amplifier comparator. An offset of ±1 volt causes a balance display offset of ± 1 step.

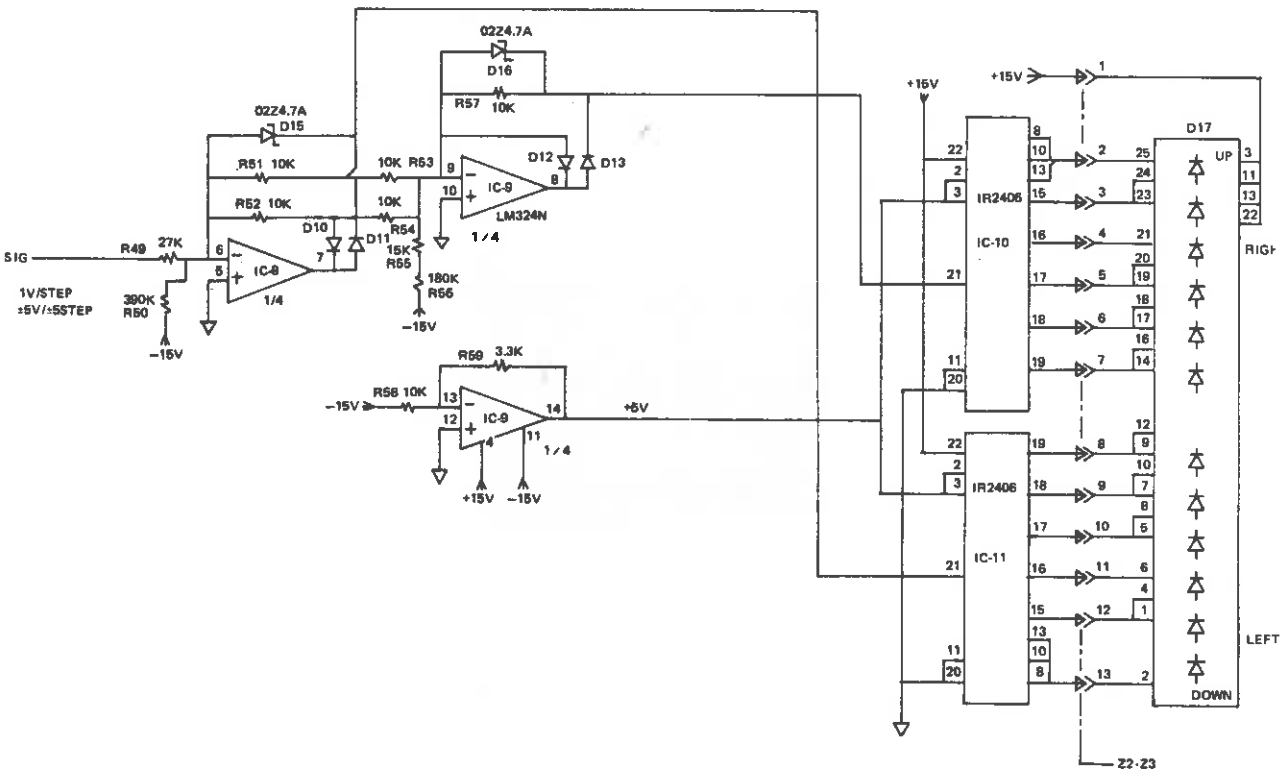


Figure 13. Balance Control Circuit Diagram.

4.4 FUNCTIONAL PARTS DESCRIPTION

This section describes the functional parts. Figure 14 shows a front view of the optical unit.

4.4.1 Balance Control

The balance control operates a null glass in the optical beam. This control is used for recorder baseline adjustments. Its adjustable range is 5.5 x 10^{-4} RIU. Usually, the optical zero is fixed in the center of the LED display. The LED display shows the offset from optical zero.

4.4.2 Flow Cell Outlets

There are outlet tubes from the reference and sample cells. To begin use, route the outlets to a drain.

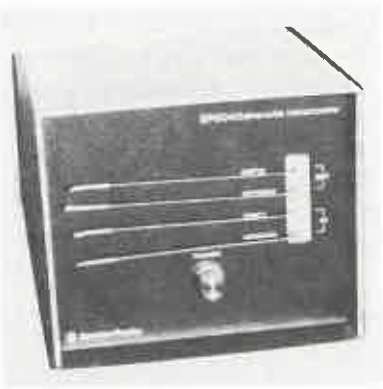


Figure 14. Front View of Optical Unit.

4.4.3 Flow Cell Inlets

There are inlet tubes for the reference and sample cells. To begin use, connect the sample inlet to the column outlet. The reference inlet can be with solvent flow or sealed for a trapped reference.

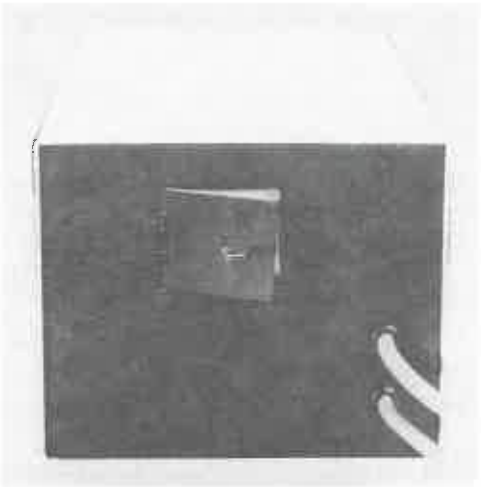


Figure 15. Rear view of optical unit.

4.4.4 Door for Mirror Adjustment

The four screws for mirror adjustment are accessed by opening the door at the rear of the optical unit. The mirror adjustment is a coarse adjustment of the position of the beam on the photo detector. The right and left side screws are used for the adjustment of the beam towards the right or left position, while the upper and lower screws are used for the adjustment of the beam towards the upper or lower position.

These adjustments are not necessary in normal operation because the optical zero adjustment is sufficient.

> **NOTE**
>
> The vertical adjustments should not be used except by a qualified service representative.

4.4.5 Connection Cable (5 Pin)

This cable connects the electrical unit to the optical units. Cable consists of ±15V and pre-amplifier signal.

4.4.6 Connection Cable (7 Pin)

This cable connects the electrical unit to the lamp, heater and thermistor in the optical unit.

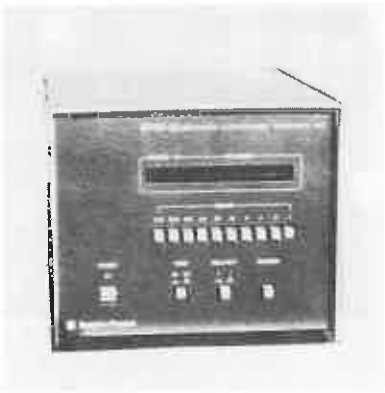


Figure 16. Front view of Electrical Unit.

4.4.7 Display of Optical Balance

The LED (12 segment) is lined across the front panel. When the optical beam is balanced, the two segments in the center position are lit. When the cell is out of balance, the left or right LEDs are lit.

4.4.8 Range Selection Switch

The switch has 10 steps from 1 x to 512 x (1 x corresponds to a maximum sensitivity of 1×10^{-6} RIU/full scale for a 10 mV output).

4.4.9 Event Marker

The event marker switch can be used at the time of the sample injection. The chart is marked by adding a 1mV signal to the recorder output signal.

4.4.10 Polarity

The polarity switch is used to reverse the output signal to the recorder. If the refractive index of the sample is larger than that of the solvent, the polarity switch should be set to plus (+), thus causing the signal to respond to the (+) side of the recorder or integrator.

4.4.11 Temperature Control

Refractive index is very sensitive to temperature variations (dn/dt about 10^{-4} RIU per °C). The optical bench of the SP6040 can be precisely thermostated by using the temperature control system. This allows the SP6040 to be used at its highest sensitivity.

The SP6040 can be operated at temperatures from ambient to 60°C. In normal operation, the optical bench temperature is controlled by the heater, which is set at the factory to between 35°C and 40°C.

With the temperature control on, and the detector at higher than ambient temperatures, the solvent should be degassed before use to eliminate degassing in the detector. It is possible to adjust the temperature control on the electrical board. Refer to the adjustments section of the manual.

4.4.12 Power Switch

The power switch is used to turn the SP6040 on and off.

Figure 17. Rear View of Electrical Unit.

4.4.13 Recorder/Integrator Output

The recorder cable is used to connect the SP6040 to a recorder or integrator. The red terminal is plus (+) and black is minus (-). The recorder output is 10 mV/full scale.

4.4.14 Fuse & Line Voltage Adjustment (Transformer)

The main fuse is 2 ampere for 115V, or 1 amp for 230V.

Line voltage is selected by the following procedure:

1. Unplug power cord from line voltage wall outlet. Unscrew the fuse holder and remove fuse.

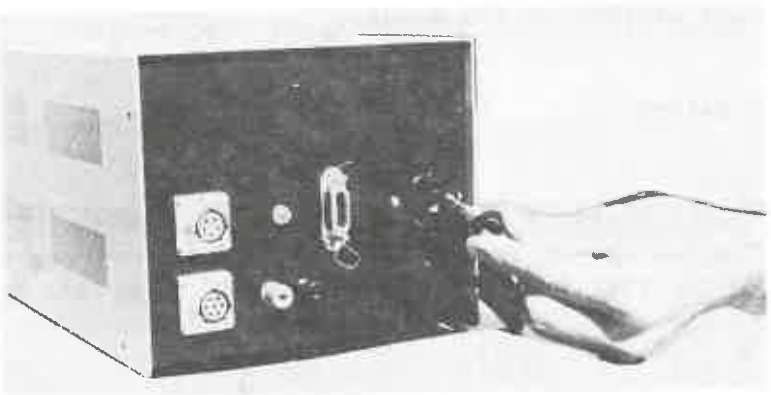


Figure 18. Location of Fuse and Holder.

2. Remove voltage changing plug as shown in Fig. 19.

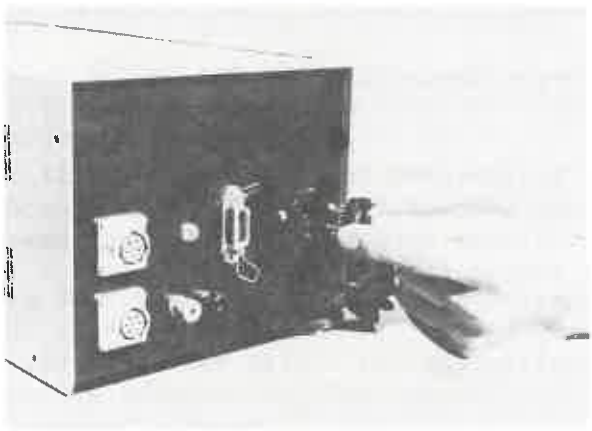


Figure 19. Removing Transformer.

3. Align the plug so that the desired line voltage indicator is visible. Push plug into place.

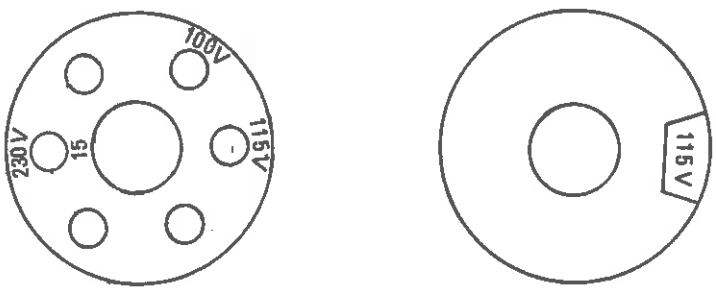


Figure 20. Various voltage options.

4. Reinsert fuse and tighten fuse holder.

4.4.15 Power Cable

The power cable provides electrical service. Make sure the power switch is off before inserting power cable into the wall outlet.

4.4.16 Multiple Output Connector

This terminal is provided for integrator signal output. Pin 1 is the signal after the attenuator switches, 2 is ground, 3 is the 0-10 volt adjustable integrator output. Pin 4 is the summed signal of the reference and sample pre-amp outputs.

4.4.17 Power Indicating LED

The power switch is provided with an LED. The LED glows when the power is being supplied to the refractometer.

4.4.18 Temperature Control Indicating LED

The temperature LED indicates the heating action of the temperature control unit. When the switch is pressed, the indicating LED goes on. After the optic block reaches the set temperature, usually within 90 minutes, the indicating LED will dim.