

SECTION 3

**INFRACORD
SPECTROPHOTOMETER
DETAILED THEORY
OF OPERATION**

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ENGLAND.

DETAILED THEORY OF OPERATION

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GENERAL

1. A thorough understanding of the operating principles will help the operator to use his instrument with maximum efficiency.

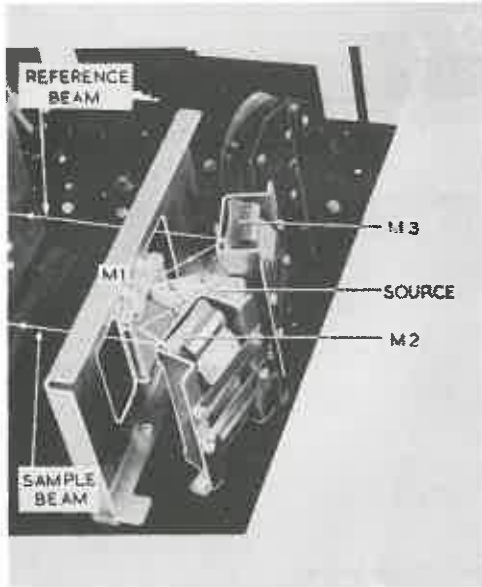


Fig. 3.1 Source Optics

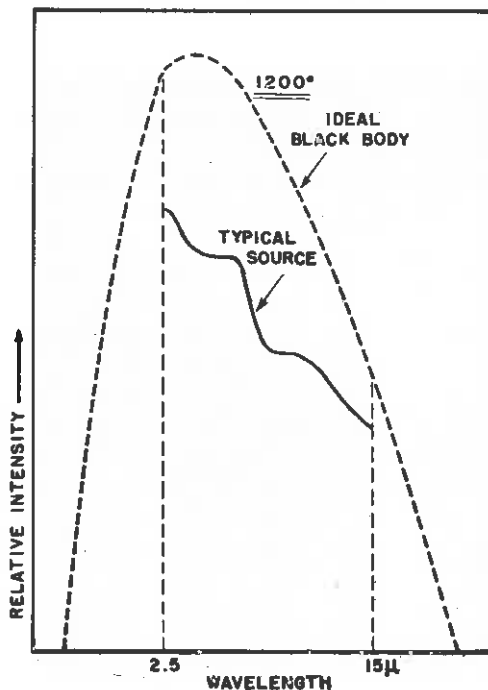


Fig. 3.2 Radiated Power Curves

SOURCE UNIT AND SAMPLE

2. The radiant energy in the sample and reference beams is emitted by a length of ceramic tubing heated by an internal metallic heater. The ceramic is heated to about 1200°C and produces a continuous spectrum of electromagnetic energy most of which falls within the infrared region from 2.5 to 15 μ . The incandescent portion of the tubing is about 15 mm. long and 3 mm. in diameter. It is largely enclosed by two heat shields to minimize the power required to operate it. The source has a positive temperature coefficient of resistance. No ballasting or water cooling system is needed.

3. So that the instrument will have a uniform response and noise level at all wavelengths, the energy available at the detector must be kept relatively constant over the entire wavelength range. Fig. 3.2 shows, however, that, even if the source were perfect (an ideal black body), at 1200°C the energy radiated would vary considerably over the wavelength range of 2.5 - 15 μ . The solid line in Fig. 3.2 shows the variations in relative intensity from a typical source.

4. Compensation for changes in the power radiated by the source and of the dispersion of the prism over the wavelength range is accomplished by programming the widths of the monochromator slits. At longer wavelengths the energy emitted from the source decreases and the slit widths must be increased. The energy transmitted by the monochromator is proportional to the square of the slit width. Fluctuations of the source intensity due to changes in line voltage are automatically cancelled by the double beam photometer system. It may be useful to note that an increase in the energy emitted from the source (especially at the shorter wavelengths) may be obtained by changing the transformer tapping to increase the voltage of the heater coil at the source. The increased temperature of the source will, however, shorten its life.

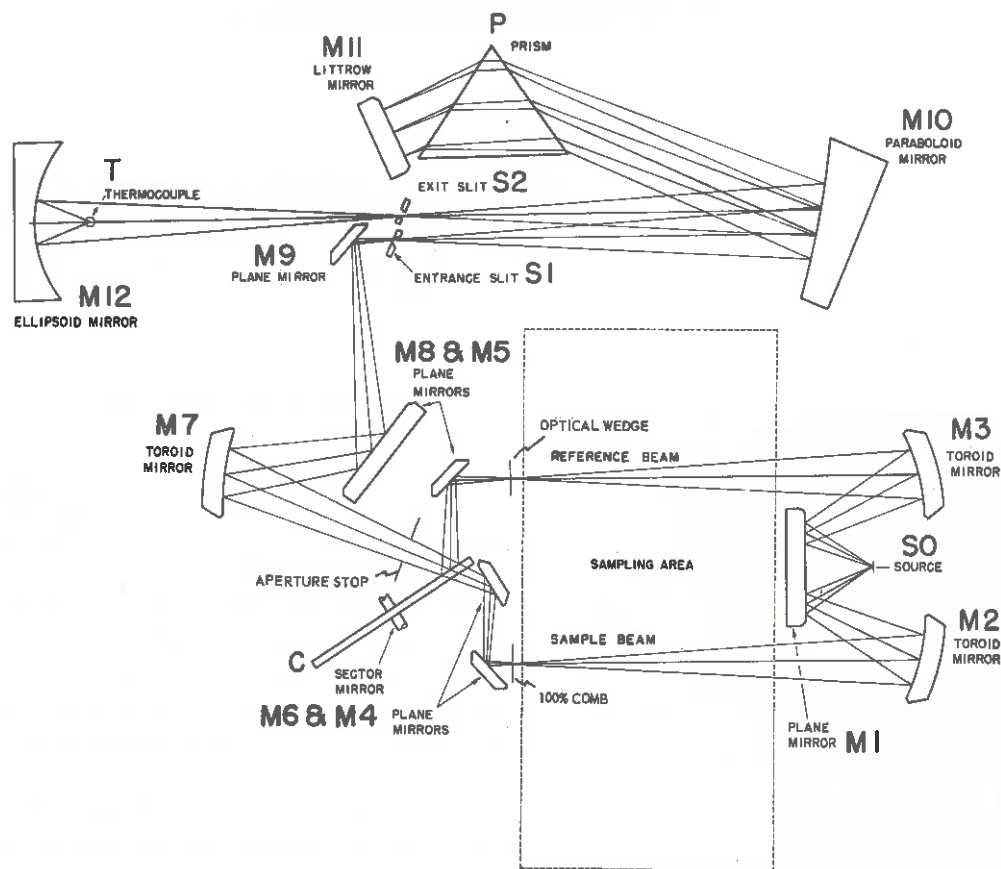


Fig. 3.3 Optics of Infracord Spectrophotometer

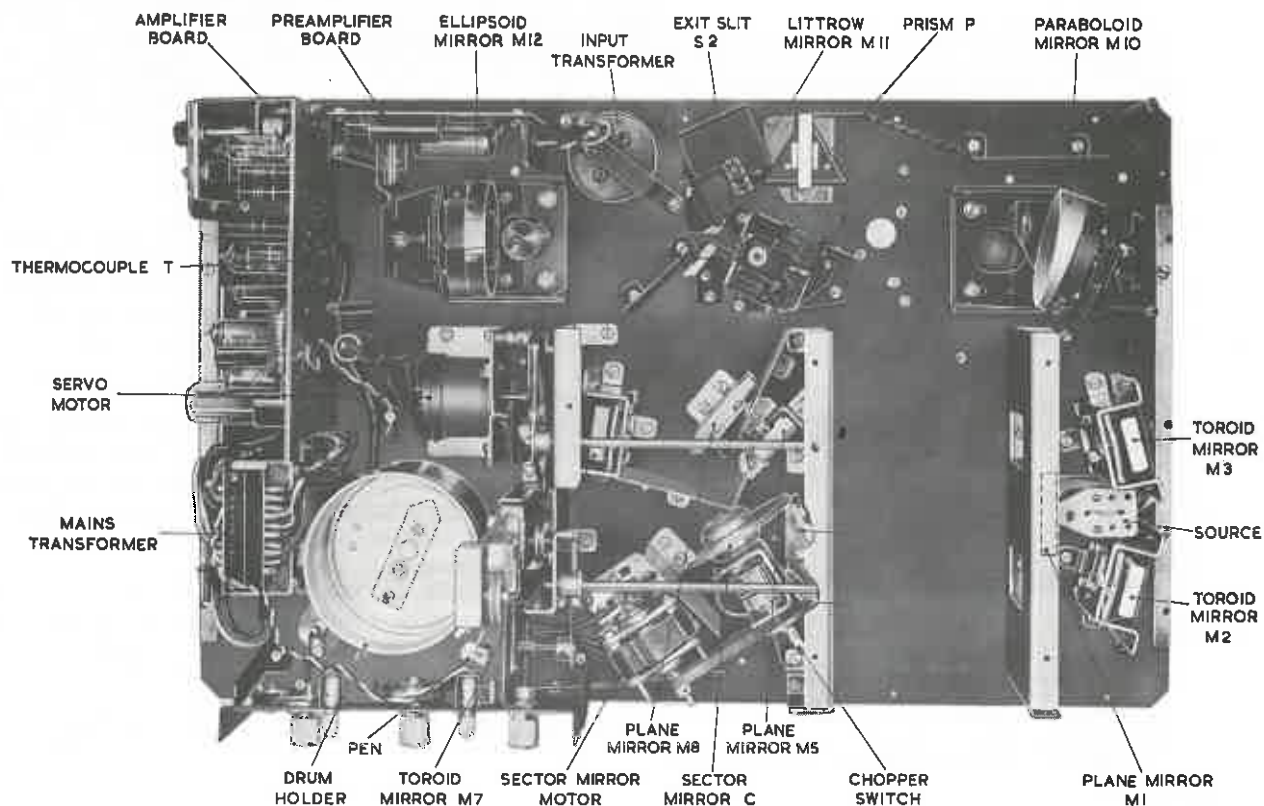


Fig.3.4 Component Layout

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5. As shown in Fig. 3.3 and 3.4, the energy radiated by the source is split into the sample and reference beams by the flat mirror M1 and toroidal mirrors M2 and M3. Toroidal mirror M2 focusses the sample beam on the 100% comb, and M3 focusses the reference beam on the optical wedge. To ensure that the optical system is filled uniformly, the image of the ceramic tube is double size, i.e. 6 mm. wide. The use of toroidal mirrors eliminates astigmatism in the source images of the two beams.
6. The sample is normally placed in the sample beam just in front of the 100% adjusting comb where it absorbs some of the radiated energy at its characteristic wavelengths.

PHOTOMETER

7. The photometer combines the sample and reference beams into a single beam consisting of alternate pulses of reference and sample beam radiation. If there is any difference in intensity between the sample and reference beams the combined beam will fluctuate in intensity. The magnitude of any such fluctuation will be proportional to the difference between the sample and reference beam intensities. The photometer also contains the mechanism whereby the signal is nulled (the sample and reference beams are equalised).
8. The sample and reference beams are combined by the semicircular sector mirror C, (shown in Fig. 3.3) which rotates at 11 revolutions per second. It can be seen that, with plane mirrors M4, M5 and M6 oriented as they are, the sector mirror will alternately reflect the reference beam and allow the sample beam to pass unimpeded through the aperture stop. The aperture stop, which is imaged on the Littrow mirror in the monochromator, ensures that both the sample and reference beams are the same size and follow identical paths through the remainder of the optical system.
9. When the intensities of the two beams are the same, the energy of the combined beam through the aperture stop is constant. Should the intensity of one beam differ from that of the other, the combined beam intensity would vary at the rate of 11 c/s and an alternating electronic signal would be generated at the detector. The phase of the electronic signal will depend on whether the sample is tending to absorb more or less of the incident energy.
10. The electronic signal is used to make the intensity of the reference beam equal to that of the sample beam. This is accomplished by using the signal to drive a servo motor which (1) moves an optical wedge into, or out of, the reference beam and (2) drives the pen.
11. The wedge is designed so that, as it is moved into the reference beam, the attenuation of the beam intensity is linearly proportional to the rotation of the wedge shaft. The wedge position thus becomes a direct measure of the relative intensities of the sample and reference beam, (the transmittance). Since the pen is directly coupled to the wedge, its movements are actually a record of the wedge position, or sample transmittance.
12. Because the sample and reference beams are combined by the sector mirror C, toroidal mirror M7 alternately "sees" images of the source at the wedge and at the 100% comb. It focusses these images on the entrance slit, S1, of the monochromator with no change in image size and no astigmatism.

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- Note 1. 100% Adjustment - The 100% Control is used to position the pen at 100% Transmittance when no sample is present. It consists of a manually operated comb which is similar to the wedge in both operation and appearance. The comb is located at a position in the sample beam corresponding to the wedge position in the reference beam. When the comb is moved into or out of the sample beam, the servo motor will drive the wedge in such a way as to equalise the beam intensities. This, of course, moves the pen and provides a means for setting the pen exactly at the 100% Transmittance mark when no sample is present.
- Note 2. The comb and wedge are constructed from very thin sheet metal in order to improve their optical performance and can be damaged very easily. Do not handle them. Do not poke fingers or objects into the apertures in the sample area.

MONOCHROMATOR

General

13. The monochromator performs three functions which are basic to the operation of the spectrophotometer:
- (a) It disperses the radiation into its component wavelengths.
 - (b) It selects the particular wavelength of radiation transmitted at the detector.
 - (c) It maintains approximately constant energy at the detector when no sample is in the system, regardless of wavelength.

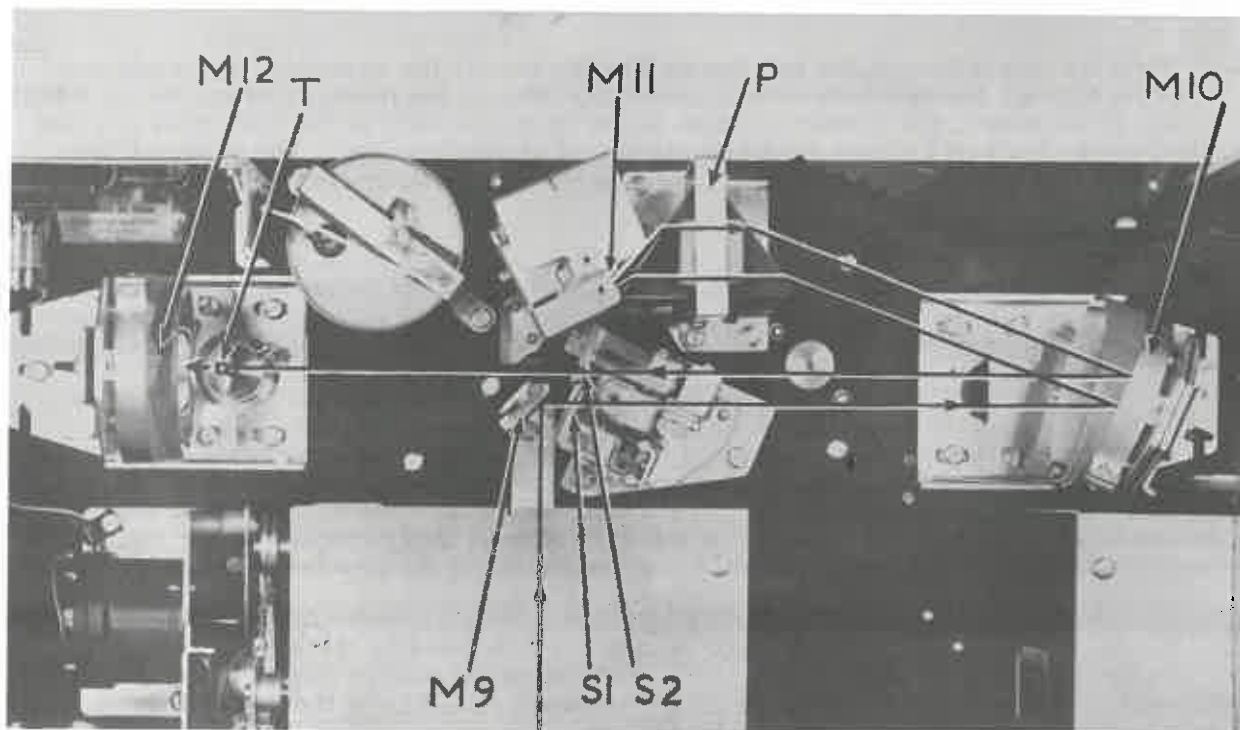


Fig. 3.5 Monochromator Optics

14. The monochromator consists of the entrance and exit slits S1 and S2, a 23° off-axis paraboloid mirror, M10, a 70° NaCl prism, P, and Littrow mirror, M11, as shown in Figs. 3.3, and 3.5. It is enclosed by a gasketed cover which protects the optical elements and keeps out dust, hence reducing scattered light. The cover also protects the prism against moisture and houses the detector and pre-amplifier. The prism is 55 mm. high and is 77 mm. across its base. The paraboloid mirror M10 has a focal length of 203 mm.

Dispersion into Wavelengths

15. The plane mirror M8 reflects the combined beam through the rock-salt entrance window in the monochromator cover onto mirror M9. This in turn reflects the beam onto the entrance slit S1 which is coincident with the focal point of mirror M7. From S1, the beam diverges until the off-axis paraboloid mirror M10 reflects it as collimated light (all rays parallel to each other) into prism P. At the prism, the component wavelengths of the beam are refracted, or bent, at angles that vary according to the particular wavelength. The emergent beam, now dispersed into a spectrum of infrared wavelengths, is reflected by the Littrow mirror back into the prism where it is further dispersed. It is then focussed on the plane of exit slit S2 by paraboloid M10 as a band of individual wavelengths falling across the slit (see Fig. 3.6).

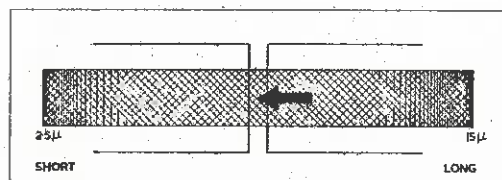


Fig. 3.6 Dispersed Radiation at Exit Slit

The variation is the result of two opposing effects - the increase in dispersion of the prism as the wavelength increases and the increase in mechanical slit width required to keep the energy constant.

CAUTION *The prism and entrance window of the monochromator are made of rock salt. Because this material is easily damaged by moisture, it is especially important to keep any water away from the monochromator section of the instrument. It should never be exposed to a relative humidity of greater than 60%.*

Selection of a Particular Wavelength

16. The particular wavelength which emerges from the exit slit is determined by the rotational position of the Littrow mirror relative to the prism. This mirror, rotated at a predetermined rate by the Littrow cam, moves the spectrum across the slit so that, roughly speaking, each wavelength from 2.5 to 15 microns is transmitted in sequence by the exit slit. Due to the finite slit width, it is more exact to say that a band of wavelengths emerges from the exit slit. The effective width of the wavelength interval emerging is termed the spectral slit width and varies with wavelength.

17. The rotation of the Littrow mirror is controlled by the Littrow cam on the recorder drum shaft (see Fig. 3.7). An arm with one end riding on the Littrow cam and the other attached to the Littrow mirror bearing, co-ordinates Littrow rotation with drum rotation. This ensures that the wavelength scale on the chart corresponds to the wavelength passing through the exit slit since no lag or backlash is possible.

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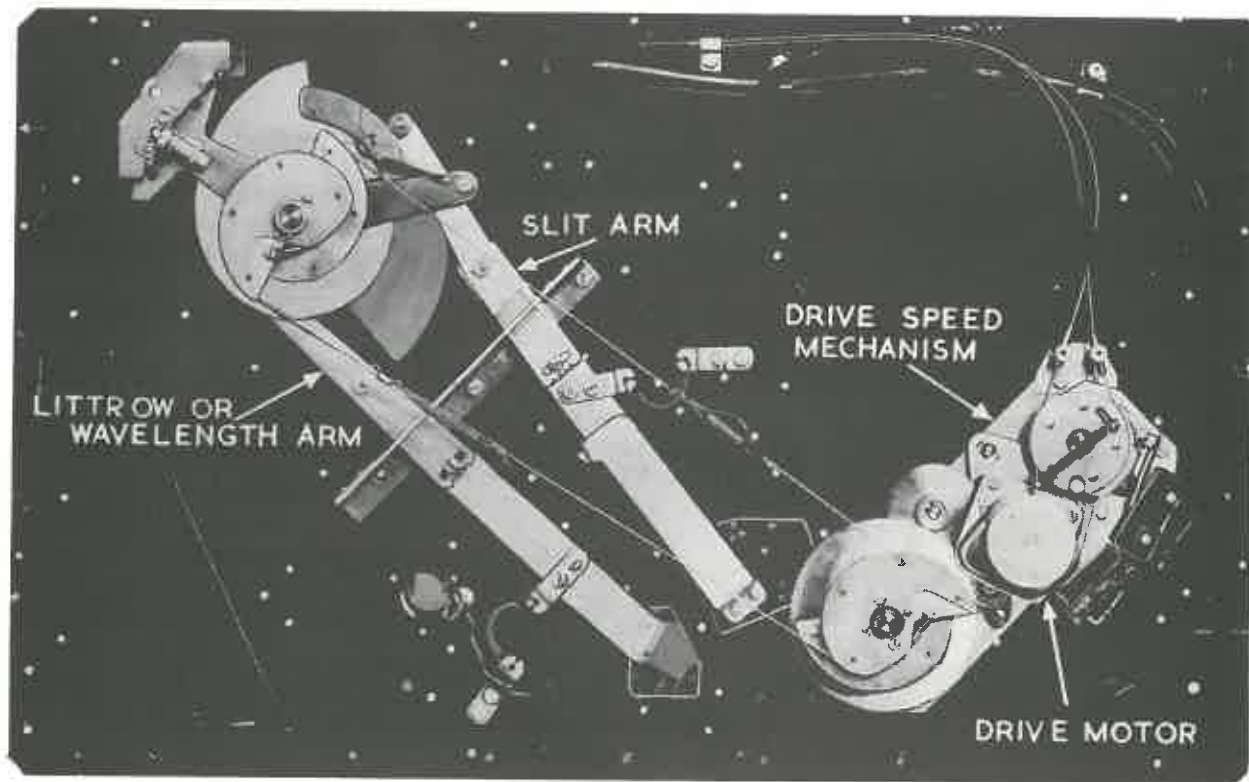


Fig. 3.7 Underside of Base Plate

18. Because temperature variations cause the refractive index of the prism to change slightly, the angular position of the Littrow must be corrected in order to maintain a true correlation between the chart wavelength scale and the wavelength transmitted by the monochromator exit slit. An accurately positioned bimetal strip on the Littrow mount is provided to achieve this effect.

Entrance and Exit Slits

19. The entrance and exit slits consist of pairs of accurately machined metal jaws which are opened and closed in a carefully controlled manner. Both jaws of each slit move and the two pairs are ganged so that they operate simultaneously. The jaws of the entrance slit, S1, are curved and this curvature is exactly the reverse of the curvature introduced in the slit image by the prism and paraboloid mirror. The image falling on the exit slit, S2, therefore, is straight.

20. As mentioned in paragraph 4, in order to keep the energy level at the detector constant throughout the wavelength range it is necessary to vary the mechanical slit width. This is determined by the position of an arm (Slit Arm in Fig. 3.7), the free end of which rides on a ratio arm, which in turn rides on the slit cam. Since the slit cam is rigidly fixed to the recorder drum spindle, its position, and consequently the mechanical slit width, is directly related to the rotation of the drum. The profile of the slit cam is designed to lift the ratio arm, and with it the slit arm, so that the slits are at the correct width for constant energy at all wavelengths.

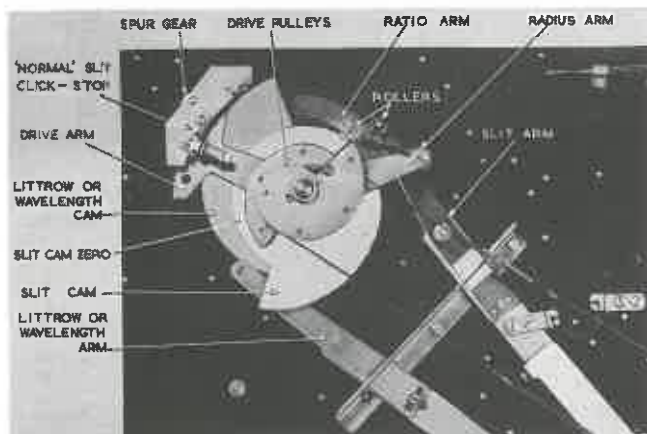


Fig. 3.8 Drive & Slit Assemblies (Narrow)

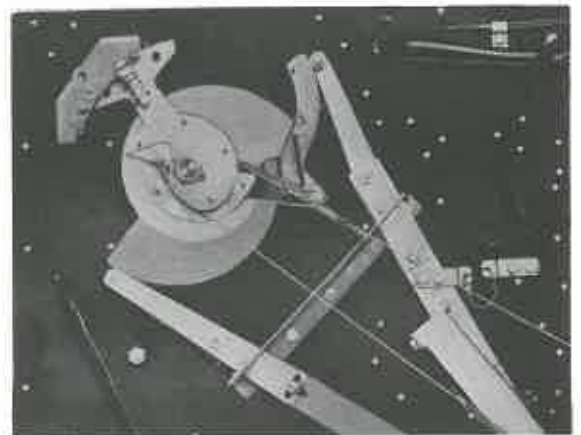


Fig. 3.9. Drive & Slit Assemblies (Wide)

Slit Control

NOTE: This section should be read in conjunction with paragraph 15 of the OPERATING INSTRUCTIONS which describes the function of the SLIT control.

21. Two factors are closely related to the mechanical slit widths, firstly the level of energy reaching the detector and secondly, the bandwidth of the dispersed radiation reaching the detector. As previously explained, an operator may wish to select high energy or high resolution by operation of the SLIT control on the instrument front panel. The SLIT control is connected via a flexible drive cable to the spur gear shown in Fig. 3.8. The spur gear and drive-arm operate as a rack and pinion to set the radius arm position such that the ratio arm provides a different slit programme for each setting of the SLIT control. Figure 3.8 shows the positions of the arms when the SLIT control is set to the NARROW position and Figure 3.9 shows their positions when the control is set to WIDE. The spring catch on the drive-arm is limited by stops at the extreme settings of the SLIT control to prevent the control from being adjusted beyond its limits. At the normal (N) setting a notch in the spring catch engages with a small pillar to ensure an accurately reproducible setting.

DETECTOR

22. The function of the detector is to convert the radiant energy from the source into electrical energy that can be used to drive the optical wedge and the pen. The unit consists of the ellipsoidal mirror M12 and the thermocouple T (see Fig. 3.10). The two foci of the ellipsoid are located at the exit slit of the monochromator and the sensitive area of the thermocouple, the ratio of the focal lengths being 6:1. The radiant energy leaving the exit slit is, therefore, focussed on the thermocouple as an image

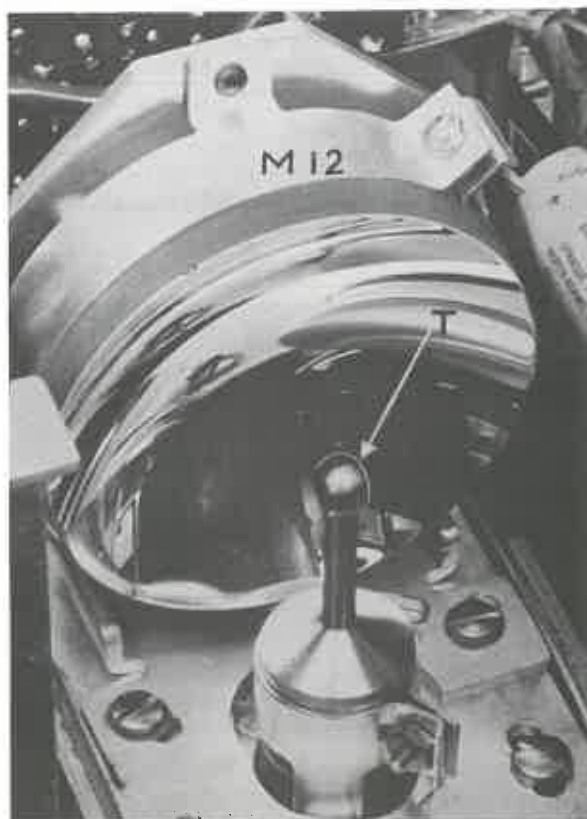


Fig. 3.10 The Detector

of the slit reduced in linear dimensions by a factor of six. The signal from the thermocouple for a full scale error (one beam at full intensity, the other beam zero) is about $0.2 \mu\text{V}$ r.m.s. at 11 c/s.

23. The high speed, high sensitivity thermocouple is a delicate unit. Its construction is shown schematically in Fig.3.11. The target on which the ellipsoid, M12, focusses is a $2 \times 0.2 \text{ mm}$. strip of blackened gold leaf welded to two pins. The blackened gold leaf is an excellent absorber of radiant energy and, due to the heating effect of infrared radiation, indicates the amount of incident energy, J , by its temperature.

24. Temperature differences at the junctions between the gold leaf and the pins set up a thermoelectric potential which becomes the electrical signal. The sensitive element of the thermocouple is enclosed in a steel casing to eliminate pick-up, and evacuated to increase its signal-to-noise ratio. A potassium bromide window, transparent to infrared energy wavelengths to about 25μ , is used to expose the sensitive element to the light beam.

CAUTION: Handle the thermocouple carefully. Rough treatment may break the thermocouple junction. The potassium bromide window is very soluble in water and should never be touched or placed in a damp atmosphere.

PREAMPLIFIER AND ELEVEN C/S AMPLIFIER

25. As discussed previously, when the intensities of the sample and reference beams are equal, the thermocouple will produce only a direct voltage. On the other hand, when one beam is more intense than the other, a 11 c/s alternating voltage arises. This alternating voltage, modified to 50 c/s, drives the servo motor which controls the optical wedge and re-establishes the equilibrium between the two beams. The

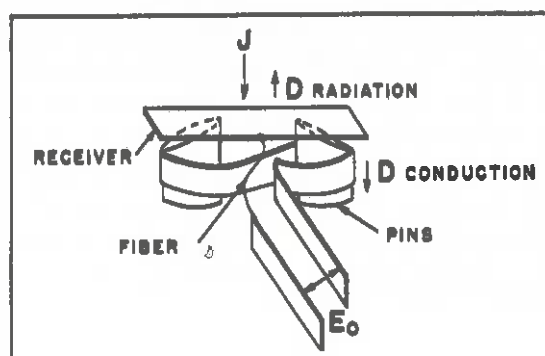


Fig.3.11 Schematic Diagram of Thermocouple

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functions of the pre-amplifier and the 11 c/s amplifier are to eliminate the direct voltage and to amplify the 11 c/s signal.

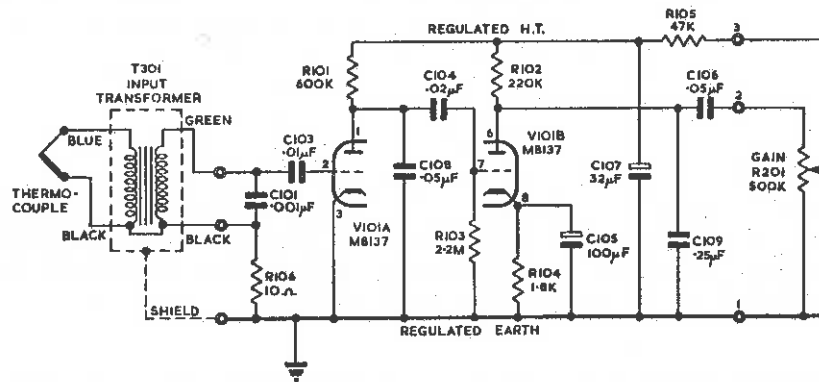
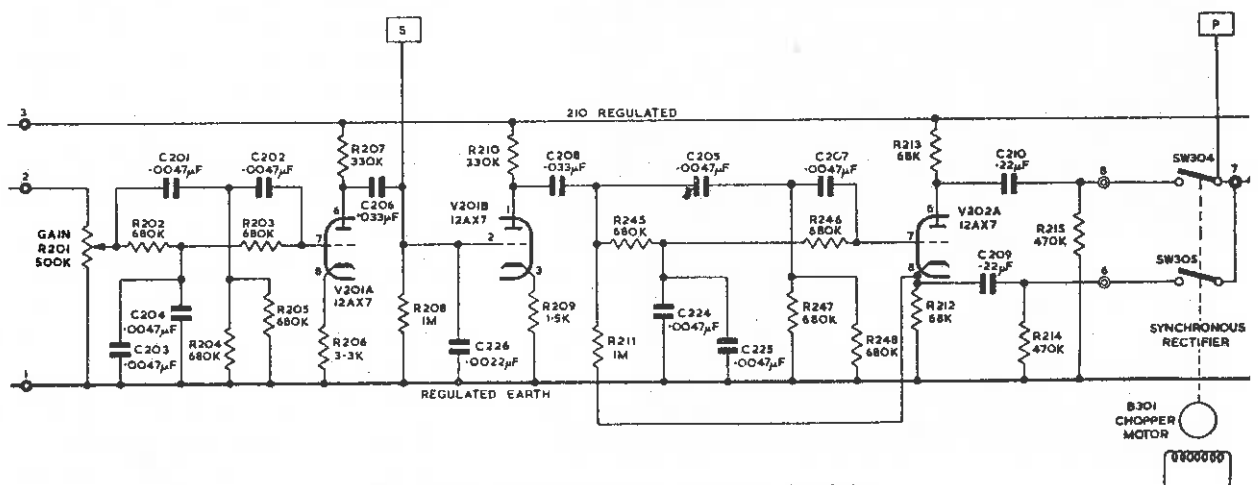


Fig.3.12 Circuit Diagram of Detector and Preamplifier

26. The input transformer, T301, (Fig.3.12) serves to couple the thermocouple to the grid of V101A in the preamplifier, and also to block any D.C. signal. Its secondary winding is tuned to 11 cycles and has a voltage step-up of 600:1.

27. The preamplifier has two stages which amplify the error signal by about 800. The "floating" grid of V101A (no grid resistor) establishes a high input impedance to the amplifier to prevent any reduction of the effective voltage step-up in the transformer. After amplification by the preamplifier, the signal is fed to the grid of V201A (Fig. 3.13) in the 11 c/s amplifier through GAIN control R201 and a parallel T, 50 c/s rejection filter. The two halves of V201 comprise the two stages of gain, and amplify the signal by a factor of about 500. In addition to amplifying the signal, the 11 c/s amplifier provides a signal to the synchronous rectifier. This is accomplished by coupling the output signal from V201B to the grid of V202A through a second parallel T, 50 cycle rejection network.



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28. The amplifier also amplifies a noise signal which tends to obscure information when the error signal is very small. This noise is primarily due to Johnson noise in the thermocouple, and to a lesser extent, to flicker noise in the first stage of the preamplifier tube. Normally the peak-to-peak noise voltage is of the order of 2×10^{-9} to 4×10^{-9} volts for the conditions selected with the slit programme set to N. The significance of this is that any reading on the Transmittance scale can vary by about 1% of full scale.
29. Since the pen drive system is a closed-loop system the bandpass will increase as the gain is increased (GAIN control turned clockwise). If the gain is increased excessively, the system will break into a spontaneous oscillation at about 2 cycles per second. When the gain is properly adjusted as described earlier, the bandwidth of the pen servo is about 1 cycle per second.
30. The bandpass is one of the factors that determines the noise level of the system. It also has a further significance. A 1 cycle per second bandpass means that all spectral information changing at rates ranging from 0 - 1 c/s is faithfully reproduced. Spectral information at higher frequencies, if present, would not be faithfully reproduced. Scanning speed and spectral slit width, in the Infracord spectrophotometer have been programmed in such a way that the spectral information produced by the instrument whilst scanning at the slow speed and with the slit control set to N lies within the bandpass of the instrument and is faithfully reproduced by the pen. Faster scanning of wavelengths may lead to slight inaccuracies when substances with very sharp bands are being examined.

THE SYNCHRONOUS RECTIFIER

31. The synchronous rectifier changes the 11 c/s signal obtained from V202A into direct voltage whose polarity indicates the beam, sample or reference, which is more intense. It consists of two switches SW304 and SW305 operated by a magnet mounted on the same shaft as the sector mirror (see Fig. 3.14). During one half cycle of sector mirror rotation, SW304 is closed and SW305 is open, and on the succeeding half cycle SW304 is open and SW305 is closed.

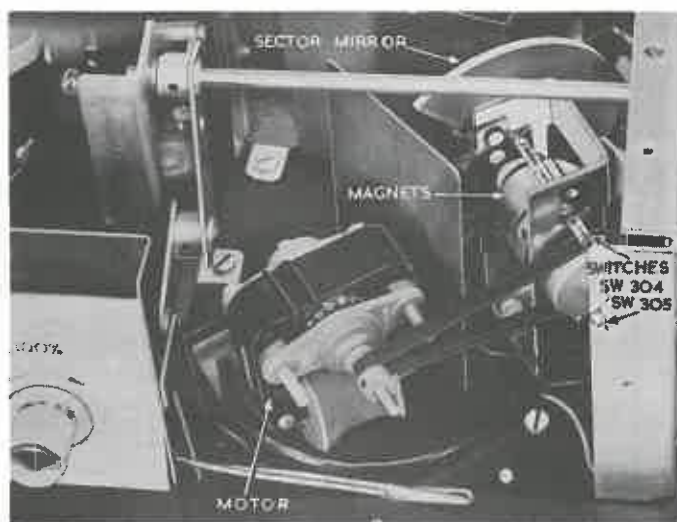


Fig. 3.14 Sector Mirror and Drive

For the sake of illustration, assume that SW304 is closed only when the sample beam is transmitted by the sector mirror and that negative direct voltage represents less intensity in the sample beam than in the reference beam (increasing absorption). Under the assumed conditions, when the intensity of the sample beam is reduced by absorption and SW304 is closed, the signal at the anode of V202A is in a negative half cycle. A negative signal is applied, in turn, therefore, to the 50 c/s modulator. During the succeeding half cycle, SW304 is open and SW305 is closed and again a negative signal is applied to the modulator, since SW305 is connected to the cathode of V202.

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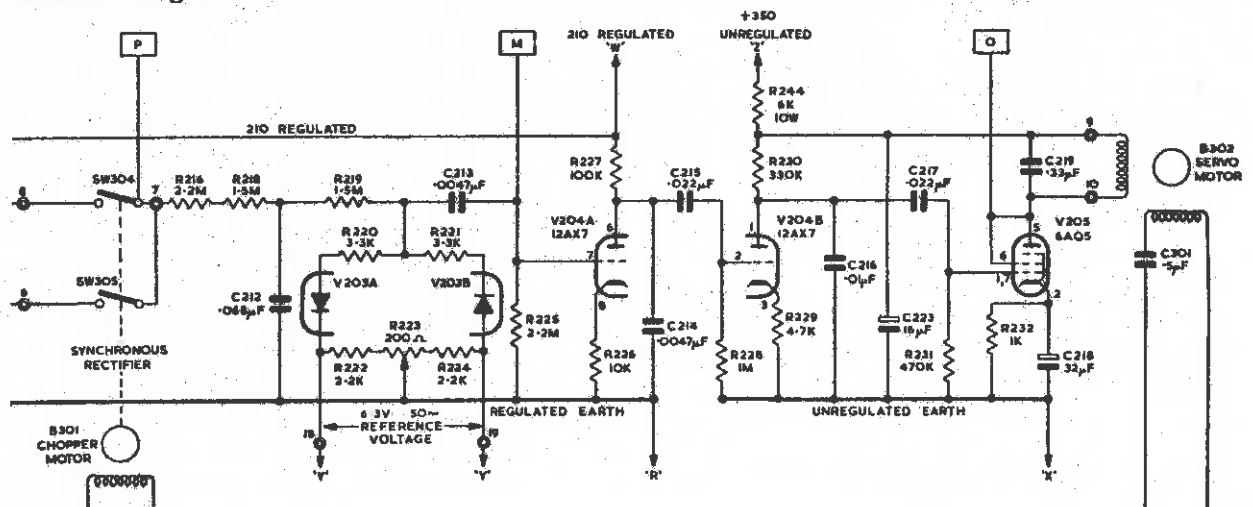
The effect, therefore, is to transmit negative full wave rectified voltage to the modulator whenever the intensity of the sample beam is less than that of the reference beam.

32. The reverse occurs when the intensity of the sample beam becomes greater than that of the reference beam due to decreasing absorption by the sample. The effect here is to transmit positive full wave rectified voltage to the modulator. These descriptions will be somewhat more easily understood if a little time is taken to study the signal waveforms shown in Section 1, INTRODUCTION.

NOTE: In practice, a phase shift of about 90 degrees is introduced between the flickering light signal at the thermocouple and the electrical signal at the synchronous rectifier. This shift is due to the effects of the thermocouple and the amplifiers. It is compensated by rotating the magnet on the sector mirror shaft with respect to the sector mirror.

FIFTY C/S MODULATOR AND FIFTY C/S AMPLIFIER

33. The 50 cycle modulator (Fig. 3.15) changes the direct voltage from the synchronous rectifier to a 50 cycle alternating voltage which can be used to drive the pen servo motor. It consists of two silicon diodes, V203A and V203B built into a plug-in unit with a B7G base. The anode of one diode is in series with the cathode of the other. A 50 cycle signal from transformer T302 is connected to the diode circuit. The diodes will conduct only during the half cycle of the 50 cycle signal when the anodes are positive and the cathodes negative.



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signal already present. This control can therefore be used to balance out any constant undesirable signals which arise in the instrument.

36. The 50 c/s signal passed by C213 is amplified about 600 times by the two stages of the 50 c/s amplifier V204A and V204B. It is then fed through power amplifier V205 to the control winding of the pen servo motor.

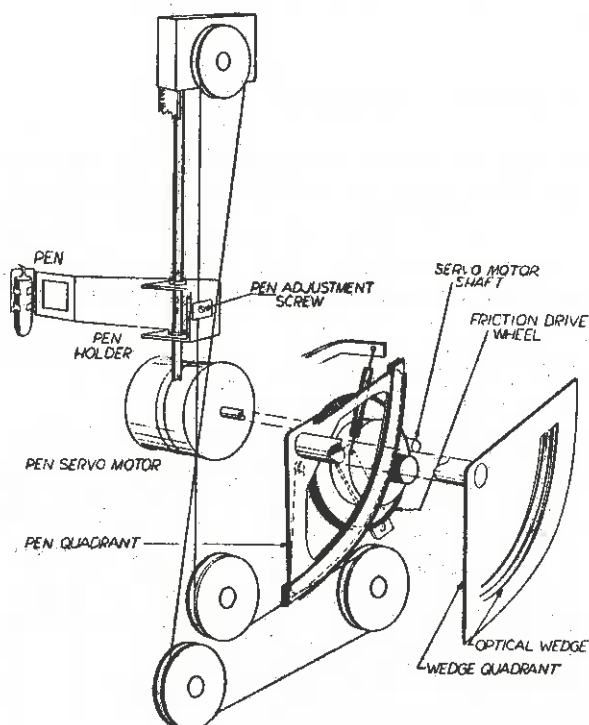


Fig. 3.16 Schematic Diagram of Pen Drive

THE PEN DRIVE

37. **Electronics** The pen servo motor converts the 50 c/s signal from the anode circuit of V205 (Fig. 3.15) into mechanical rotation and drives the pen and moves the optical wedge to null the signal. The pen servo motor has two windings, a control winding energized by the signal, and a reference winding connected directly to mains voltage through a phase-shifting capacitor C301. The motor phasing is such that the control winding voltage either leads or lags the reference winding voltage by 90° , depending upon which beam, sample or reference, is more intense. If the signal voltage leads, the motor turns in one direction; if it lags, the motor turns in the opposite direction.

38. **Mechanics** A nylon friction drive wheel (Fig. 3.16) mounted on the servo motor shaft drives a second shaft on which are mounted two quadrants. One quadrant carries the wire cable connected to the pen holder and the other the optical wedge. With both the pen quadrant and the wedge quadrant on the same shaft, the pen must move an amount proportional to the wedge movement, and non-reproducibility due to slippage

between pen and wedge is eliminated. The size of the drive wheel is such that the motor shaft rotates at about 100 times the speed of the quadrant shaft. With the servo motor operating at full speed (slewing), this gives a response time of about 1.2 seconds in which the pen drives full scale. The edge of the pen quadrant which is driven is rubber covered except at its ends to ensure a positive drive. At the ends the drive wheel slips on bare aluminium, thus preventing damage due to the quadrant being driven beyond its limits.

POWER SUPPLY

39. The power supply provides the necessary direct voltages for the instrument. It consists, basically, of a transformer, a rectifier power supply and a regulator.

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The alternating voltage, obtained from the high voltage winding of transformer T302 (Fig. 3.17) is converted to a direct voltage using a conventional full wave rectifier. This voltage is somewhat smoothed by resistor R233 and capacitor C220 to a direct voltage with a large 100 c/s ripple.

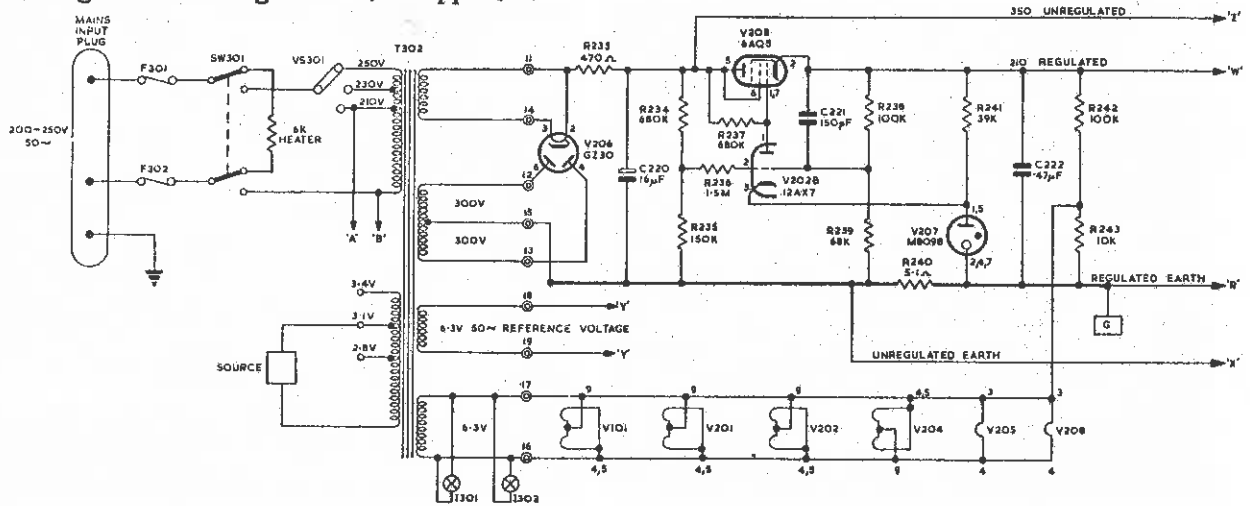


Fig. 3.17 Circuit Diagram of Power Supply

40. The power supply load can be considered in two sections. The major portion of the load is represented by the last amplifier stage V204B and the power amplifier V205 which provides the power to the control winding of the servo motor. Since these stages occur late in the amplifier chain, variations in anode voltage and large amounts of ripple on these stages do not affect the performance of the instrument. This being the case, it is not necessary to provide them with regulated power.

41. The gain of the earlier stages of the amplifier is such, however, that they do require a regulated power supply providing constant voltage and low output impedance. The regulator is designed to deliver approximately 210 volts to the early stages of the amplifier regardless of normally encountered variations in line voltage or load current. It also minimizes the amount of 100 c/s ripple at its output.

DRUM(WAVELENGTH) DRIVE

42. The drum, or wavelength, drive consists of the scanning motor (Fig. 3.18) and the associated mechanical linkages used to rotate the drum. It also alters the wavelength setting and opens or closes the slits since the wavelength and slit cams are mounted on the drum shaft.

43. The drive unit is controlled via a flexible drive cable by the SCAN switch on the instrument front panel. This switch has three positions; FAST, STOP and SLOW. In the SLOW position the motor is swung into contact with the slow gear train (Fig. 3.18) so that the complete wavelength range is scanned in approximately twelve minutes. The engaging of the motor is accurately controlled by a cam and spring mechanism which ensures that the motor is guided into gentle engagement and then held firmly in position. Selection of FAST scan is identical except that the fast gear train is engaged with the result that the complete wavelength range is scanned in three minutes (six minutes in some earlier models).

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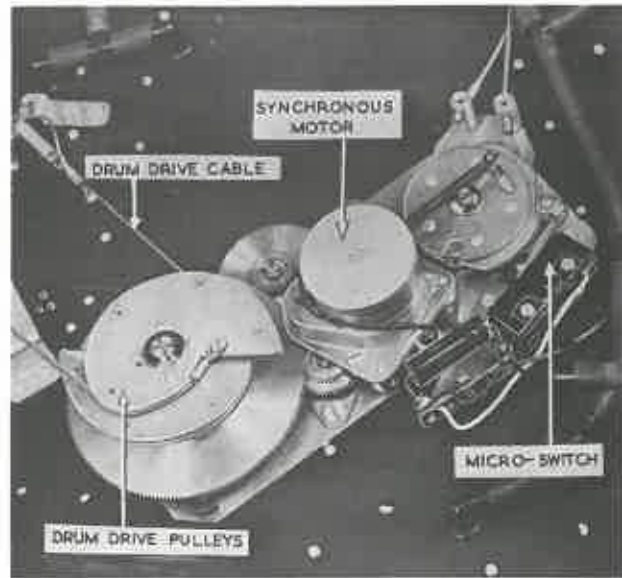
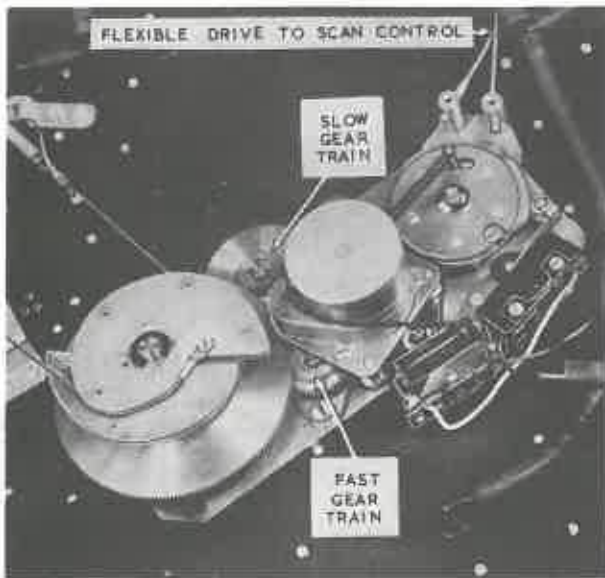


Fig. 3.18 Synchronous Motor and Two-Speed Drive (Slow) Fig. 3.19 Synchronous Motor and Two-Speed Drive (Stop)

44. In the STOP position (Fig. 3.19) the motor is held securely in a non-engaged position and its power supply is disconnected. The cam-operated micro-switch shown in Fig. 3.19 switches the power to the motor only when one of the driving positions is selected.

45. The SLOW scanning speed has been chosen to allow the pen servo system, when using the normal (N) slit programme, to follow the sharpest absorption bands at all wavelengths. Since the spectral slit width, in terms of wavelength, increases at longer wavelengths the scanning speed must also increase with wavelength in order to maintain the optimum rate of scan. This is accomplished by driving the drum through a set of non-linear pulleys. The drive pulley driven by the motor and the drive pulley fixed to the drum shaft are connected by two drive cables which are held at the correct tension by springs. With the FAST scanning speed some lag will occur in the record when scanning very sharp bands, but the speed has been chosen to allow useful survey work to be carried out on even the sharpest bands.

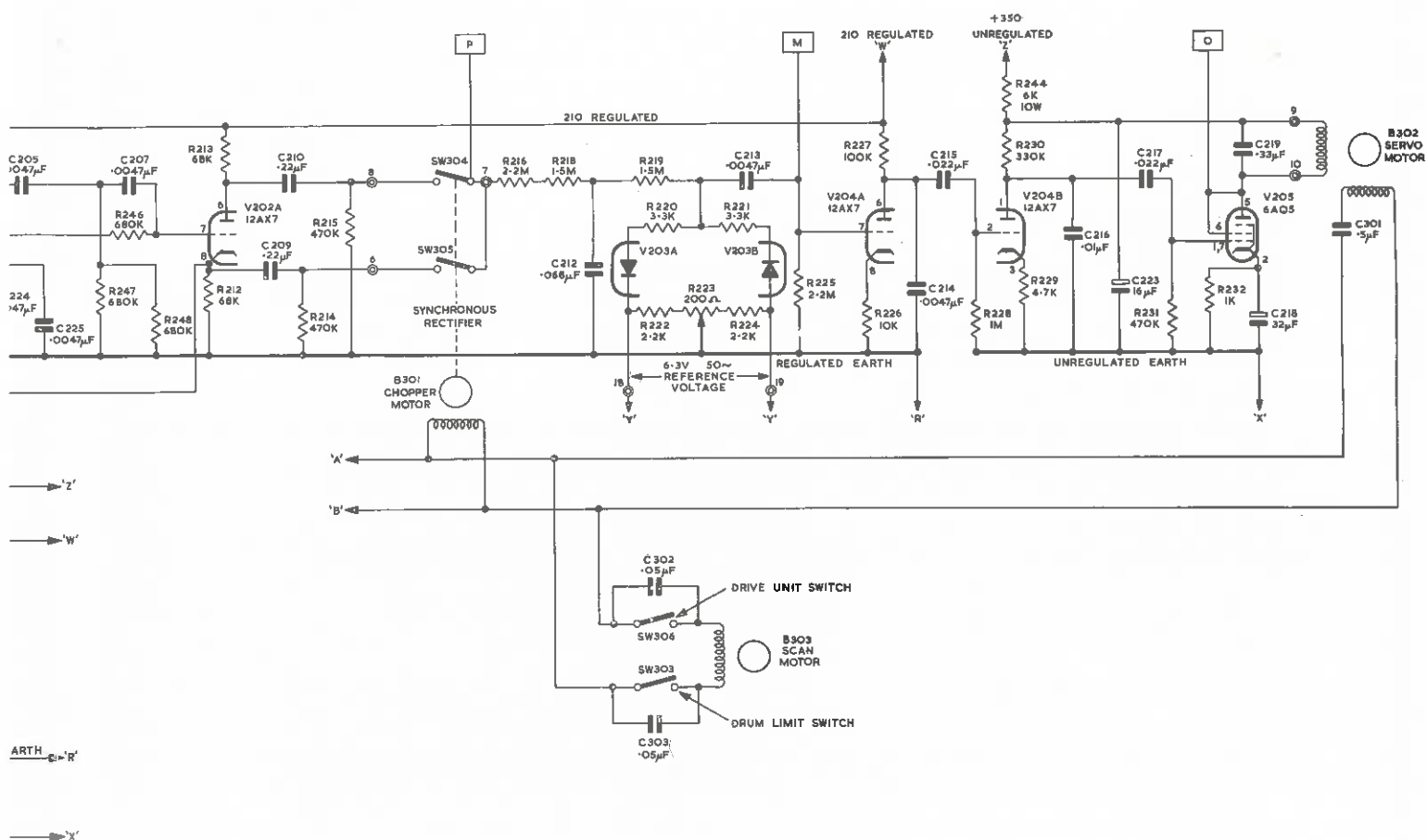


Fig. 3.20 Complete Circuit Diagram of Infracord Spectrophotometer

