2. OPERATING INSTRUCTIONS

GENERAL

1. The Model 257 Spectrophotometer is an extremely sensitive instrument and has been carefully aligned and fully tested before leaving the factory. In order to achieve maximum performance it is essential that the following installation and operating instructions be followed carefully.

2. Operators are warned that the instrument operates from the mains voltage and no attempt to handle any component within the protective cover should be made with the power supply connected.

3. The instrument should not be exposed to conditions of high relative humidity which may result in fogging of the potassium bromide window of the thermocouple.

INSTALLATION

Preliminary

4. Figures 2.1 and 2.2 show the location of controls referred to in the following instructions.

a) Place the instrument on a firm surface.

b) Check that the POWER/RANGE switch on the side panel is set to OFF.

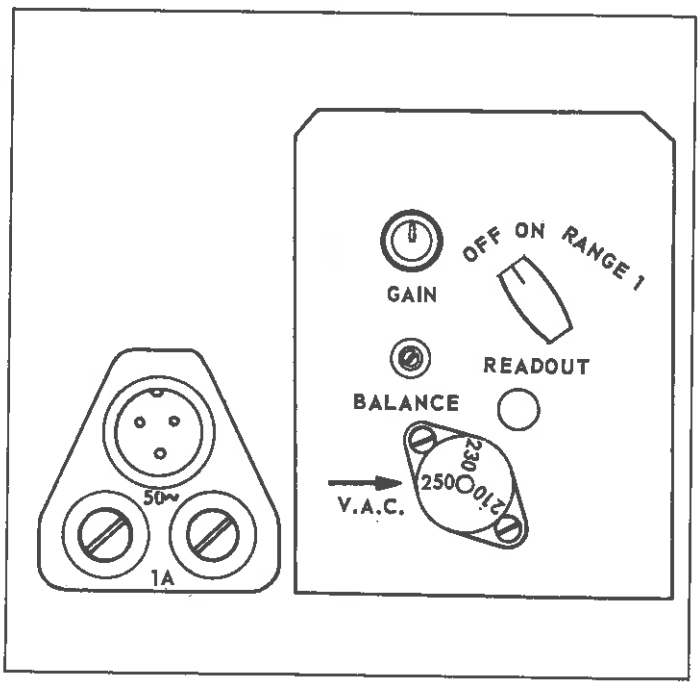


Fig. 2·1 Rear and Side Panel Controls

c) Set the voltage selector on the side panel to correspond with the supply to be used.

d) Connect the power supply cable between the 50 c/s socket and the mains power supply. The 3-core cable must be used and the following colour code observed.

RED	–	LINE
BLUE OR BLACK	–	NEUTRAL
GREEN	–	EARTH

e) Rotate the SLIT control to the N (Normal) position.

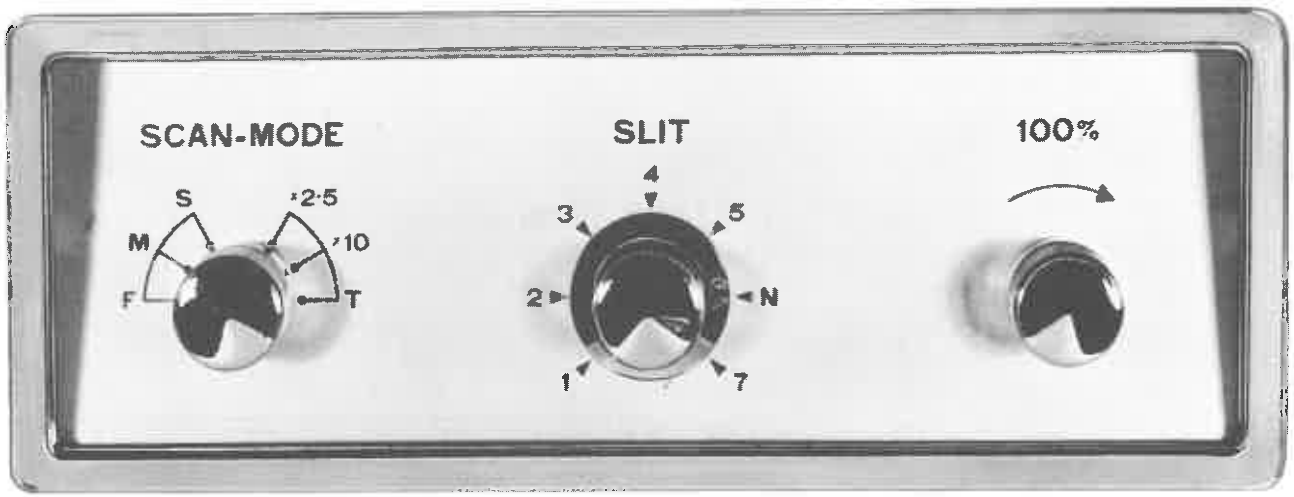


Fig. 2·2 Front Panel Controls

f) Switch the instrument ON. A 10 to 15 minute warm-up period should elapse before the instrument is operated.

g) After setting the GAIN, BALANCE and 100% controls according to the following instructions (paragraphs 2·19 – 2·23) the basic instrument is ready for operation.

Preparing and Cleaning the Pen.

5. Before the pen supplied is used on the instrument, its protective cap must be removed. The following steps should be carried out, where appropriate, when initially filling and subsequently refilling the pen.

a) Unscrew and remove the pen from its holder.

b) Draw the reservoir from the stem using a slight twisting action.

c) Remove any ink residues from the reservoir junction on the pen body by tapping it on blotting paper.

d) Fill the reservoir to the lower edge of the chromium ring.

e) Press the reservoir onto the pen body.

f) Shake the pen and test until the ink flows freely. Wipe any ink from the outer surface of the capillary. A rattling sound when shaking the pen indicates that the regulating device is operating correctly.

g) Holding the cone downwards, remove surplus ink from around the air vent (situated at the base of the writing cone) with blotting paper.

h) Screw the pen into the carriage as far as it will go.

NOTE: The pen should be refilled before the contents of the reservoir drop to half its capacity.

i) Dismantle the pen and wash in water periodically. Care should be exercised when removing the writing cone with the cone key. The regulating device automatically cleans the pen internally.

Fig. 2-3 Filling the Pen Reservoir

Fitting the Chart Roll

6. To fit a new roll of chart paper, raise the pen bridge and magazine cover. Transfer the end plugs to the new roll, lay it in the magazine and close the cover. Engage the chart perforations with the drive sprockets so that the marked pins and holes coincide. Lower the pen bridge to latch into position. If necessary, turn the sprockets by depressing and turning the MANUAL CHART DRIVE control, until the settings of the chart and wavenumber scale are synchronized. Finally, turn this control a little in both directions without depressing to check the engagement of the paper drive.

NOTE: Only a light torque is required to drive the chart. Do not attempt to force or to apply excess torque to the manual chart drive control.

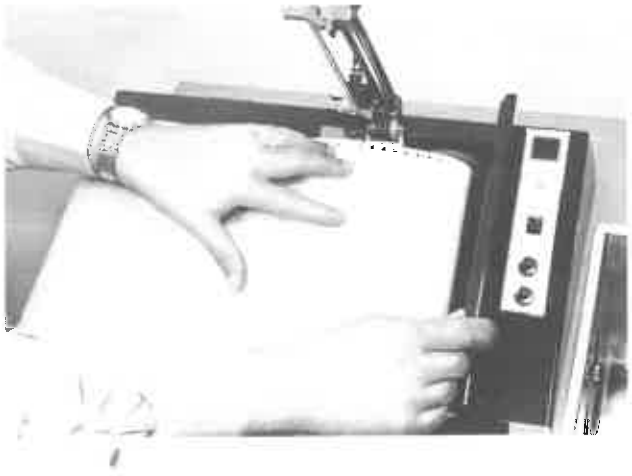


Fig. 2·4 General View

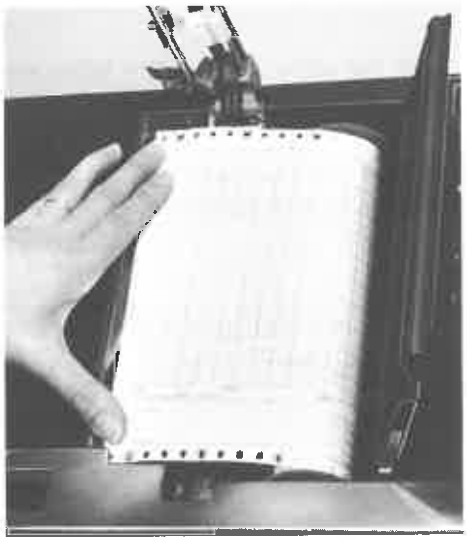


Fig. 2·5 Marked Sprocket and Perferation.

FUNCTIONS OF CONTROLS

POWER/RANGE Switch

7. The instrument covers the infrared spectral range from 4000 to 625 cm^{-1} in a continuous scan. The POWER/RANGE switch situated on the side panel controls both the power supply to the instrument and selects the range of operation. In the ON position the full range is scanned, and in the RANGE 1 position the range 4000 cm^{-1} to 1250 cm^{-1} is scanned. There is an inoperative position between the ON and RANGE 1 positions.

SCAN-MODE Switch

8. The scan drive is controlled by a six-position switch marked SCAN-MODE. The normal scan positions 'S' (slow), 'M' (medium) and 'F' (fast) correspond to scan rates of 100, 400, and 1000 cm^{-1} per minute in the region 4000 to 2000 cm^{-1} and 50, 200, and 500 cm^{-1} per minute in the region 2000 to 625 cm^{-1}. The FAST scan is suitable for rapid SURVEY work, the MEDIUM scan for work of a QUALITATIVE nature and the SLOW scan for QUANTITATIVE intensity measurements. The interpretation of the scanning speeds will also depend on the nature of the sample being studied, e.g. the full information content of a broad absorption band will be obtained on MEDIUM scan speed. In the 'x 2.5' or 'x 10' positions the abscissa of the recorded spectrum is expanded by the amount indicated. With the switch in the 'T' position a recording of transmittance as a function of time is obtained.

100% Control

9. The 100% control provides a manual means of controlling the sample beam intensity and is used to set the 100% transmission level. When the control is turned clockwise a wedge-shaped comb is moved out of the sample beam and the pen moves up-scale.

CAUTION: The 100% control operates the comb through a screw which has a large mechanical advantage in order to facilitate very fine adjustment. For this reason the control should not be forced beyond its limits or it may become irreparably damaged.

Variable SLIT Control

10. The source does not emit the same amount of energy at all wavelengths. Due to this and other instrumental factors the slits are programmed by a cam to maintain the energy falling on the detector at an approximately constant level throughout the instrument range, thus keeping the pen response and signal to noise ratio constant. The cam programme can be multiplied and divided by rotating the SLIT control. Thus the energy available at the detector differs for each setting of the control but is approximately constant throughout the instrument range for any given setting. The slit control setting used will depend on the particular application, as discussed in paragraph 2.31. The significance of the slit control graduations is as follows:–

'N' In the N, or normal position the level of the slit programme has been adjusted to give a signal to noise ratio of not less than 200:1 in the range 4000 to 2000 cm^{-1} and 100:1 in the range 2000 to 625 cm^{-1}.

'1, 2, 3, 4 and 5' These settings give progressively narrower slit programmes corresponding to approximately 0.4, 0.5, 0.6, 0.7, and 0.8 times the normal programme slit width, respectively. All these settings give a smaller spectral slit width, i.e. higher resolution, at the expense of lower signal to noise ratio since a higher gain setting is required to provide adequate pen response.

'7' This setting corresponds to approximately 1.5 x N slit widths thus allowing a greater signal to noise ratio at the expense of reduced resolution.

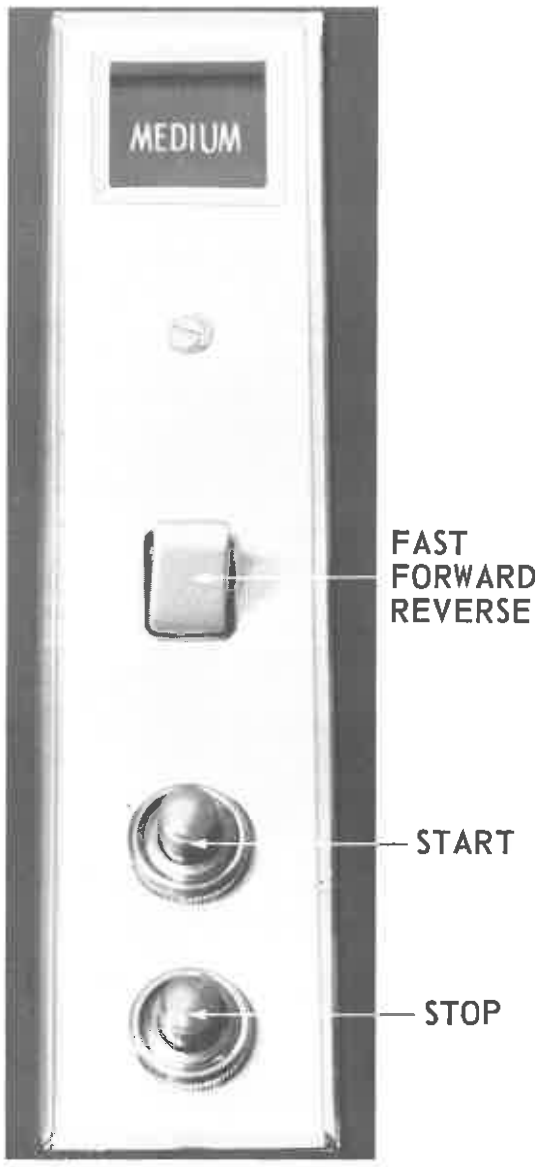


Fig. 2·6 Top Panel Controls.

START and STOP Buttons

11. Having switched the instrument on and selected the range and scan mode, the instrument and chart drives are energized by pressing the green START button. When the red STOP button is pressed the scan stops with the pen lifted clear of the chart.

VISUAL MODE Indicator

12. A green signal is shown on the indicator when the green START button has been pressed and the instrument is scanning. A red signal appears whenever the instrument is not scanning, for example, when the red STOP button has been pressed. The display changes according to the selected SCAN-MODE, and an 'X' is superimposed on the display when the POWER/RANGE switch is in the 'RANGE 1' position.

FAST FORWARD/REVERSE Switch

13. Forward or backward movement of the switch from the central rest position rapidly advances or reverses the wavenumber setting of the instrument, the chart being driven in synchronism. Actuation of the FAST FORWARD/REVERSE switch de-energizes the scan and causes the pen to lift.

WAVENUMBER Indicator Scale

14. The wavenumber setting is displayed on an illuminated scale which can be viewed through the aperture near the front of the instrument. The scale is calibrated on the inside from 4000 to 1250 cm^{-1} (the complete range of the first grating) and on the outside from 2000 to 625 cm^{-1} (the range of the second grating) the part of the scale that is illuminated being in accordance with the grating in operation. In a complete scan the wavenumber scale rotates from 4000 to 2000 cm^{-1}, reverses to 2000 cm^{-1} on the outer scale and continues to 625 cm^{-1}. In the RANGE 1 mode the scale rotates continuously from 4000 to 1250 cm^{-1}.

GAIN Control

15. The GAIN control varies the amplification of the signal fed to the pen servo-motor and is adjusted for optimum pen response. Adjustment will be necessary periodically to compensate for ageing of electronic components and will also be necessary when the SLIT control is varied.

BALANCE Control

16. The BALANCE control provides a means of compensating for the effects of random electrical pick-up which would otherwise result in an erroneous signal to the pen servo system. The pen balance should be such that, with no energy passing through either the sample or reference beams, the pen will remain stationary at mid-scale, or move slowly upscale.

IMPORTANT: The BALANCE control should be checked and, if necessary, adjusted after changing the GAIN control setting.

MANUAL CHART DRIVE Control

17. The MANUAL CHART DRIVE control is situated at the rear of the recorder. Pressing and turning this control disengages the chart drive coupling to the instrument, and advances or reverses the chart. If more than one grid length of chart is to be re-wound into the magazine the pen bridge and magazine cover should be raised to permit direct access to the chart roll for re-winding. Before commencing a scan the MANUAL CHART DRIVE control should be used to synchronize the chart grid with the wavenumber setting of the instrument. Check the re-engagement of the chart drive by releasing the pressure on the knob and turning it slightly from side to side until resistance is felt.

NOTE: Only a light torque is required to drive the chart. DO NOT ATTEMPT TO TRANSMIT EXCESS FORCE OR TORQUE TO THE INSTRUMENT THROUGH THIS CONTROL.

Fig. 2·7 Manual Chart Drive Control

Readout Accessory

18. The sealed hole in the instrument side panel is provided for fitting a socket through which the signal to an external recorder is fed from the pen position readout accessory.

Preliminary Steps

19. The following steps should be taken before attempting to record a spectrum.

a) Check that the Voltage Selector corresponds to the power supply.
b) Switch the instrument on, and it will automatically return to the start limit position. Allow 10 to 15 minutes warming up period.
NOTE: At the start limit the pen is dead, but on starting the scan it becomes live before 4000 cm^{-1} is reached.
c) Fill the pen and fit a chart roll complete with end plugs in the magazine (paragraph 2.6), engaging the marked chart perforations with the marked sprockets. By means of the MANUAL CHART CONTROL, position the paper so that the chart grid is synchronized with the wavenumber scale setting and ensure that the chart drive is engaged.
d) Select the SCAN MODE.
e) The GAIN and BALANCE controls should be checked at least once daily and the 100% control setting should be checked before every spectrum is run. Weekly checks on the zero setting of the pen should be made, but this should rarely need adjustment. If adjustment is necessary, the procedure detailed below should be followed.
f) After positioning the sample in the sample compartment, to commence a scan press the green START button. When the final limit is reached the instrument will automatically reverse to the start limit, the chart remaining stationary.
g) After a scan is completed the used chart is detached from the roll by running a blade or finger nail along the transverse printed line on the chart after positioning the line over the cutting groove which is slightly in front of the pen. The ball pens supplied incorporate a knife edge designed for this purpose.

GAIN Control Adjustment

20. The amplifier GAIN control determines the amplification of the signal to the pen servo-motor. The higher the gain, the greater is the voltage applied to the servo motor for a given difference signal between the two beams, and the faster the pen response. For normal operation, the GAIN control should be set so that there is no "dead space" in the pen response.

a) Select a region free from atmospheric absorption, say 1250 cm^{-1}
b) Set the 100% control so that the pen indicates 90% transmittance.
c) Reduce the transmittance reading by approximately 10% by inserting the index finger into the sample beam at the right-hand side of the sample tray.
d) Remove the index finger quickly from the sample beam and notice the manner in which the pen returns to its original position; note also the final transmittance reading. One of the following conditions will be noted, and an incorrect GAIN setting should be corrected as indicated.

GAIN TOO HIGH-the pen returns quickly and overshoots the original position by more than 1% of full scale. Turn the GAIN control counter-clockwise to decrease the gain.

CORRECT GAIN-the pen returns quickly to the original position or overshoots by less than 1% of full scale.
GAIN TOO LOW-the pen moves sluggishly towards the original position. Turn the GAIN control clockwise to increase the gain.

e) The above operations should be repeated until no "dead spot" or excessive overshoot is apparent. When the GAIN control is correctly set, the BALANCE control should be checked.

BALANCE Control Adjustment

21. The pen balance should be such that, with no energy in either the sample or reference beams, the pen does not move or moves very, very slowly upscale. The correct procedure for adjusting the BALANCE control is as follows:—

a) Place a strip of opaque material (such as thick cardboard) in the sampling area so that it blocks both sample and reference beams.
NOTE: Try to block <u>both</u> beams simultaneously, as any time lag will cause a temporary pen drift which must be allowed to settle. Manually move the pen carriage <u>slowly</u> to the middle of the transmittance scale. The pen should either remain in position or drift slowly upscale.
b) adjust the BALANCE control on the side of the instrument until the proper balance is established.

IMPORTANT: Following adjustment of the BALANCE control the GAIN control setting should be checked; some readjustment may be required after any major change in the BALANCE control setting.

Zero Adjustment

22. The ZERO setting should be checked about once a week but should rarely need adjustment. At true zero transmittance (when there is no radiation in the sample beam) the pen should rest at zero transmittance on the chart.
The correct procedure for adjustment is as follows:—

a) Select a region free from atmospheric absorption, say 2500 cm^{-1}.

b) Using a strip of cardboard (as in the setting of the BALANCE control) block both the sample and reference beams and check that the pen remains stationary or drifts only very slowly upscale. If necessary adjust the BALANCE control as described in paragraph 21 of this section.

c) Carefully slide the cardboard a short distance towards the front of the instrument to allow a very small amount of radiant energy into the reference beam window. The pen should move slowly down scale at a rate of not more than ½% transmittance per second. Do not move the cardboard again until the pen has stopped moving.

d) When there is no pen movement, slide the cardboard a little further out of the reference beam and wait until the pen has stopped moving.

NOTE: The sample beam should remain blocked throughout the whole of this procedure.

e) Continue to withdraw the cardboard strip – slowly – in small increments, until the pen comes to rest with the reference beam completely unobstructed.

f) Press the pen against the chart paper, if the mark made by the pen does not correspond with zero on the chart paper, raise the pen bridge, slacken the pen adjustment screw (Fig. 2.8) and reposition the pen carriage along the drive chain. Re-tighten the screw, lower the pen bridge into the operating position.

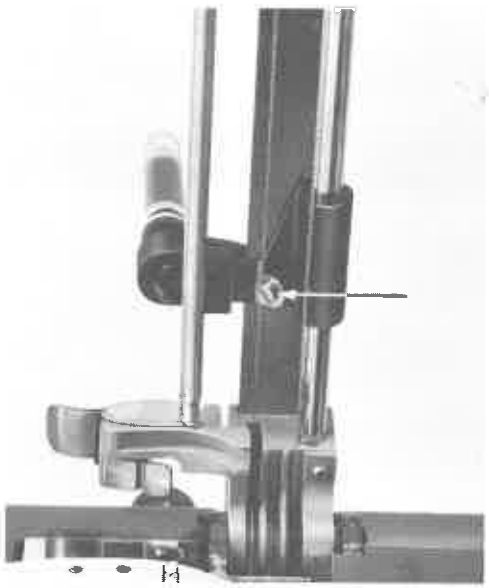


Fig. 2-8 Pen Carriage Adjustment Screw

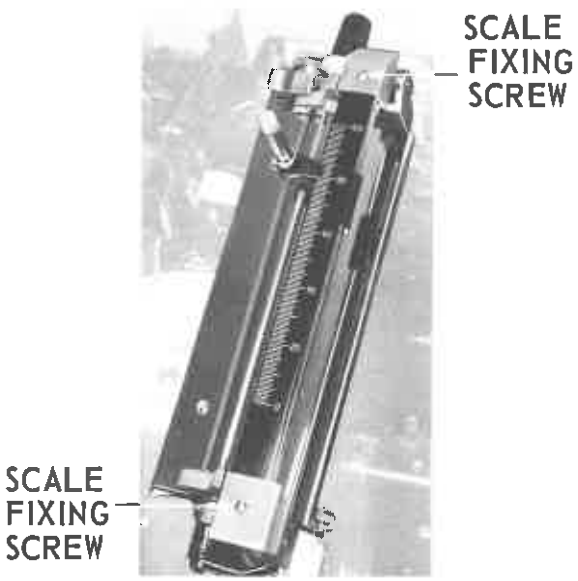


Fig. 2-9 Transmittance Scale Adjustment.

g) Repeat steps b) to f) to confirm that the setting is correct.

h) If necessary to reset the transmittance scale to read correctly, slacken the two securing screws and slide the scale so that the pointer on the pen holder indicates the same transmittance as the chart. Tighten the two securing screws.

100% Adjustment

23. The 100% adjustment control permits balancing of any slight inequalities between the two beams. With no sample in either beam, rotate the 100% control until the pen reaches 100% on the chart. THE INSTRUMENT IS NOW PROPERLY SET UP TO MAKE TEST RUNS.

TEST RUNS

24. For an adequate check on the instrument performance, test runs with indene in a 0.05 mm sealed cell are recommended. (A suitable cell with rock salt windows can be obtained from Perkin-Elmer, Part No. INF 1176, 0.05 mm thick). For comparison with the reference spectra (paragraph 2.36) the test runs should be made on the SLOW scan speed and the N (Normal) slit programme. All accurate readings of transmittance must be made with respect to the printed scale on the chart.

To record the spectra, proceed as follows:–

a) Carry out the preliminary steps described in paragraph 19 of this section.
b) Fill the indene cell and mount it in position in the sample compartment.
c) Scan on the SLOW speed and N slit programme. On pressing the START button the instrument will automatically record the absorption spectrum of the sample from 4000 to 625 cm^{-1}. At the conclusion of the scan the instrument will automatically return to the start limit. During this operation the chart remains stationary.
d) The stray light check with quartz and calcium fluoride may be similarly obtained. The double beam I_o line (i.e. no obstruction in either beam) should be recorded after setting the pen to approximately 90% transmission.

25. The test spectra recorded immediately after installation should be comparable with the reference spectra in Figs. 2.10 to 2.13 within the limits of the specification in the following respects:–

a) Wavenumber accuracy
b) Resolution
c) Stray light
d) I_o (100% line) straightness
e) Noise

The repeatability of the instrument should be checked by making two consecutive scans on the sample under identical conditions on the same chart paper. Near the completion of the first scan reverse the instrument from 625 cm^{-1} to the start limit using the FAST FORWARD/REVERSE switch, thus reversing the chart in synchronism with the instrument drive. If the instrument reaches the final limit and automatically reverses leaving the chart stationary, reverse the chart by means of the MANUAL CHART DRIVE control. Repeat the scan.

26. The polystyrene film provided with the instrument may also be used to check performance. It does not, however, provide such an accurate calibration check due to perturbation of absorption band positions by overlap with other bands or with interference fringes. In addition, the sample lacks calibration points in the 4000 cm^{-1} region. A reference spectrum is given in Fig. 2.12.

Selecting the Range

27. The POWER/RANGE switch on the side panel controls the scan range. In the ON position the full range from 4000 – 625 cm^{-1} is covered automatically and continuously. The RANGE 1 position covers from 4000 – 1250 cm^{-1} with a chart presentation of 100 cm^{-1} per cm throughout. This facility aids those who wish to use the greater signal to noise ratio (without significant change in resolution) that can be obtained in this mode in the 2000 – 1250 cm^{-1} region.

Interrupted Scanning

28. Should it be desired to only record one or more parts of the spectral range, the FAST FORWARD/REVERSE switch may be used to drive the instrument and chart between the regions of interest and the START button actuated to continue the scan. If recordings are on the continuous grid chart, the length used can be minimized whilst driving fast in the forward direction by pressing the MANUAL CHART DRIVE control to disengage the chart drive. Before actuating the scan, manually move the chart forward until the drive is re-engaged.

Repeated Scanning

29. Repeated scans may be desired to observe the effect of changed instrument parameters or to compare the spectra of two similar samples. Such recordings may be made sequentially along the chart or superimposed on the chart.

a) Repeated Scanning with Superimposed Recording.

After completing the full range recording of the first sample's absorption spectrum the instrument will automatically return to the start limit. Reverse the chart using the MANUAL CHART DRIVE control to synchronize the same chart grid with the instrument wavenumber scale and check the chart drive engagement. The second recording can then be superimposed on the previous spectrum. To repeat and superimpose two or more spectra over a reduced range, drive fast forward to the commencement of this range then actuate the scan. Using the FAST FORWARD/REVERSE switch, reverse from the long wavelength limit of the region of interest to the short wavelength limit of this region. The chart drive remains engaged and therefore the instrument and chart reverse in synchronism. The second recording can now be superimposed on the first.

b) Repeated Scanning with Sequential Recording.

Repeated full range scanning with sequential recording is carried out in an identical manner to normal recording (paragraph 2.24). Limited range scanning with sequential recording is accomplished by driving fast forward to the chosen start position, scanning through the region of interest and then whilst reversing from the long to the short wavelength limit at the region of interest depressing the MANUAL CHART DRIVE control to disengage the chart drive. Prior to actuating the scan again, manually advance the chart until the drive is re-engaged.

Abscissa Expansion

30. This permits the expansion of the fine detail of closely spaced absorption lines as would occur for example in studies on gases. Such information is usually required over a limited wavenumber region. In the 'x 2.5' and 'x 10' modes, the chart abscissa is expanded by the amount indicated, the instrument scan speeds being as for medium and slow scans respectively. A motor directly geared to the main shaft of the recorder and separate from the instrument drive unit drives the chart, the normal chart drive system being disengaged from the instrument drive. (The same motor

also drives the chart in the Time Drive mode). The recorder chart drive is then independent of the instrument drive and for this reason in the full range scan mode, it continues during the grating changeover at 2000 cm^{-1} (paragraph 3.59). A small part of the spectrum above 2000 cm^{-1} will be repeated when the scan continues at the start of the range of the second grating. For accurate calibration of absorption bands recorded in the mode, an internal standard should be used (paragraph 2.34).

Variable Slit Control

31. The normal (N) position of the SLIT control has been selected to satisfy the requirements of most applications. However, as implied in paragraph 10 it may be necessary in certain instances to accept a lower signal to noise ratio in order to obtain greater resolution, or vice versa. Two special applications of the wide slit programme are :—

a) For greater precision in quantitative analysis where some sacrifice of resolution may be acceptable in order to have lower gain and therefore improved signal to noise ratio.

b) In difference spectroscopy to compensate for loss of energy by absorption in the reference beam. Use of the wide slit programme will assist in achieving a satisfactory pen response without the need to operate on excessively high gain and therefore low signal to noise ratio.

The narrower slit programme may be preferred

a) To resolve two overlapping absorption hands

b) In gas studies where resolution can be exchanged for a lower signal to noise ratio, or

c) For studies on spectral line widths.

NOTE: The GAIN and BALANCE controls must be checked and if necessary readjusted when the SLIT control is altered and when an absorbing sample is placed in the reference beam.

Precise Wavenumber Calibration

32. The repeatability of the instrument is such that the effect of chart printing errors in the calibration can be eliminated to give a wavenumber accuracy significantly better than the instrument specification.

To avoid errors due to incorrect registration of the chart grid with the chart perforations, the instrument is calibrated (prior to shipping) using the printed grid as a guide but using the centre lines of corresponding hole and slot chart perforations as the true datum lines. These centre lines (nominally 1 em apart) correspond to the true positions of the 4000, 3900.... 2000, 1950, 1900....., 650 cm^{-1} grid lines. Chart printing errors can therefore be avoided by interpolating an absorption band position from the nearest pair of chart perforations. This is easily accomplished using a transparent ruler engraved with conveniently spaced lines, say ½ or 1 mm spacing, and of length equal to the chart width. Superimpose the ruler on the recorded absorption band, line up the engraved lines with the centres of the perforations neighbouring the absorption band and interpolate the band position with respect to the engraved lines. Hence from the chart grid dispersion (cm^{-1} per centimetre of chart) calculate the frequency of the absorption band. To be strictly correct, the measurements should be made from the leading (i.e. the driven) edges of the perforations and allowance made to correct to the centre of the 3 mm diameter sprockets. The chart perforations are however a close fit on the sprockets, minimizing any possibility of error.

The effect of systematic and slowly varying errors such as those due to temperature variation can be reduced by using an internal standard when recording the sample absorption spectrum. Again, to obtain maximum precision, measurements along the chart should be made from the perforations as described above.

The following method may be adopted:–

Scan the spectrum of a reference compound for which the position of the absorption line is known with precision. Select two extreme absorption lines and, by linear interpolation from these two datum lines, calculate the apparent wavenumber of the intermediate absorption lines of the reference compound. The difference between the true and apparent wavenumber position of the lines is due to both systematic and random errors in the instrument calibration. An error curve can now be constructed to show this difference as a function of wavenumber. Scan a spectrum of the sample alone and finally a continuously run composite spectrum obtained by interchanging the sample and reference compound during the scan. Construct two error curves showing the difference in position along the chart of corresponding absorption lines of (a) the reference compound on the first and third scans and (b) the sample on the second and third scans. Hence, by successively applying the shifts indicated by these two error curves, tie in the three recordings to have the first scan of the reference compound as a common datum. The sample absorption lines can then be linearly interpolated from the two datum lines of the reference compound and a correction for systematic errors applied from the first error curve. Reproducible deviation of the reference lines from a linear calibration gives the systematic error; scatter about the error curve indicates the random error or positional tolerance of the measured absorption lines.

This technique is particularly valuable when precisely calibrating the rotational structure of the absorption bands of gases using abscissa expansion. A calibration accuracy of $\pm$ 1 cm^{-1} can be achieved over most of the range of the instrument and better than $\pm$ ½ cm^{-1} over part of the range. The following source of wavenumber standards is recommended:–

"Tables of Wavenumbers for the Calibration of Infrared Spectrometers", I.U.P.A.C., published by Butterworths, 1961.

Time Drive

33. This mode of operation is used to record the transmittance of a sample as a function of time at a fixed wavenumber and will usually be employed after the spectrum has been scanned by one of the previously described methods. Set the SCAN-MODE switch to 'T' and select the desired wavenumber position using the FAST FORWARD/REVERSE switch. The STOP and START buttons now energize the time drive motor which advances the chart at the rate of 10 cm/min, the wavenumber setting of the instrument remaining locked. This provides a continuous recording of transmittance as a function of time. Motor units giving alternative chart speeds are available. Note however, that in the event of change of the time drive motor, the abscissa expansion factors, 'x 2.5' and 'x 10' will be altered in proportion to the ratio of the motor speeds.

Flushing with Dry Nitrogen

34. Absorption by atmospheric carbon-dioxide or water normally does not affect transmittance readings in excess of the tolerances required for routine analysis. There may be occasions, however, when particularly accurate information is required in the wavenumber regions at which either of these substances absorbs strongly (4.1 to 4.5 and 14 to 16 microns for carbon dioxide and 2.6 to 2.8 and 5.5 to 7.0 microns for water vapour). On such occasions it is possible to reduce the

concentrations of these substances to relatively insignificant levels by flushing the optical path with dry nitrogen.
The following is the recommended procedure for flushing:–

a) Attach a 3/16 in i.d. tube from the dry nitrogen source to the fitting at the rear of the right-hand side of the instrument base.
b) Open the regulator valve at the nitrogen source and adjust the pressure to the instrument to be not more than 5 lb/in^2.
c) After approximately ten minutes reduce the pressure to 1 lb/in^2 and maintain it during measurements.

35. The following specification represents the maximum tolerance limits permitted in the manufacture of the Model 257 Spectrophotometer. The performance of each instrument will be equal to or better than the figure given below. Note that these specifications relate to the full range scan mode of operation. Measurements and calibrations are made at an ambient temperature of about 20 degrees, C.

SPECIFICATION

Principle	Double beam, optical null, linear transmittance versus linear wavenumber recording.
Optics	f/5 monochromator, two gratings, reflecting optical system employing planar and aspheric mirrors.
Chart Presentation	Chart size 21½ x 52 cm with a grid size 15 cm ordinate and 47½ cm abscissa.
Abscissa Range	Full Range: 4000 to 625 cm^{-1} with the grating change at 2000 cm^{-1}. Range 1: 4000 to 1250 cm^{-1} using the first grating only.
Abscissa Accuracy	$\pm$ 5 cm^{-1} at 3000 cm^{-1} $\pm$ 2.5 cm^{-1} at 1750 cm^{-1} $\pm$ 2 cm^{-1} at 625 cm^{-1}
Abscissa Repeatability	3 cm^{-1} at 3000 cm^{-1} 1.5 cm^{-1} at 1750 cm^{-1} 1 cm^{-1} at 625 cm^{-1}
Ordinate Range	0 to 100% Linear in Transmission
Ordinate Accuracy	$\pm$ 1% of full scale
Ordinate Repeatability	4000 to 2000 cm^{-1}: within ½% of full scale 2000 to 625 cm^{-1}: within 1% of full scale
Double Beam I_o	Flat within 4% of full scale. I_o change at 2000 cm^{-1} not more than 2% of full scale.

SPECIFICATION

Noise	4000 to 2000 cm^{-1}: not more than ½% of full scale 2000 to 625 cm^{-1}: not more than 1% of full scale
Resolution : Normal Programme Narrow Programme	4 cm^{-1} at 3000 cm^{-1} 2 cm^{-1} at 1000 cm^{-1} 2 cm^{-1} at 3000 cm^{-1} 1 cm^{-1} at 1000 cm^{-1}
Slit Control	The slit programme gives approximately constant energy over the complete wavenumber range and allows operation at energy levels differing by a factor of approximately 8.
Stray Light	Less than 1% from 4000 to 666 cm^{-1} Less than 2% at 645 cm^{-1} Less than 3% at 625 cm^{-1}
Scan Times	Approximately 5, 12 and 48 minutes for the full range scan including automatic grating change.
Pen Servo Response	Full scale deflection of the pen in 0.8 seconds with maximum signal to the servo motor.
Supply Requirements	210 to 250V, 50 c/s, 150W.
Overall Dimensions	70 x 46 x 38 cm
Weight	57 kg.

TEST SPECTRA

36. The instrument performance should be checked immediately after installation, and thereafter periodically, by comparing spectra from the instrument with the following test spectra.

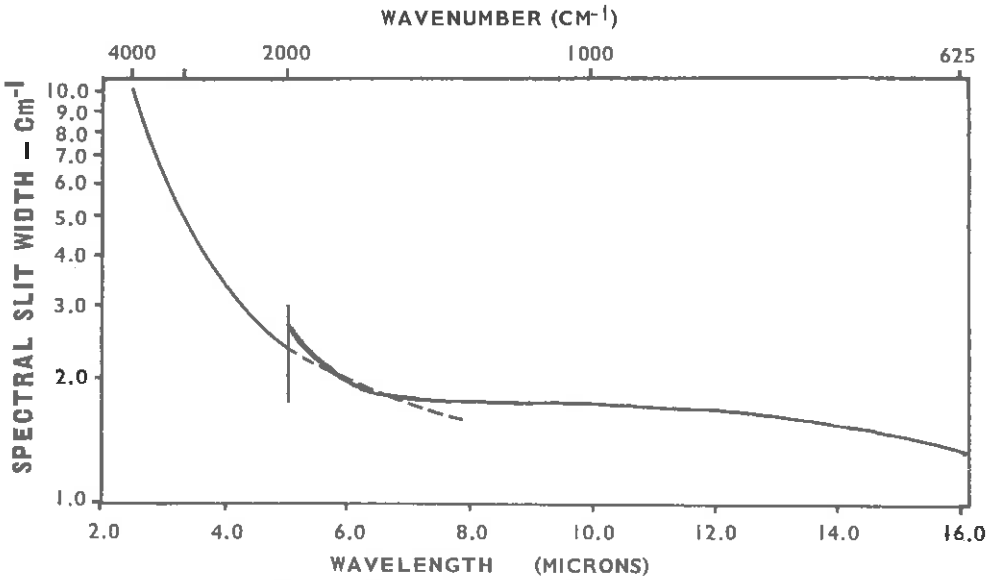
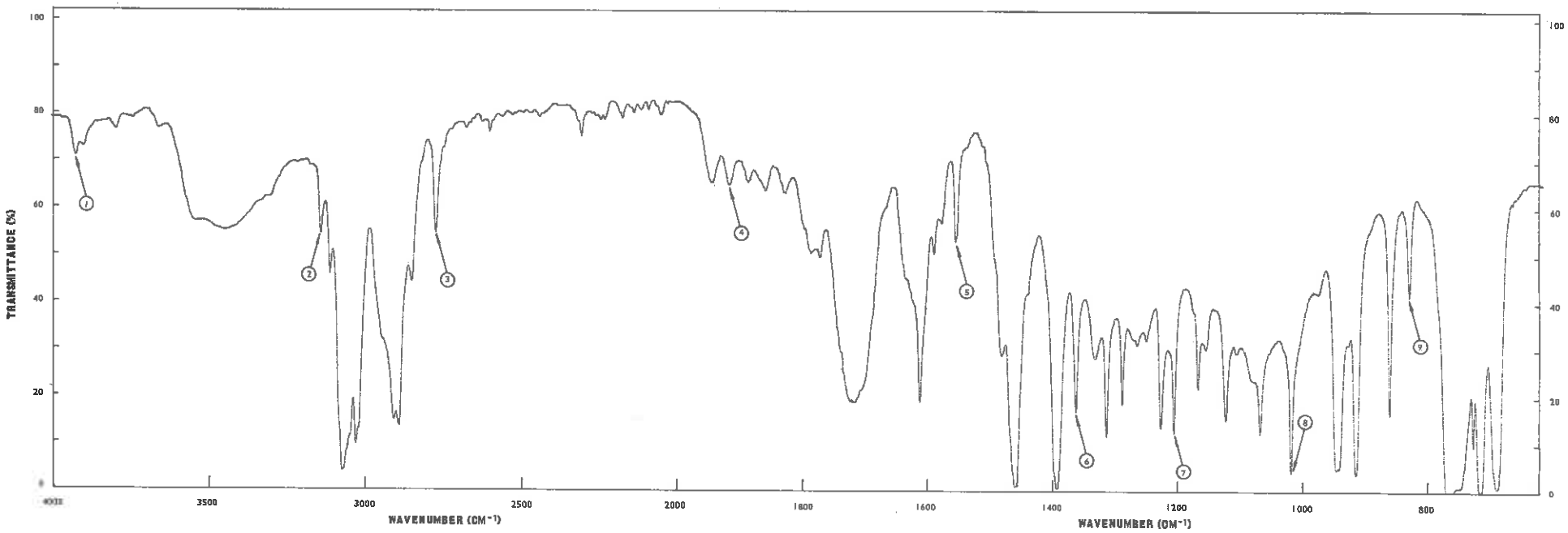
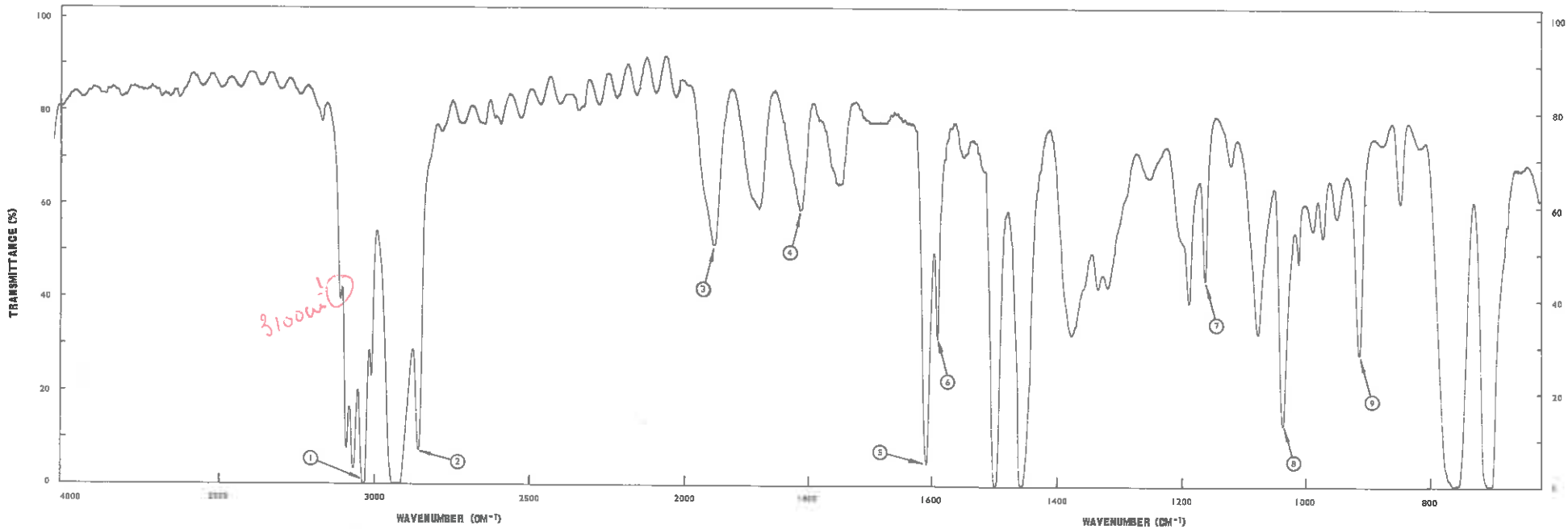


Fig. 2·10 Spectral Slit Width Curve



CALIBRATION PEAK	1	2	3	4	5	6	7	8	9
WAVENUMBER CM⁻¹ (VACUUM)	3827.2	3139.6	2778.9	1915.3	1663·2	1361.1	1205.1	1018.5	830.5

Fig. 2·11 Indene Calibration Spectrum. Normal Slit Programme.



CALIBRATION PEAK	1	2	3	4	5	6	7	8	9
WAVENUMBER CM^{-1} (VACUUM)	3027.1	2850.7	1944.8	1801.6	1601.4	1583.1	1154.3	1028.8	906.7

Fig. 2·12 Polystyrene Spectrum. Normal Slit Programme.

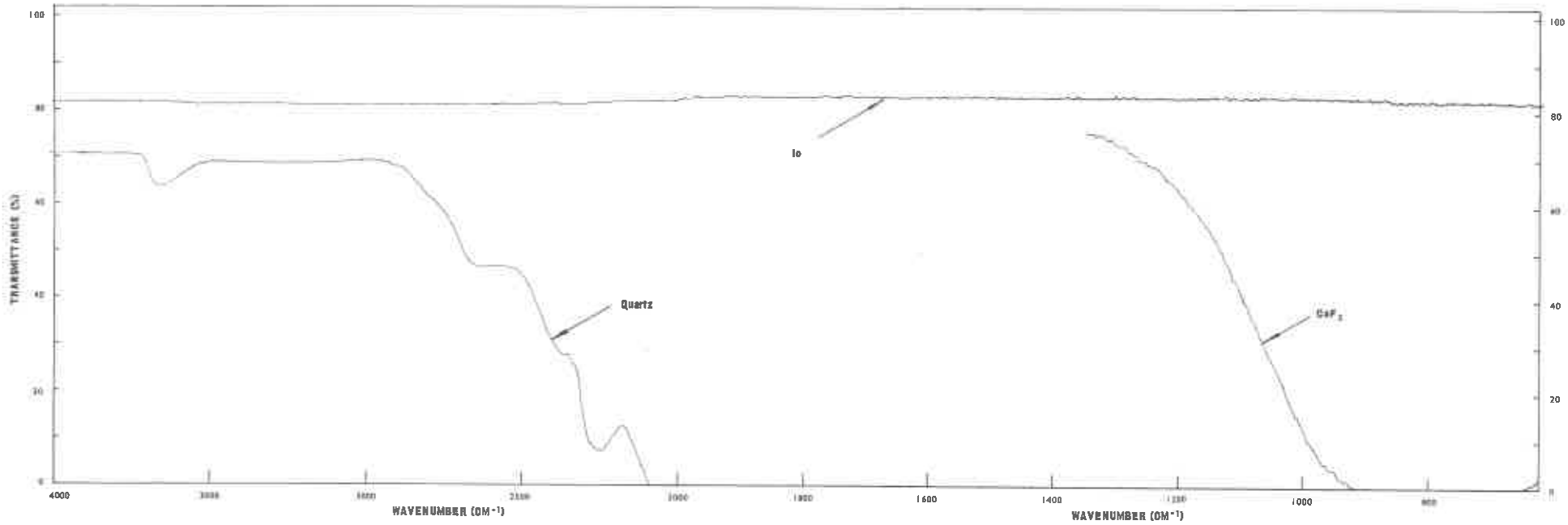
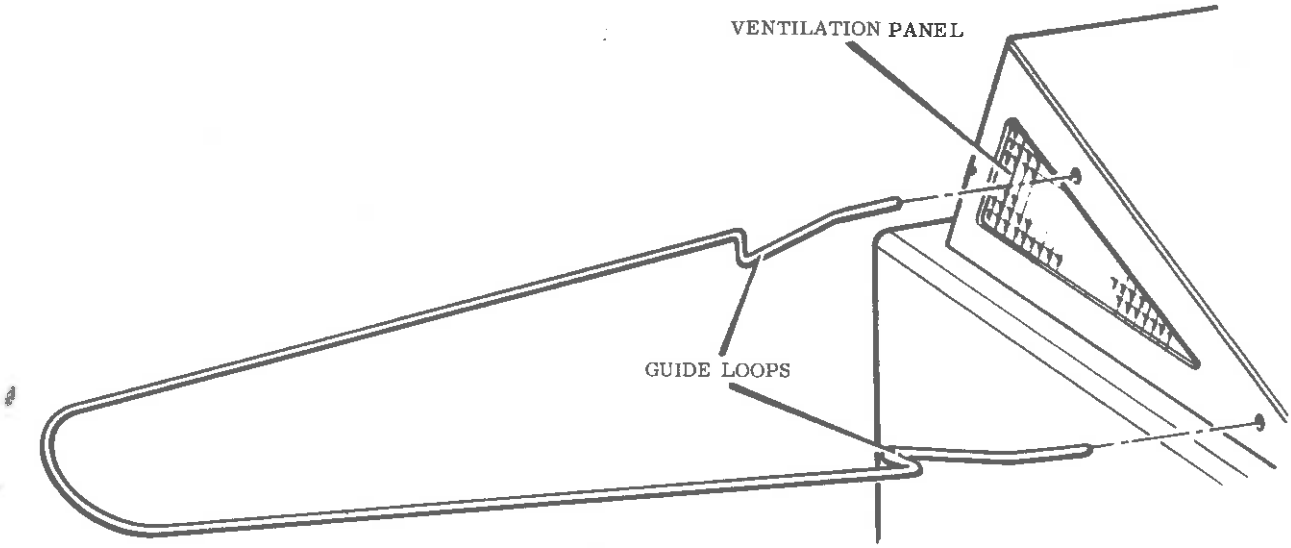


Fig. 2·13 Quartz and Calcium Fluoride Stray Light Run and I_0 Line. Normal Slit Programme.



INSERT WITH GUIDE LOOPS INCLINED DOWNWARDS

FITTING THE CHART SUPPORT

PERKIN - ELMER LIMITED, BEACONSFIELD, BUCKS., ENGLAND