ZA 003

INFRACORD

SPECTROPHOTOMETER

INSTRUCTION MANUAL

PERKIN-ELMER LIMITED, BEACONSFIELD, BUCKS.,
ENGLAND.

SECTION 1

INFRACORD
SPECTROPHOTOMETER
INTRODUCTION

PERKIN-ELMER LIMITED, BEACONSFIELD, BUCKS.,
ENGLAND.

INFRACORD SPECTROPHOTOMETER

INTRODUCTION

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ILLUSTRATIONS

INTRODUCTION

Fig.1.1 The Model 137 Infracord Spectrophotometer

GENERAL

1. The Perkin-Elmer Model 137 Infracord Spectrophotometer is a double-beam infrared instrument designed for laboratory use and covers the wavelength range 2.5 - 15 microns. It is manufactured, aligned and tested with the care and precision that has gained Perkin-Elmer world-wide recognition as manufacturers of scientific instruments.

2. In accordance with the general policy of the Company, improvements are constantly being sought and when developed are immediately built in to the instrument. For this reason the current Infracord Spectrophotometer differs in detail from earlier models although the principle of operation remains unchanged.

SCOPE OF MANUAL

3. The main object of this manual is to help users obtain the maximum performance from their instruments. This end is best achieved by a thorough understanding of how the instrument operates and the use of the correct operating procedures. No details of sample preparation are given since this subject is covered by separate publications.

4. A detailed theory of operation is given in Section 3, and this together with the correct setting up and performance checking procedures will enable competent

engineers to diagnose most faults. Resistance and voltage tables, giving figures which are typical for a standard instrument, are included but it is recommended that the engineer responsible for the instrument compiles individual tables for each instrument for subsequent comparison purposes.

5. Obscure faults, especially when misalignment of the optics is suspected, are best dealt with by contacting the Perkin-Elmer Service Department.

WHY INFRARED?

6. The Perkin-Elmer Infracord Spectrophotometer provides a means of applying one of the most powerful non-destructive analytical methods available today for identifying organic and inorganic compounds and determining their concentrations - infrared spectroscopy. It exposes materials placed in the sample chamber to infrared radiation and records the intensity of the transmitted radiation one wavelength interval at a time. The result, plotted against wavelength, is a transmittance spectrum of the sample which, as a measurement of a fundamental property of the material, can be used to characterize the material and determine the quantity present.

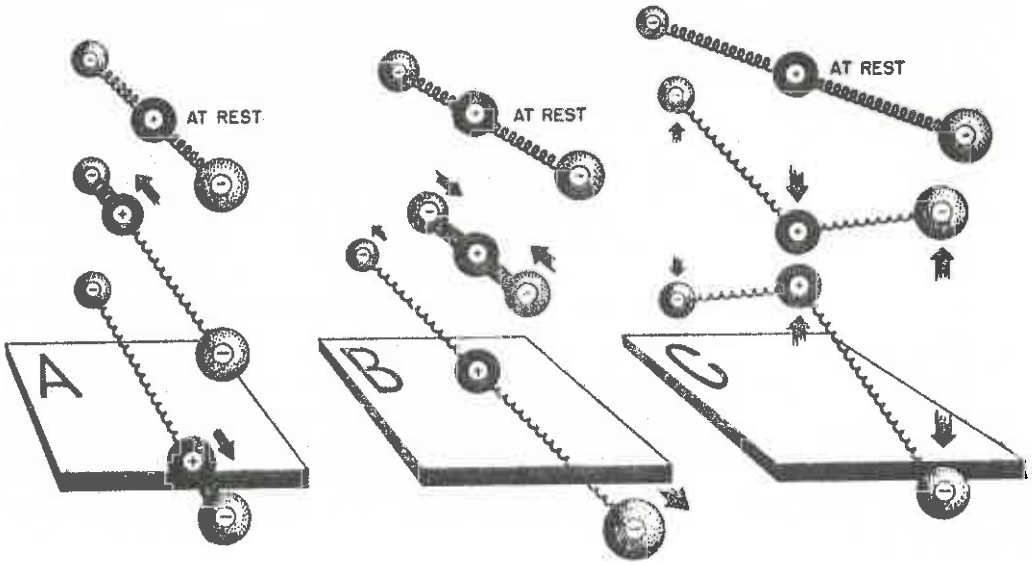

Fig. 1.2 Characteristic Vibrations of the CO_2 Molecule

(Note that Dipole Moment Changes in Vibrations A and C. Vibration B is Inactive - Dipole Moment does not Change).

7. The question might logically be raised as to the reasons for using infrared radiation. For an answer, it is necessary to examine the structure of the molecule. Greatly simplified, a molecule can be considered as a geometric arrangement of atoms united by chemical bond forces equivalent to small springs (see Fig. 1.2). Each atom can be represented by a minute sphere having a mass equal to the atomic weight. The result is analogous to a mechanical system which, were it subjected to an impact, would vibrate with apparently random chaotic motions. In actual fact, however, these motions consist of a discreet number of simple periodic vibrations, each with a definite frequency. For

nearly all molecules with N atoms, there are 3N-6 characteristic frequencies of vibration, though not all will be seen in an absorption spectrum.

8. The characteristic frequencies for a particular molecule are determined by the masses of the constituent atoms, their spatial geometry, and the strengths of the connecting bonds. Mathematical analysis using these data indicates that such frequencies lie in the range of 12×10^{13} to 0.6×10^{13} cycles per second (2.5 μ to 50 μ)*, the infrared frequency range. Thus, molecular vibration frequencies are of the same order as those of infrared radiation. The connection between the two is found in the electrical properties of each.

9. The atoms in a molecule have either positive or negative electrical charges. During the characteristic vibrations of the molecule, the atomic motions usually, though not always, cause a periodic change in dipole moment (see Fig.1.2). When electromagnetic energy of the same frequency (infrared frequencies) as that of the molecular vibration is incident on the molecule, there will be a resonant energy exchange in which radiant energy is absorbed by the molecule. (The degree of absorption is a function of the change in dipole moment which occurs during vibration). If the radiant frequency differs from a characteristic molecular frequency, the radiant energy will pass through the molecule undiminished.

10. A plot of the absorption of infrared energy versus the frequency of the radiation is an infrared spectrum. A spectrum, therefore, is really a graph of the mechanical vibrations of a molecule. Since these vibrations depend on the atomic masses, their spatial geometry, and the chemical bond forces (the three characteristics used by the chemist to differentiate one molecular species from another), the infrared spectrum is a unique characteristic of a molecular species. The degree of absorption is a measure of the quantity of a molecular species present in a sample.

GENERAL DESCRIPTION OF THE INSTRUMENT

11. The Infracord Spectrophotometer provides a continuous record of the infrared transmittance of a sample as a function of the wavelengths of the incident radiation. This is done by moving a pen vertically over a chart which is moved horizontally by a rotating recording drum. The rotational position of the drum relative to the pen designates a particular wavelength and the pen position along the drum axis is proportional to the transmittance at that wavelength. As shown in Fig.1.3, the scanning motor drive both the drum and the wavelength scanning mechanism. The drum position always corresponds exactly with the wavelength of radiation under observation.

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* In this manual, units of wavelength will be used wherever possible because wavelength is one of the co-ordinates of the Infracord chart. For the convenience of those who are more familiar with other terms the following conversions are given :-

$f = c\nu$ — f = frequency in cycles/sec.

$\nu = \frac{1}{\lambda} 10^4$ — c = speed of light in air = 3.0×10^{10} cm/sec.

$1\nu = 10^{-4}$ cm — ν = wavenumber = cycles/cm in cm^{-1} units. (u is sometimes used as the symbol for frequency)

$d\nu = -\frac{d\lambda}{\lambda^2} 10^4$ — λ = wavelength in microns (ν).

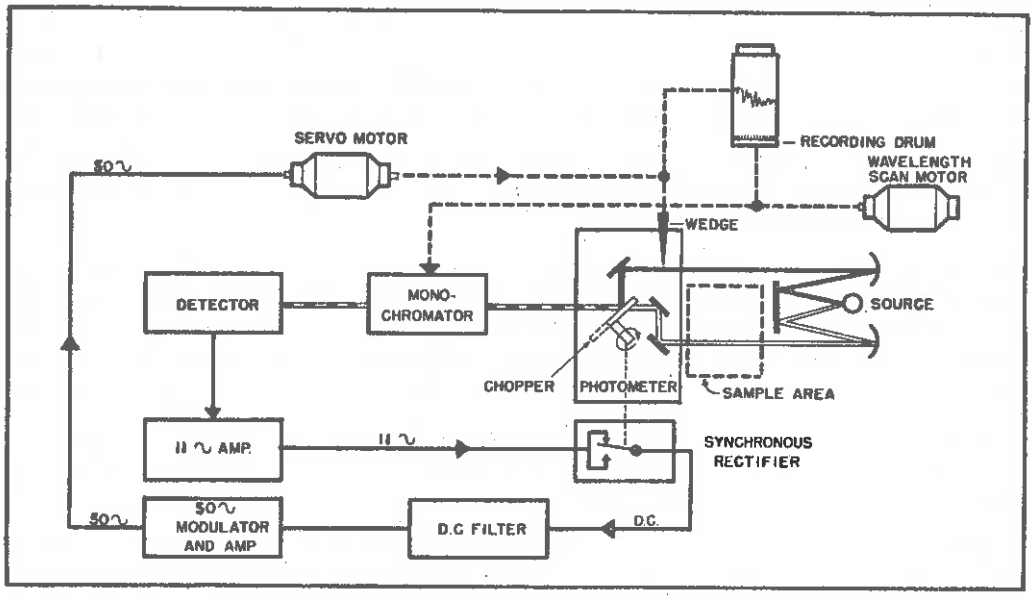


Fig. 1.3 Infracord Block Diagram

12. The radiation produced by the source is divided into two beams. One beam passes through the sample which absorbs certain characteristic wavelengths. The other serves as a reference. The two beams are recombined by a rotating semi-circular sector mirror into a single beam consisting of alternate pulses of sample and reference radiation. The monochromator disperses the combined radiation into its constituent wavelengths and transmits one wavelength at a time. If the energy in both beams is equal, the pulses from the two beams are equal in intensity. The result is that the thermocouple produces a direct voltage which is not amplified by the A.C. amplifier of the instrument.

13. When, at the characteristic wavelengths of the sample, the intensity of the sample beam is reduced by absorption, the two signals are unequal and the combined beam flickers at 11 c/s, (the rotational frequency of the semicircular sector mirror). The amplitude of the variation is proportional to the difference in intensity of the two beams, and the phase depends on which of the two beams has the higher energy. (It is possible for the reference beam to be less intense than the sample beam because of the optical null system employed).

14. The pulsating portion of the light beam is converted by the detector to an alternating voltage and amplified by the 11 cycle amplifier. This alternating signal is called the error signal and is proportional to the error between true transmittance and indicated transmittance. It is then converted to 50 c/s alternating voltage which operates the servo motor and moves the pen and an optical attenuator until the beam intensities are equalized. The function of the servo system, therefore, is to ensure that the error between true transmittance and indicated transmittance is negligible.

15. Note that the pen can be in any one of three states while the instrument is scanning. (1) It can be in a state of equilibrium, at a position on the chart indicative of a particular level of absorption which may, or may not, be zero. (2) It can be moving from

a chart position representing low absorption to one representing high absorption. (3) It can be moving from a chart position of higher absorption to one of lower absorption. The signals corresponding to each of these states are as follows:

1. Equilibrium - no error signal - intensities of sample and reference beams equal. (D.C. light signal is not amplified.)

2. Movement to chart position showing *greater absorption* - intensity of sample beam *less* than that of reference beam. (This is a transient condition which is minimized by the servo system.)

3. Movement to chart position showing *less absorption*- intensity of sample beam *greater* than that of reference beam. (This is also a transient condition kept to a minimum by the servo system).

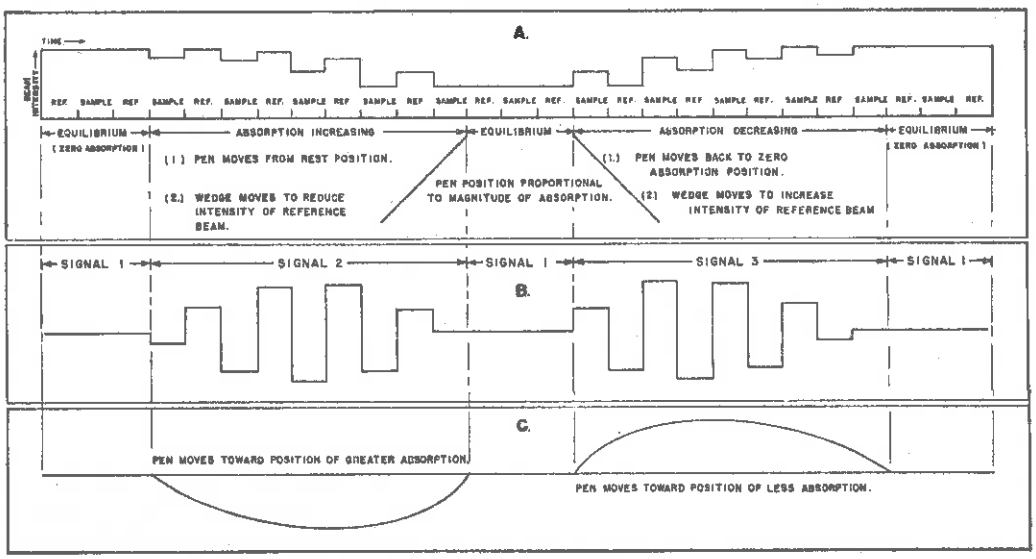


Fig. 1.4 Signal Waveforms - A. Signal at Thermocouple. B. Signal from 11c/s Amplifier. C. Signal from DC filter

16. Signal 1 (equilibrium) is brought about by nulling, or eliminating, any alternating system by making the intensity of the reference beam the same as that of the sample beam. This is done by changing the position of the optical attenuator, or wedge, in the reference beam (see Fig.1.3). Signal 2 results from increased energy absorption by the sample so that there is less energy in the transmitted sample beam than in the reference beam. Signal 3 is the result of a reduction of sample absorption which makes the intensity of the sample beam greater than that of the reference beam.

17. As can be seen, signal 3 is exactly 180 degrees out of phase with signal 2 (see Fig.1.4). It is this phase shift that causes the reverse rotation of the servo motor to drive the pen in the direction of lower absorption. Note that signals 2 and 3 are transient, while signal 1 represents a normal condition. The duration of signals 2 and 3 is kept short with respect to the duration of signal 1 by properly scheduling the scanning time from spectral interval to spectral interval.

SECTION 2

INFRACORD

SPECTROPHOTOMETER

OPERATING INSTRUCTIONS

PERKIN-ELMER LIMITED, BEACONSFIELD, BUCKS.,
ENGLAND.

OPERATING INSTRUCTIONS

CONTENTS

GENERAL

1. The Infracord Spectrophotometer is an extremely sensitive instrument and has been carefully aligned and fully tested before leaving the factory. In order to maintain 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 connected.

3. If preventative measures were not taken, damage could be caused to the monochromator window, prism and thermocouple window through condensation of water vapour under conditions of high relative humidity. This is prevented by:

   (a) Provision of a dessicant box, fitted in the base of the instrument below the sample tray, which keeps the monochromator compartment dry. The dessicant, accessible from the front of the instrument behind a small panel held by two Dzus fasteners, should be inspected at least once a month and replaced by the spare when necessary. The box removed can then be re-activated ready for subsequent use.

   (b) Fitting of a small heater below the monochromator window, to prevent fogging of its outside surface. This heater is operative when the power supply is connected. As heating of the window is especially necessary when the instrument is cold and not in use, it is important that the wall switch be left on at all times.

4. It is important to remember that all accurate measurements should be made with respect to the wavelength and transmittance scales on the pre-printed chart papers. The scales engraved on the instrument are only intended as a guide.

INSTALLATION

Preliminary

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

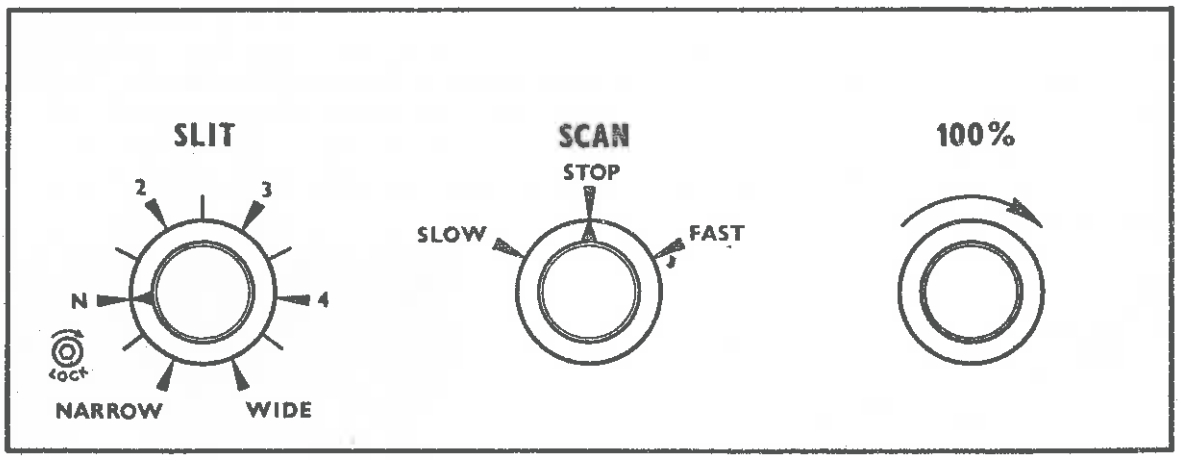


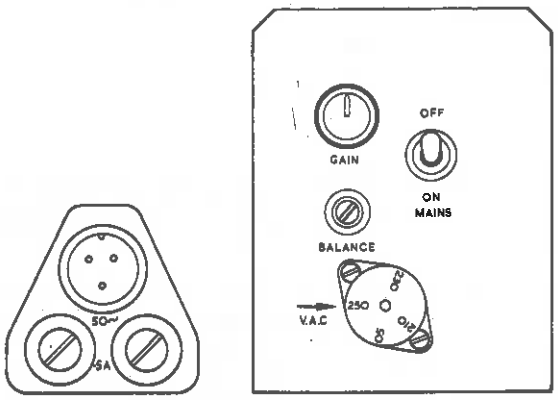
Fig. 2.1 Front Panel Controls

   (a) Place the instrument on a firm surface.

   (b) Check that the power switch on the side panel is set to OFF and the SCAN control on the front panel set to STOP. Ensure that the pen-holder is pulled back from the drum.

(c) Set the voltage selector (Fig. 2.2) at the side of the instrument to correspond with the supply to be used.

(d) Connect the power supply cable between the 50 c/s ONLY socket at the rear of the instrument and the mains power supply. The three-core cable must be used and the following colour code observed:-



Red - Live, Green - Earth

Blue or Black - Neutral

Fig. 2.2 Side Panel Controls

The power should be left connected to the instrument at all times except when servicing (see paragraph 3).

(e) Rotate the 100% control in a counterclockwise direction several complete turns. This control rotates easily and NO FORCE should be used.

(f) Release the locking screw near the SLIT control with an Allen key and rotate the control to the N position.

NOTE: When the instrument is to be moved the SLIT control should be locked in the WIDE position, the drum set to 15 μ and the SCAN control set to SLOW.

Preparing Pen and Fitting Chart Paper

6. Two types of pen (not interchangeable) have been fitted to the Infracord. The reservoir of the pen should be filled with ink as shown in Fig. 2.3a or 2.3b. Should the pen fail to write due to a block in the capillary tube, it may be cleaned by using a hypodermic syringe to flush the tube with warm water (pen shown in Fig.2.3a) or by inserting the cleaner wire supplied (pen shown in Fig.2.3b).

7. As the pre-printed chart papers are cut to exactly the right size, no difficulty should be experienced in fitting a fresh chart in a matter of seconds. Figures 2.4 and 2.5 show the correct manner in which the chart drum should be held. To insert the chart paper under the clamping spring, depress the locking lever on the side away from the chart. Adjust the paper so that the index lines on the chart and the drum coincide and so that the top of the paper is flush with the underside of the drum cap. Release the locking lever.

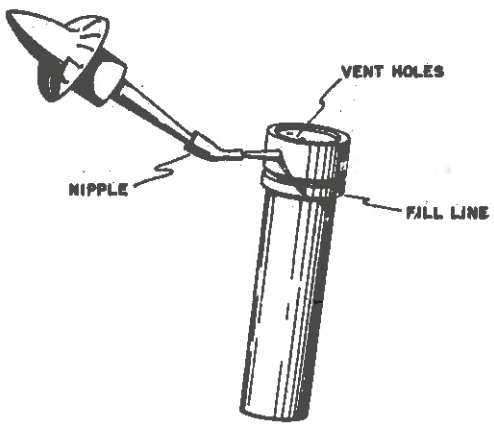
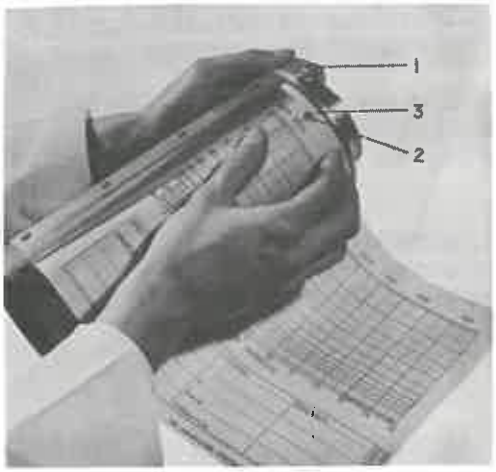


Fig.2.3a

Filling Pen Reservoir

Fig.2.3b

8. The drum should then be rotated in the hands whilst the paper is smoothed down to ensure that it is free from wrinkles. To secure the end of the chart paper, depress the locking lever with the right thumb as shown in Fig. 2.5 and guide the paper beneath the clamping spring. Release the locking lever.



1. Lever
2. Index Line (Drum)
3. Index Line (Paper)

Fig. 2.4 Fitting Chart Paper

Fig. 2.5 Securing end of Chart Paper

FUNCTIONS OF CONTROLS

General

9. The OFF/ON, GAIN and BALANCE controls are located on the left-hand side of the instrument (Fig. 2.2) and the SCAN, 100%, and SLIT controls are located on the front panel (Fig. 2.1). All controls operate easily and NO FORCE should be used.

Off/On Switch

10. This is the main power switch of the instrument and should be set to ON approximately fifteen minutes before it is desired to use the instrument in order to warm up the amplifier. The monochromator window heater is on even when this switch is in the OFF position, provided the electrical supply is connected to the instrument.

Gain Control

11. The GAIN control varies the gain of the amplifier and its function is to adjust the response of the pen to the optimum value. Adjustment will be necessary periodically to compensate for the ageing of electronic components and will also be necessary when the setting of the SLIT control is altered. For example, if the slits were opened wider than N (normal setting), say to position 3, and there were no compensating sample in the reference beam, the energy reaching the thermocouple would be some three times greater than normal. The GAIN control would then need to be adjusted to reduce the loop gain of the servo system to prevent excessive over-shoot or oscillation of the pen.

Balance Control

12. 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. The pen balance should be such that, with no energy passing through either the sample or reference beams, the pen will remain stationary.

Scan Control

13. The SCAN control has three positions designated, SLOW, STOP and FAST. The function of the control in each of these positions is as follows :-

SLOW - In this position the scanning motor drives the drum, causing the monochromator to scan the complete wavelength range of 2.5 - 15 μ in approximately twelve minutes.

STOP - When the control is in the STOP position the mechanical drive is disconnected and the operator is able to rotate the drum manually to any desired position, thereby setting the instrument to the required wavelength.

FAST - In this position of the SCAN control, conditions are exactly the same as in the SLOW position except that the complete wavelength range is scanned in approximately three minutes. (In some earlier models, this time is six minutes).

100% Control

14. The 100% control provides a manual means for controlling the intensity of the sample beam. When the knob is turned clockwise, the comb in the sample beam is moved out of the beam and the pen moves upscale.

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.

Slit Control

15. To give 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. Compensation for changes in the energy radiated by the source over the wavelength range is made by varying the widths of the monochromator slits. The programmed rate at which the slits open or close is the same for all settings of the SLIT control, but the level of slit width and hence the amount of energy that is transmitted, is different for each setting of the control. The SLIT control setting used will depend on the particular application, as discussed in paragraphs 33 and 34. The meaning of the graduations around the SLIT control is as follows :-

NARROW - In the NARROW position the programmed slit widths are at their minimum value. This setting gives maximum resolution but, as the energy level is low, a high gain setting is required to provide adequate pen response and this leads to an increase in the noise level.

N - In the N, or normal, position the level of the slit programme has been chosen to give a signal-to-noise ratio of not less than 100:1. This gives a good level of resolution without undue noise on the recording.

2, 3, & 4 - At these settings approximately two, three and four times the energy in the N position is transmitted, thus allowing a progressive reduction of the noise level, but at some cost in resolution.

WIDE - Approximately five times the normal energy is available. This allows the lowest gain setting to be used and consequently the lowest noise level.

NOTE The Allen key locking screw adjacent to the SLIT control enables the control to be locked at any position between NARROW and WIDE. The control should be locked whenever a constant energy level is required over a long period of time and when the instrument is to be transported.

PRE-SET CONTROL ADJUSTMENTS

Preliminary Steps

16. The following preliminary steps should be taken before any spectral run or adjustment of pre-set controls.

Fig. 2.6 Correct Way to Rotate Drum Manually

(a) Fill the pen reservoir with ink and fit a chart paper to the drum as described in paragraphs 6, 7 and 8.

(b) Check that the SCAN control is at STOP and turn the drum to the start position, holding it as shown in Fig. 2.6.

NOTE : The SCAN switch must always be in the STOP position when the drum is manually operated, or damage may result to the drum drive mechanisms.

(c) Switch the OFF/ON switch to the ON position and allow fifteen minutes for the amplifier to warm up.

General

17. The time interval between checks on the settings of the pre-set controls depends entirely on the nature of the analyses to be carried out. Experience will soon show how often each control needs to be checked. Until such experience is acquired the GAIN and BALANCE controls should be checked before every spectral run and the ZERO control should be checked once a week. If adjustment is necessary, the procedure detailed below should be followed.

Gain Control

18. The amplifier GAIN control determines the amount by which the signal to the pen servo motor is amplified. The greater the gain, the greater the voltage to the pen servo motor for a given out of balance signal, and the higher the speed of response of the pen. The GAIN control is located on the side panel of the instrument and should be adjusted as follows :-

(a) Set the drum manually to 10 μ. In order to conserve chart paper, the pen may be lifted from the chart and pen movements checked by observing the pointer over the engraved transmittance scale on the pen carriage.

(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 - pen overshoots original position on return by more than 1% of full scale. Turn GAIN control counter-clockwise to decrease gain.

CORRECT GAIN - pen returns quickly to original position with about 1% (of full scale) overshoot.

GAIN TOO LOW - pen moves sluggishly and does not return immediately to original position, indicating "dead spot". Turn GAIN control clockwise to increase gain.

19. 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

20. 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 upward. The correct procedure for adjusting the BALANCE control is as follows:-

(a) Set the drum manually to 10 μ.

(b) 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 both beams simultaneously as any time lag will cause a temporary pen drift which must be allowed to settle.

Manually move the pen holder slowly to the middle of the Transmittance scale. The pen should either remain in position or move upward with a very slow upscale drift.

(c) 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 will be required if any considerable change in the BALANCE control setting has been necessary.

Zero Adjustment

21. 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) With the pen off the chart paper and with the SCAN control in the STOP position, manually rotate the drum to the 10 μ region.

(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 moves only extremely slowly upscale. If the pen is out of balance, adjust as described in paragraph 18.

(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 $\frac{1}{2}$% Transmittance per second. Do not move the cardboard again until the pen has stopped moving.

(d) When the pen has stopped moving, 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) Having made sure that the pen is seated correctly in its holder, press the pen against the chart paper and check that the mark it makes corresponds with the zero on the chart paper. If the pen setting is not correct, remove the pen cover by releasing the screw at the top of the Transmittance scale, lifting the cover slightly and pushing the base of it backwards to release the spring-clip fastener. (To avoid hitting the pen wires it is advisable to turn the pen cover in a counter-clockwise direction as it is being lifted clear). The pen adjustment screw (Fig. 2.7) may now be loosened and the pen holder moved along the drive wire until it is correctly positioned. Tighten the pen adjustment screw.

(g) Repeat steps (b) to (f) to confirm that the setting is correct and then replace pen cover.

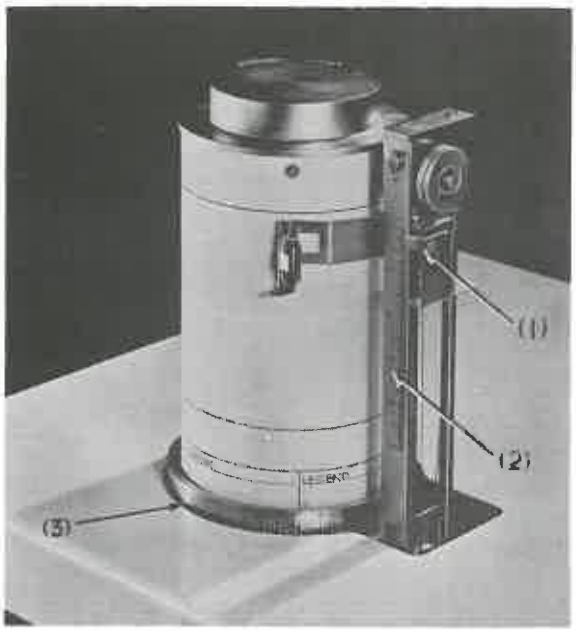


Fig. 2.7 Infracord Recorder Drum

1. Pen Adjustment Screw
2. Transmittance Scale
3. Wavelength Scale

100% Adjustment

22. At 100% Transmittance the energy in both the sample and the reference beams is equal and at maximum intensity. The 100% adjustment control permits balancing of any slight inequalities between the two beams. Adjustment should be made as follows :-

Starting with the 100% adjustment knob at

its limiting counter-clockwise position, and with no obstruction in either beam, slowly rotate the knob in a clockwise direction until the pen reached the 100% line *on the chart.* The control is now correctly set.

REMINDER All accurate readings of Transmittance and wavelength must be made with respect to the scales printed on the chart paper.

TEST RUNS

Polystyrene Spectrum

23. A polystyrene test film is provided with the instrument together with a polystyrene spectrum for comparison purposes. Assuming that the installation and setting-up instructions detailed earlier in this section have been carefully followed, a test run may now be made.

24. Having set the chart drum by hand to the beginning of the wavelength scale (2.5 μ) and allowed 15 minutes for the instrument to warm up, insert the polystyrene film in the adapter provided and place adapter over the sample beam window. Push the pen into contact with the chart paper.

25. Set the SLIT control to N (normal), and put the scanning switch to the SLOW position. The instrument will now automatically plot Transmittance with respect to wavelength to give a spectrum characteristic of polystyrene.

26. After approximately 12 minutes the instrument will automatically stop scanning at about 15 μ. Pull the pen back from the chart paper and set the scanning switch to STOP. The drum may now be lifted from the instrument and the chart paper removed.

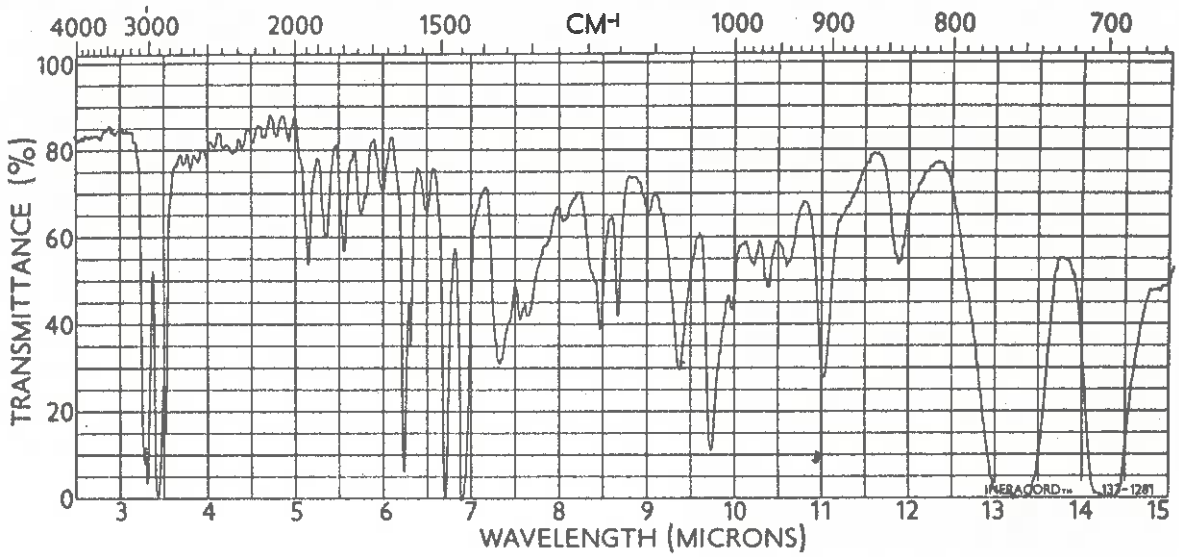


Fig.2.8 Typical Polystyrene Spectrum

27. Compare the polystyrene spectrum obtained with the example provided, or that shown in Fig.2.8. The following points should be noted :-

(a) The side bands at 3.5 μ and 6.3 μ give representative examples of the resolution that can be expected. These bands should be distinct and well-defined within the limits of the thickness of the pen line.

(b) The Transmittance under the broad band at 14.3 μ should be 3% or less. Radiation re-emitted by the sample can account for up to 2% Transmittance and the remainder is due to the scattered or stray light. (A more accurate measurement of the stray light in the 12 - 15 μ region can be obtained by measuring the transmittance of a calcium fluoride shutter, see paragraph 31).

(c) The polystyrene spectrum can be used to check the reproducibility of the instrument. This can be done by re-running the spectrum on the same chart paper before it is removed.

(d) Interference bands are responsible for the weak bands at 2.5 - 5 μ and will be superimposed on the absorption spectrum in other regions. The amplitude and spacing of the interference bands, being dependent on the evenness and thickness of the film, will vary from sample to sample and cannot be used to measure the resolution of the instrument.

100% Transmittance (I_0) Line

28. Rotate the drum manually to the beginning of the wavelength scale and turn the 100% adjustment knob to give a Transmittance reading of 90%. With the SLIT control in the N position, push the pen into contact with the chart paper and put the SCAN control in the FAST position.

29. When the instrument has scanned from 2.5 μ to 15 μ and stopped, pull the pen back from the chart paper and move the SCAN switch to the STOP position. Remove the chart paper from the drum and compare the I_0 line with the example shown in Fig.2.9. The peak-to-peak noise signal of the instrument should not be greater than 1% of the full scale reading and the Transmittance line should be flat to within ± 2% from 2.5 μ to 15 μ wavelength.

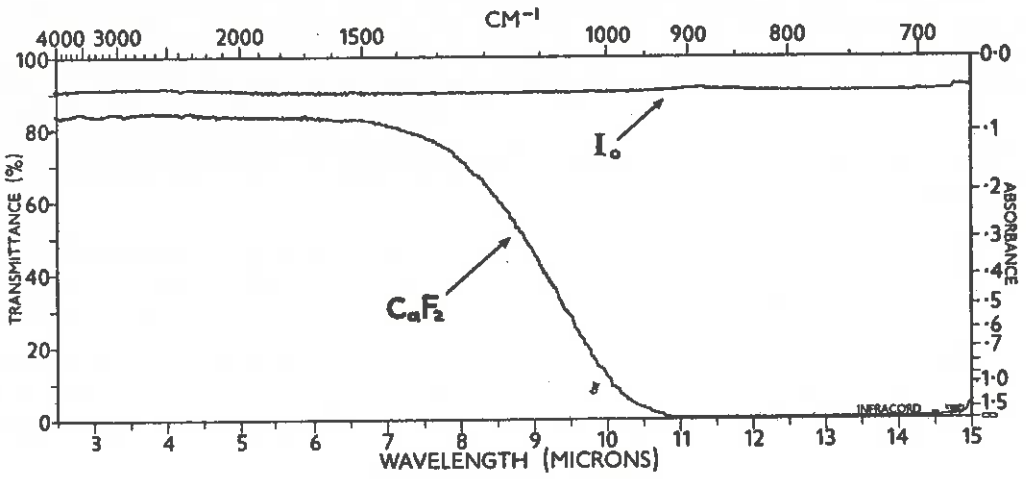


Fig.2.9 Typical I_0 Line and CaF_2 Spectrum

30. Fit a fresh chart paper ready for the next run and readjust the 100% control as described in paragraph 22.

Calcium Fluoride Spectrum

31. An example of a calcium fluoride spectrum is shown on the same chart as the I_0 line in Fig. 2.9. A 5 mm CaF2 shutter should be placed in the sample beam and the spectrum should be run with the SCAN switch set to FAST and the SLIT control set to N. The signal due to scattered light picked up by the detector under these conditions should not be more than 3% of full scale at 14.8 μ wavelength and less than 1% below 14 μ.

SPECIAL OPERATING PROCEDURES

General

32. For most analyses the operating procedure will be as described for the polystyrene test run (Paragraphs 23-27) except that the FAST scan may be used instead of the SLOW scan. The twelve minute scan (SLOW) has been chosen to allow the instrument time to respond fully even on the sharpest bands and should be used for all quantitative work; the FAST scan has been provided for rapid survey work. The error introduced by the higher scanning speed will be greatest for sharp bands but for certain compounds with broad bands the spectra produced with the FAST scan will be virtually identical to those produced with the SLOW scan.

Variable Slit Control

33. As will have been inferred from the description of this control in paragraph 15, high resolution may be obtained at the expense of a low noise level and *vice versa*. The generally accepted compromise is to work at a noise level not exceeding 1% and this is obtained when the SLIT control is in the N position. For certain applications however, e.g. when attempting to separate two overlapping bands, the high resolution of the NARROW position will be preferred in spite of the higher noise level.

34. The wider slit programmes offered by positions 2, 3, 4 and WIDE have two special applications:-

(a) For quantitative analyses, where some sacrifice in resolution is generally acceptable in order to allow a lower gain setting. The resultant lower noise level gives more accurate and reproducible intensity measurements.

(b) In differential analyses, to compensate for the reduction in intensity caused by absorption in the reference beam. The gain control is left in its normal position and the slits opened until satisfactory response is obtained. If the slits were not opened, satisfactory response could only be obtained at impossibly high gain settings.

NOTE The GAIN and BALANCE controls must be checked and if necessary readjusted whenever the SLIT control setting is altered, or an absorbing sample placed in the reference beam

Fig.2.10 Chart Drum Selection Mechanism

1. Selector
2. Tab
3. Drum Base Plate
4. Index Pin
5. Engaging Pin
6. Wavelength Scale

Variable Drum Setting

35. The base of the chart drum has twelve-position selector which permits changing of the setting of the drum relative to the wavelength scale of the instrument, (Fig.2.10). By using the selector tab to turn the selector in either direction any of the settings may be selected. This facility enables small regions of a spectrum to be scanned a number of times and each scan to be displayed on the same chart.

NOTE: There is only one setting at which the wavelength scale on the chart paper corresponds with that on the instrument. This setting is marked N.

Manual Scanning

36. Manual scanning is useful when it is desired to scan a wavelength range rapidly to determine the wavelengths of major absorption bands. With the SCAN control in the STOP position the pen may be pushed against the chart paper and the chart drum rotated manually whilst held as shown in Fig.2.6. In this way any wavelength range may be scanned as rapidly as desired; in most cases an adequate spectrum can be obtained in as short a time as one minute.

Flushing with Dry Nitrogen

37. 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 wavelength regions at which either of these substances absorbs strongly (4.1 to 4.3 microns for carbon dioxide and 5.5 to 7.0 microns for water). 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:-

(i) Attach a 3/16 inch I.D. tube from the dry nitrogen source to the fitting at the rear of the right-hand side of the instrument base.

(ii) Open the regulator valve at the nitrogen source and adjust to give a flow to the instrument of not more than 5 p.s.i.

(iii) After approximately ten minutes reduce the pressure to 1 p.s.i. and maintain the flow during measurements.

SPECIFICATION

38. The following specification represents the maximum production limits permitted in the manufacture of the Infracord spectrophotometer. (Measurements and calibrations are made in an ambient temperature of 70°F.) The performance of each instrument should be equal to, or better than, each specification.

WAVELENGTH RANGE - The Infracord spectrophotometer scans the infrared spectrum in the region from 2.5 μ to 15 μ. (4,000 cm^{-1} to 667 cm^{-1}).

ORDINATE - Linear Transmittance (non-linear Absorbance).

WAVELENGTH ACCURACY - A wavelength reading on the wavelength scale is not different

by more than $\pm$ 0.02 μ from the wavelength actually being scanned by the instrument. Wavelength calibration will be affected at a rate not exceeding 0.02 μ per 16°F change in the ambient temperature.

WAVELENGTH REPRODUCIBILITY - The wavelength readings registered on the wavelength scale are identical within 0.02 μ from one run to the next. (True only if the spectrum is automatically scanned at the slow speed).

ORDINATE ACCURACY - The readings on the scale of Transmittance, for any transmitted fraction of full scale energy, indicate the true value of Transmittance within $\pm$ 1% of full scale.

ORDINATE REPRODUCIBILITY - Transmittance readings do not differ by more than 1% of full scale for identical Transmittance from one run to the next. (This assumes that the sample used is constant in concentration and thickness).

NOISE - The peak-to-peak noise signal of the instrument is not greater than 1% of the full scale reading at normal slit and gain settings.

RESOLUTION - The minimum wavelength interval which can be distinguished by the exit slit of the monochromator is not greater than 0.04 μ at a wavelength of 10 μ.

DOUBLE BEAM I_O (100% LINE) - The 100% Transmittance line is flat to $\pm$ 2% from 2.5 μ to 15 μ wavelength. (No sample in either sample or reference beams)

SCATTERED LIGHT - The energy signal due to scattered light picked up by the detector as measured with a 5 mm CaF_2 shutter in the sample beam is no more than 3% full scale at 14.8 μ wavelength and less than 1% from 2.5 μ to 14 μ.

SLIT PROGRAMME - The slit programme gives constant energy over the wavelength range and allows operation at energy levels differing by a factor of approximately 10.

SCANNING SPEED - The complete spectrum is automatically scanned in approximately 12 minutes at the SLOW speed and approximately 3 minutes at the FAST speed (6 minutes in some early models).

SERVO RESPONSE - The pen moves from zero to full scale in 1.2 seconds with a maximum signal to the servo motor (slewing). The pen response becomes sluggish in the wavelength range 4.1 μ to 4.3 μ because of absorption by atmospheric CO_2. (This condition can be improved by flushing the optical path with dry N_2 or by increasing the slit width.

POWER REQUIREMENTS - 200-250 volts, 50 cycles per second, 0.5 - 0.6 amperes. In keeping with good practice with regard to electrical equipment the instrument must be earthed electrically. (In addition to being a safety precaution, earthing of the instrument improves its noise characteristic).