IBM Instruments Inc.

9400 Series UV/Visible Spectrophotometer Service Manual

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IBM Instruments Inc.

9400 Series UV/Visible Spectrophotometer Service Manual

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1.0 INTRODUCTION

1.1 TECHNICAL SKILLS REQUIRED

The IBM Instruments 9400 Series UV/Visible Spectrophotometers are sophisticated electro-optical instruments. To maintain their high level of performance, anyone who is engaged in service, repair, or calibration of the instrument should have an optical and electronic background, with experience in servicing analytical instruments. This manual is written for such skilled people. A substantial effort has been made to ensure adequate illustration of all special requirements and peculiarities. This document does not contain tutorial discussions of elementary electronic and optical practices and precautions.

2.0 MAJOR SUBSYSTEM DESCRIPTION

The instrument consists of an optical system, an electronic signal processing system, and a control system.

2.1 OPTICAL SYSTEM

The optical system of the 9410 and 9420 instruments is shown in Figure 2-1. The optical system of the 9430 instrument is shown in Figure 2-1A. Either system can be broken down into five subsystems:

a) Light Source Group
b) Monochromator Group
c) Beam Switching Group
d) Sample and Reference Stage
e) Detector Group

The combination of c, d, and e is called the photometer.

2.1.1 The Light Source Group

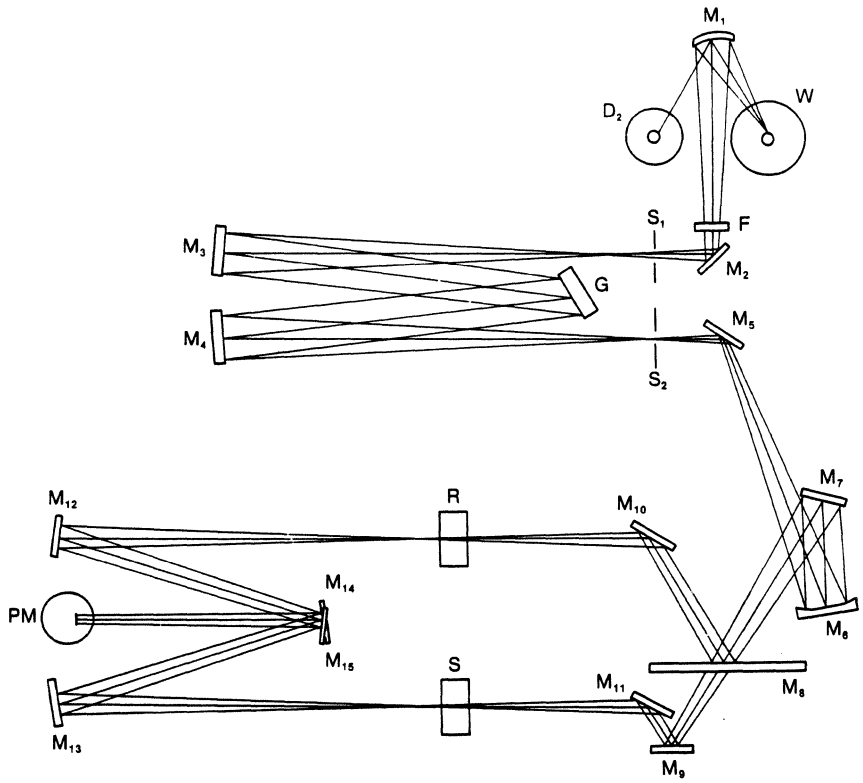
Two separate sources are used to cover the appropriate spectral range. A tungsten lamp ("W Lamp") is used for the wavelength range, 910 to 340 nm. For the range below 340 nm, where the W lamp is very weak, a deuterium lamp ("D_2 Lamp") is used.

Selection of one of the two sources is done by mechanically switching the horizontal alignment of a spherical (condenser) mirror, M_1, which concentrates the selected source into the monochromator.

2.1.2 The Monochromator Group

The monochromator group is comprised of the following elements:

a) Filter, F
b) Folding Mirror (Plane), M_2
c) Concave Grating (9430 only), G_1
d) Entrance Slit, S_2
e) Collimator (Mirror), M_3
f) Diffraction Grating, G
g) Focusing Mirror (inverse collimator), M_4
h) Exit Slit, S_2



D_2: Deuterium lamp

W: Tungsten lamp

M: Mirror

F: Filter

S_1: Entrance slit

S_2: Exit slit

G: Diffraction grating

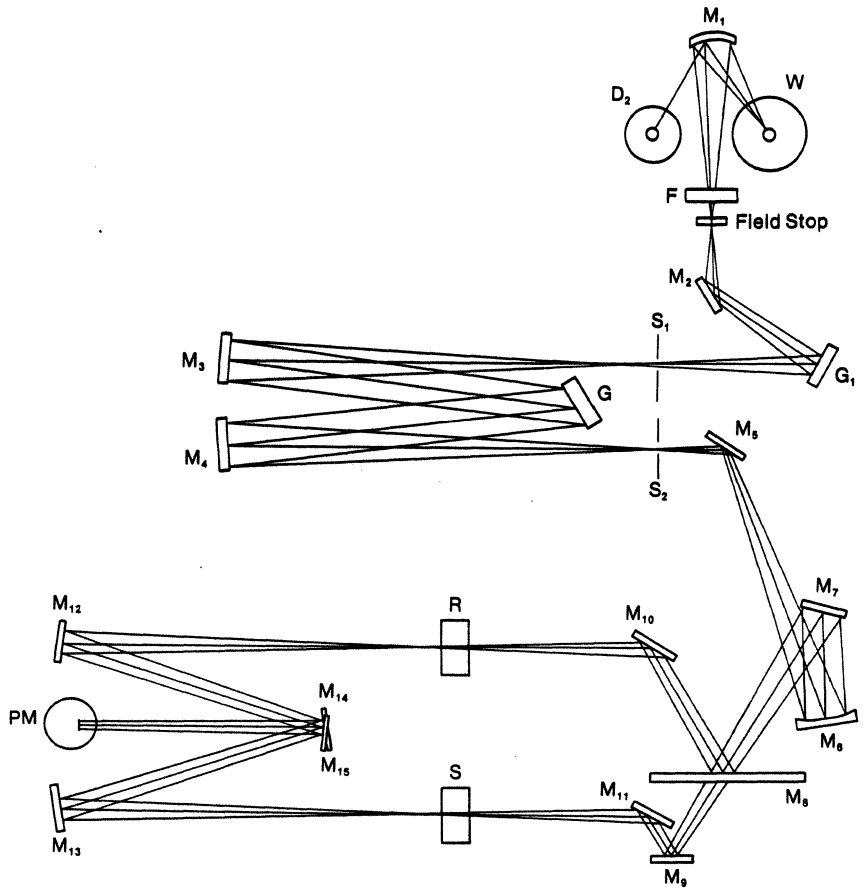
M_8: Sector mirror

S: Sample cell

R: Reference cell

PM: Photomultiplier tube

Figure 2-1. Optical System of 9410 and 9420



D_2: Deuterium lamp

W: Tungsten lamp

M: Mirror

F: Filter

S_1: Entrance slit

S_2: Exit slit

G: Diffraction grating

M_8: Sector mirror

S: Sample cell

R: Reference cell

PM: Photomultiplier tube

Figure 2-1A. Optical System of 9430

The filter, F, is a spectral pre-filter, which helps reduce stray light, undesired orders, and ghost spectra produced by the grating(s). It does this by reducing the spectrum of the source radiation. It is actually a family of filters and open spaces, which are switched as the spectrum is scanned.

All the 9400 series instruments use the yellow, order sorting filter for the long wavelength end of the spectrum. The 9410 and 9420 also use a blue, stray light filter. In the 9430, the concave grating, G_1, acts as a super-stray light filter.

The last five optical elements (d through h) comprise a Czerny-Turner type monochromator. M_3 receives diverging rays from S_1, collimates them, and directs them to the grating, which in turn delivers a collimated beam to M_4, but with each wavelength at a different angle. Changing the angle of the grating changes the alignment of all the wavelengths. The wavelength which is centrally aligned, will be focused by M_4 onto S_2. The amount of light passing through S_2 at wavelengths adjacent to the central wavelength, is determined by the width of both S_1 and S_2.

2.1.3 Beam Switching Group

The switching group consists of mirrors, M_5 through M_{11}. All of these, except M_6, are folding mirrors, which merely change the direction of the beam. M_8, the sector mirror, is continually rotated in and out of the beam, so that the beam is folded onto M_{10}, whenever one of the two M_8 sectors is in the beam.

When one of two open sectors is in the beam, it allows the beam to pass to M_9 to be folded onto M_{11}. Two other sectors are absorbing blockers, so that the dark current of the photomultiplier tube (PMT) can be monitored. M_6 is a spherical mirror which images S_2 onto the Reference and Sample cells, R and S. Thus, the beam is continually switched sequentially among three states, Reference beam, Sample beam, and "OFF," for dark current.

2.1.4 Sample and Reference Stage

As an optical element in each of the two beams, the stage represents a substantial variable. With nothing in the cell holders, it is an optical path in air. A real sample will, in general, demonstrate absorption of light, which is different at each wavelength. This, of course, provides the central value of the instrument.

As the instrument measures the amount of light absorbed in the sample, the most common extraneous effect is reflection from surfaces of the cell containing the sample. This effect is avoided if a reference cell is placed in the reference cell holder, which contains the solvent used with the sample. Then the reference beam will also experience the same surface reflection losses, and the sample data can be normalized to the reference.

Not shown in Figure 2.1 are four windows which interface the Stage with the Beam Switching Group upstream, and the Detector Group downstream. These windows ensure that the optical path outside the sample chamber is free from contamination. These windows are subject to reflection losses, but should be well balanced (reference vs. sample). However, it is imperative that they be kept clean, to ensure this balance.

2.1.5 Detector Group

This group consists of four mirrors, M_{12} through M_{15}, and the PMT. M_{12} and M_{13} are spherical mirrors which condense the two beams to image size on the PMT, after being folded by M_{14} and M_{15}. M_{14} and M_{15} are mounted one over the other, and they place the two images superimposed, on the PMT. The sequential nature of the images allows separate measurement. The electronic output of the PMT is a multiplexed composite of three measurements: sample beam, reference beam, and dark current.

2.2 ELECTRONIC SIGNAL PROCESSING AND CONTROL SYSTEMS

Figure 2-2 shows the block diagram of the electronic signal processing and control function.

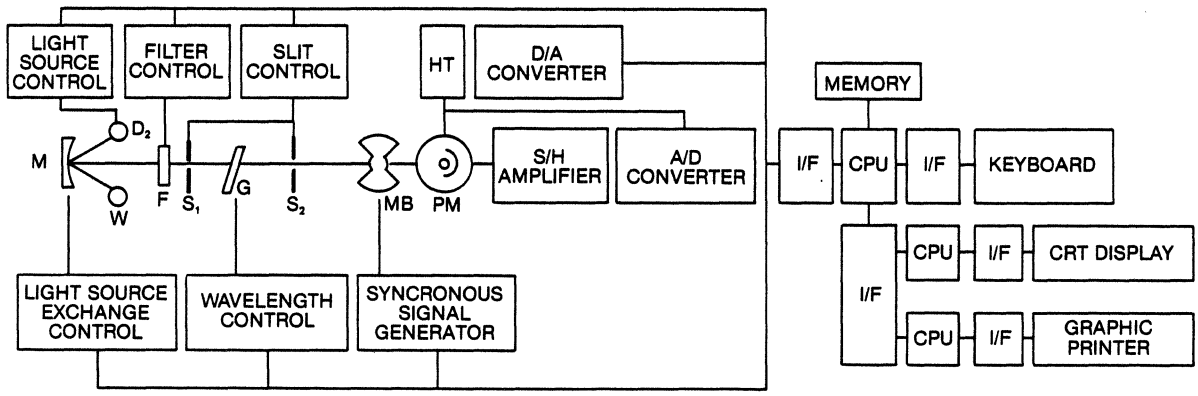


Figure 2-2. Electronic Signal Processing and Control Function

As explained in sections 2.1.3 and 2.1.5, the sample and reference beams are switched on and off by the Beam Switching Group. Since they are sequential in time, they can be multiplexed and measured by the same detector, along with PMT dark current. The chopping frequency for each signal is 25 Hz (75 measurements per second). The composite signal is demultiplexed by way of a phase sensitive synchronous demodulation technique. This circuit is contained in the block labeled, "S/H Amplifier." There are actually three amplifier channels, corresponding to the Sample signal, the Reference signal, and the dark current.

Since the raw signals all contain dark current, the dark current level must be subtracted from the Sample and Reference signals. This yields the true analog voltage for Sample and Reference. The reference signal is then used to control the H.V. power supply to the PMT during dual beam operation. This controls the gain of the PMT so that the reference signal is normalized to a constant, representing 100% transmittance. This automatically normalizes the sample signal level.

The sample signal is then converted to a digital signal and sent to the master CPU. It goes from there to the slave CPU's that control the CRT display and graphic printer.

The master CPU handles, not only the optical data, but also each of the control functions, receiving commands from the keyboard. It controls the monochromator wavelength by way of a servomechanism which controls the grating angle. Slaved to the Wavelength Control are the Light Source Exchange Control and the Filter Control. The Synchronous Signal Generator is also a control function which keeps the Sector Mirror rotation and signal demultiplexing at synchronous speed and phase angle. The slit Control is a servomechanism which sets the slit width on both slits, controlled by the keyboard. The Light Source Control provides for turning each of the light sources <u>off</u> (as distinguished from aligning a mirror to one or the other).

3.0 SPECIFICATIONS

3.1 SOURCES OF BROADBAND SPECTRAL RADIANT ENERGY

Two lamps, each selected automatically as wavelength program is executed:

Tungsten Lamp:	Operated at 9 V/2.9 A (26 W).
	Average service life: About 250 hours. Used for the infrared, visible, and near-ultraviolet portions of the spectrum.
Deuterium Lamp:	Operated at 0.3 A anode current.
	Average service life: About 250 hours. Used for shorter wavelength ultraviolet, where tungsten is very weak.

3.2 POWER SUPPLY FOR LIGHT SOURCE

Power for the D_2 lamp and W lamp is supplied by a regulated power supply.

3.3 MONOCHROMATOR

Wavelength range:	185 to 910 nm
Wavelength accuracy:	±0.3 nm
Repeatability of wavelength setting:	±0.1 nm
Wavelength display:	Digital display in four digits (least significant digit is 0.1 nm)
Diffraction grating:	1200 lines/mm, 29 mm square aperture
Spectral band width:	Continuously variable from 0.1 to 5 nm

Stray light:	Under the following conditions: Spectral band width: 2 nm, Wavelength: 220 nm, an aqueous solution of 10 grams/liter NaI in a cell with 10 mm optical path length in sample cell holder, stray light is less than 0.05% in the 9410 and 9420, and less than 0.001% in the 9430.
Wavelength scanning:	Stepping motor
Wavelength scanning speed:	4, 10, 20, 40, 100, 200 and 400 nm/min.
Repetitive scanning:	Number of scans: 1 to 100
	Repetition time 0 to 999 min and 59 sec.
Wavelength scale for plotter (λ Scale):	0.5, 1, 2, 5, 10, 20 and 40 nm/cm.
Monochromator type:	Czerny-Turner configuration
Filter	Spectral filter(s) for various wavelength regions
Radiometric Measurement Scheme:	Dual beam normalization
Sample chamber configuration:	Distance between light beams: 100 mm.
	Optical path length: 100 mm
	115 mm wide x 240 mm long x 105 mm high in overall dimensions
	Easily mounted and demounted for cleaning and maintenance.

3.4 DISPLAYED PHOTOMETRIC VALUE

1. Display of photometric value:	4 digits with minus sign, with optional fifth digit by keyboard control 0 ∿ 100.0 %T 0.000 ∿ 3.000 ABS

2.	Photometric repeatability:	±0.001 ABS (0 ∿ 0.5 ABS) ±0.002 ABS (0 ∿ 1 ABS) ±0.1 %T
3.	Photometric accuracy:	±0.002 ABS (0 ∿ 0.5 ABS) ±0.004 ABS (0.5 ∿ 1 ABS) ±0.3 %T (Calibrated with NBS 930-D standard filter)
4.	Baseline stability:	0.0004 ABS/hr Wavelength: 250 nm time constant: 4 sec Spectral band width: 2 nm After 3 hour warm-up (Power ON) with temperature variation within 5°C.
5.	Baseline Flatness:	±0.001 ABS (excluding noise) Wavelength range: 195 to 850 nm Time constant: 2 sec Spectral band width: 2 nm With baseline corrected and temperature variation within 5°C.
6.	Auto zero ("Auto Set"):	Normalizes data to present value as 100 %T or 0.000 ABS.

3.5 THE ELECTRONIC SIGNAL

Detector:	Extended red, multialkali photomultiplier tube: Hamamatsu Model R446 in 9410; Hamamatsu Model R928 in 9420/30.
Time constant:	0.4, 1, 2, 4, 8 sec.
Recording mode (hard copy) (9420 and 9430 only):	Sequential recording, overlay recording, and recorder off. Recording by solid line or three kinds of broken lines. Digital data recording Parameter recording

Recorder (9420 and 9430 only):	Heat-sensitive graphic printer, pin feed type.
Recorder numerical range:	-500 to +500 %T, -5 to +5 ABS
Chart paper:	Heat-sensitive chart paper, ruled (P/N 6493425)
	Unruled (P/N 6493426)
Chart speed:	2, 5, 10, 20, 100 and 200 mm/min.
Power consumption:	100 V, 50/60 Hz, 300 W

3.6 QUANTITATIVE ANALYSIS MODE (9420 AND 9430 ONLY)

Measurement of absorbance by one, two, or three wavelength method.

Calibration curve by piecewise linear approximation (interpolation from a series of standards).

Calibration curve by linear regression (least squares linear approximation).

Manual entry of slope and intercept of a linear calibration curve.

3.7 DATA PROCESSING FUNCTIONS (9420 AND 9430 ONLY)

Mathematical Differentiation of Spectral Data.

Smoothing operation, a digital "low-pass filter" for noise reduction.

Arithmetic operations between sets of spectral data.

Arithmetic modification of spectra (such as normalization).

Chemical Quantitative Analysis.

4.0 PERFORMANCE VERIFICATION

The specifications and performance specified in Section 3 can be verified by the following procedures.

4.1 MONOCHROMATOR

4.1.1 Energy (Light Power) Over Wavelength Range

There is much information available from the measurement of spectral energy. This information can be used to assess the "health" of the instrument, and even pin-point a defective part of the system. For instance, if the energy is low at all wavelengths (relative to normal, by a constant ratio), then optical misalignment is the most probable cause. If the energy is abnormally weak only in the very short wavelength region (below 250 nm), the probable cause is contamination of the optics (mirrors, windows, PMT, or light source envelope). Degradation only above, or only below, the lamp switching wavelength (usually 340 nm) indicates a light source fault, either the source (lamp) itself, its alignment, or the alignment of the source switching mirror, M_1. The PMT itself could be faulty in one of several ways. The most usual would be a degradation of its gain mechanism, which would affect the whole spectrum equally. Certain unusual failure mechanisms would affect the very short or very long wavelengths. Thus, the symptoms can sometimes be ambiguous.

Since the energy varies greatly over the spectrum in a normal instrument, careful comparison is required to interpret the energy spectrum in an instrument whose performance is in question. Before going into detail on this interpretation, consider a simpler test which is usually sufficient to verify a good instrument.

If a baseline spectrum is taken with the default parameters, except for narrow spectral Band Width (slit width), the instrument will be quite sensitive to an energy loss at the normally weak parts of the spectrum (below 220 nm, above 850 nm, and to a lesser extent, in the region between 320 nm and 350 nm). Whereas, in normal operation, the baseline will typically remain flat at 100% ± 0.2% due to the dual beam compensation of the energy variations, whenever the energy falls below the minimum that the instrument is able to compensate, the baseline will no longer remain flat, but will fall sharply. In addition to the obvious fall-off in the baseline, there is another warning signal that occurs when the energy is still sufficient to allow the instrument to compensate and maintain a flat baseline. It is a change in the decimal point of the digital data display on the CRT of the 9420 and 9430 and the LED display of the 9410, which also appears in the

printed data. In the 9420 and 9430, the warning is an extra decimal point which appears immediately to the right of the data (as 72.8.%T or 0.264. ABS). In the 9410, the warning is a decimal point after every digit in the data display.

When the spectral Band Width is set at 0.1 nm, it is normal for a new 9420 instrument to demonstrate a flat baseline from about 850 nm down to about 215 nm. Somewhat below 215 nm and above 850 nm, the baseline will fall sharply. There should be no notch in the baseline in the region near 340 nm. When the spectral Band Width is increased to about 0.3 nm, the baseline should be flat from 900 nm to 195 nm, but the decimal point warning may be ON at the extremities. The 9430 instrument will demonstrate somewhat less energy. Also, an older instrument will show some degradation in energy.

The relative energy of the system can be assessed by the use of the spectral Band Width. Since the energy increases, proportional to the square of the slit width (spectral Band Width), doubling the slit width quadruples the energy; ten times the slit width gives 100 times the energy, and increasing the slit width by 40% doubles the energy. For example, if 0.6 nm Band Width is needed for a flat baseline from 900 nm to 195 nm, instead of 0.3 nm, then there is apparently only one quarter the energy available from a new 9420. This amount of degradation is acceptable, but if the energy degrades to less than one-tenths of the normal energy (requiring greater than 1 nm Band Width for a flat baseline from 900 nm to 195 nm), then some maintenance will be required, according to a diagnosis as outlined in the first paragraph of this subsection.

4.1.2 Wavelength Accuracy

Wavelength accuracy is verified by measurement of absorption lines of holmium glass or emission lines of the D_2 lamp.

(a) When holmium glass is used, the measurement is made under the following conditions:

Sample:	Holmium glass
METHOD:	1
%T ABS:	%T
λ SCALE:	1 nm/cm
SCAN SPEED:	10 nm/min
TIME CONST:	0.4 sec
BAND WIDTH:	0.5 nm

The performance is satisfactory if the wavelength errors of the holmium glass absorption lines of the following wavelengths are within ±0.3 nm.

279.4 nm

360.9 nm

460.0 nm

The wavelength spectra, as measured, are shown in Figures 4-1 to 4-3.

(b) When the D_2 lamp emission lines are used, the measurement is made under the following conditions.

METHOD:	1
%T/ABS:	%T
λ SCALE:	1 nm/cm
AUTO SET:	OFF
SCAN SPEED:	4 nm/min
TIME CONST:	0.4 sec
BAND WIDTH:	0.5 nm
λ SET:	661.1 ∿ 651.1 nm.

NOTE: Method 51 must be used to select the D_2 lamp alone.

Specifications: The performance is satisfactory if the wavelength error of the emission line spectrum of 656.1 nm is within ±0.3 nm as shown in Figure 4-4.

If the wavelength error is not within ±0.3 nm at these wavelengths, calibrate the wavelength again as described in Section 5.5.

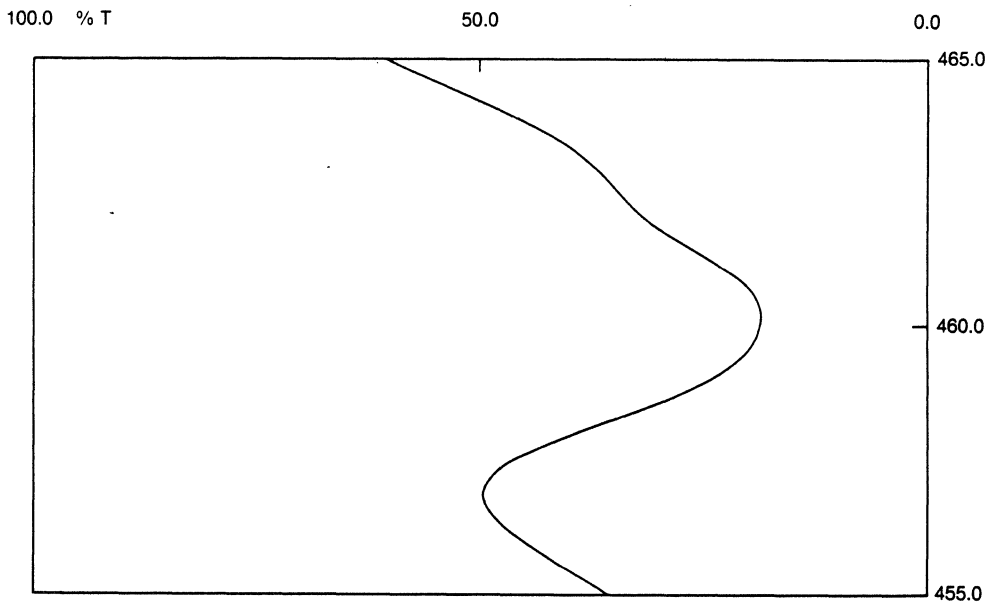


Figure 4-1. Absorption Line of Holmium Glass at 460 nm

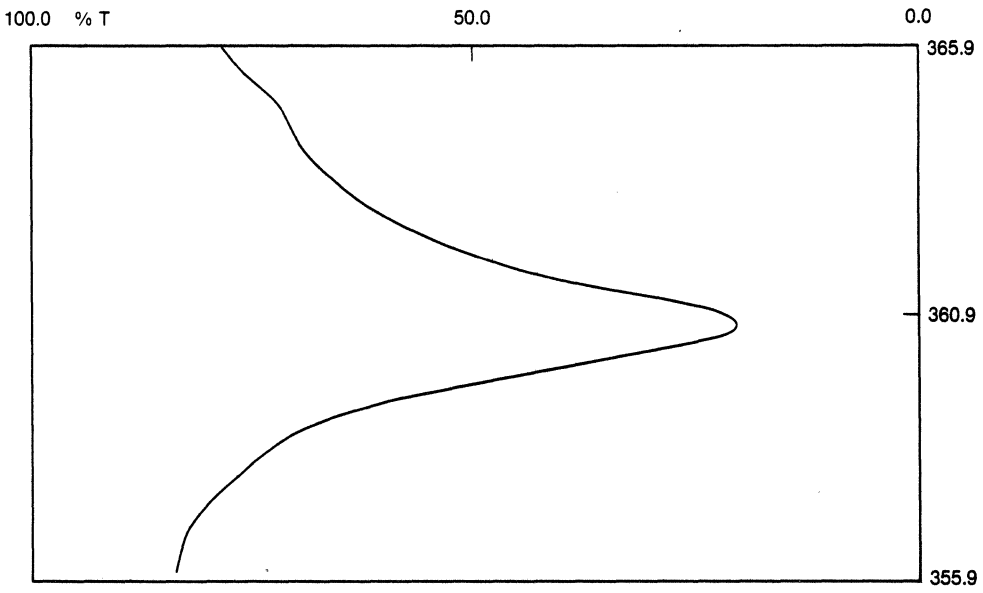


Figure 4-2. Absorption Line of Holmium Glass at 360.9 nm

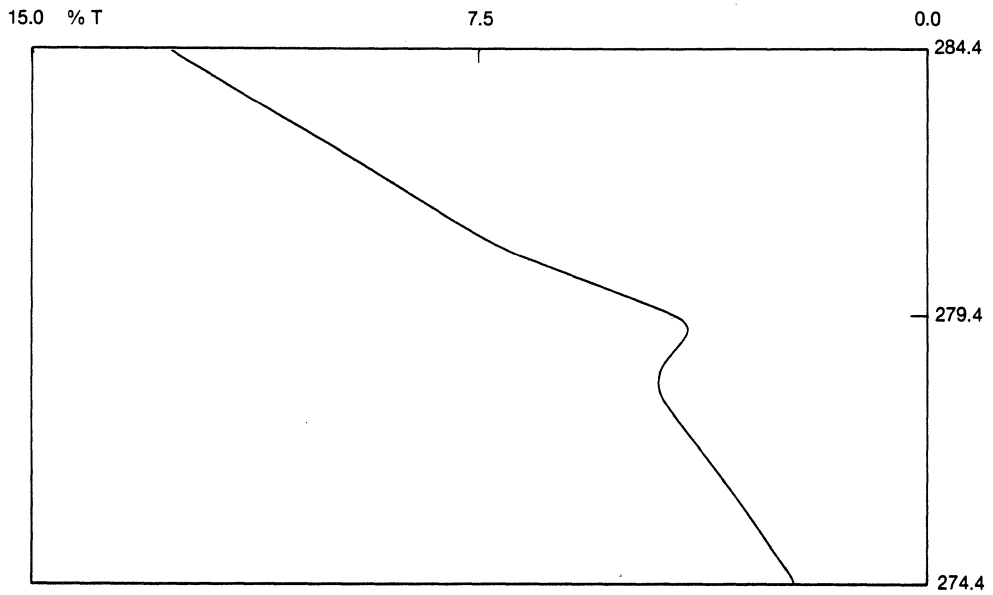


Figure 4-3. Absorption Line of Holmium Glass at 279.6 nm

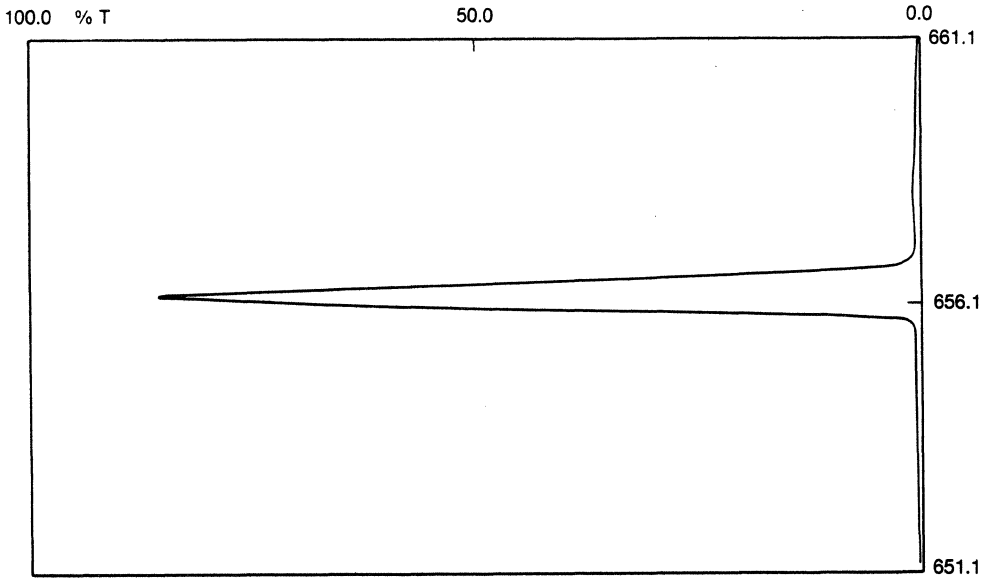


Figure 4-4. Emission Line Spectrum of D_2 Lamp at 656.1 nm

4.1.3 Wavelength Setting Repeatability

Repeat the measurement under (b) of paragraph 4.1.2 three times.

Specification: The performance is satisfactory if the deviation of the peak values of three spectra is within ±0.1 nm.

4.1.4 Wavelength Scanning Speed

Select the absorption spectrum measurement mode, set the wavelength scanning speed first at 20 and then at 100 nm/min., and measure the time required for wavelength scanning at each of the two speeds using a stop watch.

Specification: The performance is satisfactory if the measured time is within ±10% of the set value.

4.1.5 Spectral Band Width (Resolution)

Measure the spectral band width by using the 656.1 nm emission line of the D_2 lamp under the following conditions.

METHOD:	1
%T ABS:	%T
%T ABS SCALE:	0 ∿ 100%
λ SCALE:	1 nm/cm
λ SET:	658.6 ∿ 653.6 nm
SCAN SPEED:	4 nm/min
TIME CONST:	0.4 sec
BAND WIDTH:	0.1 nm in 9420 and 9430; 0.2 nm in 9410

NOTE: Use Method 54 (Method 9 in 9410) to set the PMT voltage. This must be done by trial and error until the peak at 656.1 nm reads approximately 90% transmittance.

Specification: The performance is satisfactory if the half maximum width of the spectrum is not greater than 0.11 nm (0.21 nm in 9410). If the result does not satisfy this condition, conduct the adjustment under Section 5.4, "Adjustment of Monochromator Resolution."

4.1.6 Stray Light

Prepare the following samples and filters for stray light testing.

1. Sample: NaI (10 grams/liter)

   Cell: Rectangular quartz cell, 10 mm path length

2. Sample: $NaNO_2$ (50 grams/liter)

   Cell: Rectangular quartz cell, 10 mm path length

3. Neutral filter with the transmittance of approximately 10%T (Density of 1) for testing the 9410 or 9420. For the 9430, the neutral filter to be used has a transmittance of approximately 1%T (Density of 2).

Measure the transmittance in METHOD 2 using the above samples under the following conditions.

Wavelength	Source Lamp	Sample
220 nm	D_2	NaI
341 nm	W	$NaNO_2$

Operating conditions:

%T/ABS: %T

BAND WIDTH: 2 nm

TIME CONST: 2 sec

CAUTION: Never place any attenuating filter or dense sample of any kind in the Reference cell holder until a filter or sample is first placed in the Sample cell holder, which has at least as much absorbance as the absorbing material to be placed in the Reference side. In other words, be sure that the Reference beam is not stronger than the Sample beam. This is to protect the PMT.

To measure stray light, proceed as follows:

4.1.6.1 Stray Light in 9410 and 9420 at 220 nm

Go to 220 nm. Place the neutral filter prepared in item 3 above in the designated sample cell holder, and measure its transmittance (%). The value is designated "A_1." Remove the neutral filter. Place the aqueous solution of NaI in the sample cell holder before replacing the neutral filter in the Reference cell holder. Then place the neutral filter in the Reference cell holder. Measure its transmittance (%). The value is designated "T_1." The stray light at 220 nm, designated S_1, is given by:

$$S_1 = [T_1 \times A_1/100]\%$$

At the completion of this test, remove the neutral filter from the Reference side cell holder first.

Specification: The performance is satisfactory if S_1 is not greater than 0.05%.

4.1.6.2 Stray Light in 9410 and 9420 at 341 nm

Proceed in the same manner as described in 4.1.6.1.

Go to 341 nm. Place the neutral filter prepared in item 3 above in the designated sample cell holder, and measure its transmittance (%). The value is designated "A_2." Remove the neutral filter. Place the aqueous solution of $NaNO_2$ in the sample cell holder before replacing the neutral filter in the Reference cell holder. Then place the neutral filter in the Reference cell holder. Measure its transmittance (%). The value is designated "T_2." The stray light at 341 nm, designated S_2, is given by:

$$S_2 = [T_2 \times A_2/100]\%$$

At the completion of this test, remove the neutral filter from the Reference side cell holder first.

Specification: The performance is satisfactory if S_2 is not greater than 0.05%.

4.1.6.3 Stray Light in 9430 at 220 nm

Because stray light in the 9430 is at a much lower level than in the 9410 and 9420, a more delicate test is necessary. The same solutions are used in the sample cell holder, but the neutral filter is twice as dense. This will boost the level of the stray light measurement by a factor of approximately

100, because PMT gain is governed by Reference light level. Even so, the measured quantity will be lower by a factor of approximately five, from that of the 9410 and 9420. For this reason, it is important to use the 5-digit option available in METHOD 52.

In addition, the 9430 has substantially less light available than the 9410 and 9420 have. This is due to additional light loss in the double monochromator in the 9430. Because of the availability of little energy at the two wavelengths used in the test and the use of a 1% transmittance filter, the amount of light available is marginally adequate. It is therefore necessary to verify that enough energy is available for dual beam operation.

First GO TO 220 nm. Set the spectral Band Width to 2.0 nm. Using METHOD 52, select the 5-digit display option. Return to METHOD 1. With nothing in either cell holder, verify that the data display is 100.00 ± 0.3%T. If it is not, then use the Auto Set to obtain a 100% reading. Place the neutral filter whose optical density is nominally 2.0 (1%T) in the Sample cell holder. Measure the actual transmission by first taking the data displayed in the CRT (call it A_{3d}). Then replace the filter with the shutter plate, and measure the apparent transmittance. (This should be zero, but it may not be.) This "zero-correcting" value (call it A_{30}) is to be subtracted from A_{3d}, and the result will be called A_3. (If A_{30} is negative, its numerical value is added to A_{3d}, which will increase the value of A_3.) A_3 is the true value of the transmission of the filter at 220 nm.

Remove the shutter plate, and place the NaI sample in the Sample cell holder. Then place the "A_3" filter in the Reference cell holder. Watch the CRT Data display for several seconds to see if the second decimal point warning comes on. The normal display should read thus: "00.08%T" or "00.12%T". If the second decimal point is present, the display will read "00.08.%T" or "00.12.%T."

If no second decimal point appears, it indicates that there is sufficient energy, with some margin, to continue the test in the normal manner, unless the second decimal warning is not functioning, as will be the case if the instrument has very early software. (This can be verified by reducing the spectral Band Width, which also reduces the energy, until the second decimal point appears, and then return to 2.0 nm.) If the second decimal point does appear at 2.0 nm Band Width, then increase the Band Width to 3.0 nm. If the second decimal point disappears it may still be possible to continue the test within the energy "margin." To determine this, return the Band Width to 2.0 nm; then remove the "A_3" filter from the Reference cell holder and the NaI sample from the Sample cell holder, in that order. Place the "A_3" filter in the Sample cell holder and the shutter plate in the Reference cell holder.

(This can be done without damage to the PMT because the second decimal point indicated that there is insufficient energy to damage the PMT as long as the "A_3" filter is in the Sample cell holder.) If the CRT Data display reads 110% or higher, then there is sufficient energy in the "margin" to proceed in the normal manner. If the reading is less than 110%, then an alternative procedure must be substituted to measure stray light. This reading (call it E_{3d}) will be used in the alternative method.

First, the normal procedure is presented. (Skip the following two paragraphs if the alternative method is to be used.) Remove the shutter plate from the Reference cell holder and the "A_3" filter from the Sample cell holder, in that order. Place the NaI sample in the Sample cell holder and the "A_3" filter in the Reference cell holder, in that order. The "apparent" transmission of the NaI is displayed as Data on the CRT. This figure is the reading from which the stray light is calculated. It should be a very small number (on the order of 0.10%), so it should be read carefully, taking an average of several readings. Call this figure T_{3d}. It must also be corrected with a "zero-correcting" value. It should be taken as carefully as was T_{3d}. Call it T_{30}. Subtract T_{30} from T_{3d}. (If T_{30} is negative, its numerical value will be added to T_{3d} and the result will be increased from T_{3d}.) The result is called T_3.

The Stray light level, S_3, can now be calculated by the following equation:

$$S_3 = [T_3 \times A_3/100]\%$$

The alternative method of determining stray light, which should be used only if the energy is insufficient, as determined above, is as follows. Remove the shutter plate from the Reference cell holder and the "A_3" filter from the Sample cell holder, in that order. Place the NaI sample in the Sample cell holder and the shutter plate in the Reference cell holder, in that order. The "apparent" transmission of the NaI is displayed as Data on the CRT. This figure is the reading from which the stray light is calculated. It should be a very small number (less than 0.10%T), so it should be read carefully, taking an average of several readings. Call this figure T_{3d}. It must be corrected with a "zero-correcting" value. To obtain the correction, remove the shutter plate from the Reference cell holder and the NaI sample from the Sample cell holder, in that order. Place the shutter plate in the Sample cell holder and a second shutter plate in the Reference cell holder, in that order. (A holmium glass mounting channel, turned sideward, will do.) The Data reading on the CRT is the "zero-correcting" value. It should be taken as carefully as was T_{3d}. Call it both T_{30} and E_{30}, since it will be used to correct both T_{3d} and E_{3d}. Subtract T_{30} from T_{3d} and subtract E_{30} from E_{3d}. (If T_{30} and E_{30} are negative, their numerical value will be added to T_{3d} and E_{3d}, and the results will be increased from T_{3d} and E_{3d}.)

The stray light level, S_3, can now be calculated by the following equation:

$$S_3 = [T_3 \times A_3/E_3]\%$$

4.1.6.4 Stray Light in 9430 at 341 nm

The procedure for this test is identical to that of 4.1.6.3, with the following exceptions: The 341 nm wavelength is substituted for 220 nm, and the $NaNO_2$ sample is substituted for NaI. Please follow the procedure exactly, to avoid damage to the PMT.

4.1.7 Automatic Filter and Lamp Selection

Spectral filters are used in most monochromators for reduction of stray light and "order sorting." For stray light reduction, the filters used will depend on the spectrum of the source as well as the wavelength selected. For instance, in the region from 370 nm down to 340 nm, all the 9400 series instruments normally used the W lamp (the 340 nm lower limit may actually be at any wavelength between 340 and 350 nm). In this region, the W lamp is very weak, whereas it is very powerful at longer wavelengths. A small percentage of stray light from the longer wavelength region, reaching the detector, would have a substantial effect, causing significant error. Conversely, stray light form the short wavelengths affecting the long wavelengths is an insignificant effect. Therefore a blue filter is used in the 370 nm to 340 (or 350) nm region, in the 9410 and 9420, to filter out the longer wavelengths. In the 9430, with the double monochromator, the stray light level is so low that the blue filter is unnecessary.

When a monochromator uses a diffraction grating in the <u>first</u> order, as the 9400 series does, the second and higher orders must be filtered out. For example, if the monochromator is set at 800 nm, any light of 400 nm wavelength passing the entrance slit will be subject to diffraction in the <u>second</u> order, which will pass through the exit slit as if it were 800 nm energy. This would be a much more serious problem than stray light, if an order sorting filter were not used.

An amber filter is used from just above 559 nm to the long wavelength extreme (910 nm). This filter removes all energy below about 500 nm, which means that no second order energy falls in the region between 559 nm and 910 (it would be above 1000 nm). Below 559 nm, the longest wavelength whose second order is of concern is 280 nm. However, the W lamp has no output at that wavelength, and the D_2 lamp, which does produce the short wavelengths, is optically switched out of the monochromator. No other second order wavelengths are of concern. Therefore all the 9400 series instruments use a single, amber, order sorting filter above 559 nm.

Figure 4-5 is a schedule of filter and lamp use.

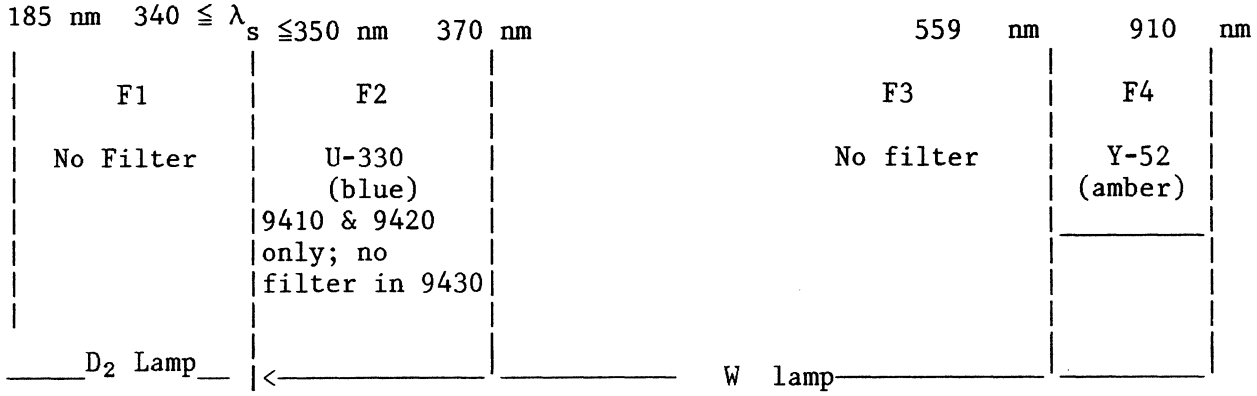


Figure 4-5. Spectral Range of Lamps and Filters

*Note: The wavelength at which the lamps are switched is the same as that of the last filter switch. It occurs between 350 and 340 nm (see Method 51). If not specified, it will occur at 340 nm. (In the 9430, there is no actual switching of filters at this point.)

To verify that the filters and lamps are switched properly, it is necessary to scan the spectrum from above 559 nm to below 340 nm (600 to 300 nm is appropriate), while observing both the filter wheel and the spherical condenser mirror (M_1) in the lamp area. In the 9410 and 9420, the lamp housing cover must be removed to observe M_1, and the monochromator seal cover must be removed to observe the filter wheel. To remove the monochromator seal cover, first null the PMT voltage by a manual voltage "0" command (Method 9 in 9410; Method 54 in 9420). Then return to Method 1, before removal of any covers. (This is necessary for execution of the command.) Remove the white, outer cover to expose the black, optical path covers. The monochromator cover is at the rear. Several cables will have to be loosened from their "tie-downs" to facilitate removal of the cover. The filter wheel is at the right rear, where the lamp energy enters the monochromator.

In the 9430, the filter wheel is in the lamp area. Therefore it is necessary only to remove the lamp housing cover to expose both lamps and filter.

4.2 ELECTRONIC SIGNAL PROCESSING SYSTEM

4.2.1 Verification of Baseline Flatness

Set the conditions as follows:

METHOD: 1

%T/ABS: ABS

λ SET: 900.0 ∿ 195.0 nm.

BAND WIDTH: 2.0 nm.

TIME CONST: 2 sec

SCAN SPEED: 100 nm/min.

%T ABS SCALE: -0.005 ∿ 0.005 ABS

Turn the power on and allow the instrument to warm up for at least one hour. Then recorrect the baseline. Record the baseline under the conditions specified above. The performance is satisfactory if the result is within ±0.001 ABS. If the baseline is not within ±0.001 ABS, investigate the following possible causes.

(a) If the flatness is unsatisfactory only at wavelengths shorter than 340 nm, the cause is likely to be the imbalance between pairs of mirrors in the photometer portion of the instrument (see Figure 2-1 and Section 5.6). Replace mirror pairs, M_{10}-M_{11}, M_{12}-M_{13}, and M_{14}-M_{15}, with new components.

(b) If the flatness is unsatisfactory at wavelengths longer than 500 nm, or if the step in the baseline corresponding to a filter or light source change is out of spec, then the likely cause is improper alignment of mirror M_{14} or M_{15}. Align them both so that they center the image on the photocathode of the PMT (see Section 5.6).

(c) A step in the baseline when the light source changes, may be caused by improper alignment of the W lamp or D_2 lamp. Refer to Section 5.2.

(d) Contamination of a filter or sample chamber window may also cause baseline drift. These components may be cleaned in a suitable solvent (mild detergent and distilled water for the filter; hexane, methanol, ethanol, or acetone for the window -- after removal).

If the baseline drift cannot be corrected optically, the electronic data processing section should be examined.

4.2.2 Equivalence of %T and ABS (Absorbance)

Turn on the instrument and allow the light sources to warm up for at least 30 minutes. Produce each %T reading listed in Table A-1 by using an appropriate sample, such as Holmium glass at an appropriate wavelength. Without changing

any condition except to change to the ABS mode, take the corresponding ABS reading. The ABS reading must be within the corresponding tolerance of the ABS equivalent.

Table 4-1. Conversion of %T to ABS

%T Reading	ABS Equivalent	Tolerance on ABS Reading
100.00	0.0000	within ±0.0010
40.00	0.3979	within ±0.0010
10.00	1.0000	within ±0.0020
1.00	2.0000	within ±0.0150
0.10	3.0000	within ±0.0500

If the ABS reading does not fall within the tolerance, the log-amp must be adjusted (see separate service manual for the electronic system).

4.2.3 Stability

Turn on the instrument and allow a one hour warm-up. Use Method 52 to yield five-digit precision. Then KEY in the following conditions:

METHOD: 3

%T/ABS: ABS

Go to λ: 250 nm

BAND WIDTH: 2 nm

%T/ABS SCALE: -0.005 ∿ 0.005 ABS

CHART SPEED: 10 mm/min.

TIME CONSTANT: 4 sec

T SET: 60 min.

Set ABS to 0.000 by using the AUTO SET, and record data for one hour. The drift tolerance is ±0.0004 ABS/hr. If the data obtained is not within the tolerance, check the following:

(a) The two light beams in the sample chamber must be precisely centered in the cell holders.

(b) The light beam incident on each mirror must fall completely within the reflective surface and should be well centered.

(c) The light beam must be centered on the photocathode of the photomultiplier (PMT).

(d) All of the optical elements must be mounted securely (not loose).

If any of these conditions are not met, then an optical alignment of all affected components must be done. If (d) is the defect, then the unstable component must be secured before the alignment of other affected components.

5.0 OPTICAL SYSTEM

The instrument has been aligned and adjusted prior to shipment from the factory to meet all specifications and normally requires no further adjustment of the optical system. However, if any of the optical elements need to be replaced, the system must be realigned. This section describes the procedure for all adjustments of the optical system.

5.1 GENERAL

5.1.1 Precautions in Optical Adjustment

5.1.1.1 Do not view the light source with the naked eye.

During any alignment procedure in which the cover of the light source is removed, eye protection must be worn, to protect the eyes from ultraviolet and high intensity visible and near infrared radiation.

5.1.1.2 Do not touch the optical surfaces of the mirrors or diffraction grating.

If an optical component is contaminated with body oils, it is unlikely to be successfully cleaned and must be replaced. This is especially true of the grating, where wiping will destroy it. Never try to wipe off dust or the like on the reflector surface with gauze or brush. (A puff of dry nitrogen may be effective in removing a particle or dust.) Great care must also be used in storing optical components, to protect them from a hostile environment.

5.1.1.3 Dust will adversely affect instrument performance, especially where stray light is concerned.

Therefore, it is advisable to select a location for the instrument as free of dust and smoke as possible.

5.1.1.4 Prior to adjustment, wash the hands to reduce oils before handling even the optical mounts.

Wearing laboratory gloves is generally recommended, although gloves may hinder some tasks.

5.1.1.5 Before installing the optical elements, remove dust and dirt completely from the optical unit frame using a delicate technique without excess air motion.

5.1.1.6 Be sure all screws are tight.

5.1.2 Optical Alignment Jigs

The Optical alignment procedures described in Section 5.3 and succeeding sections require adjustment accessories and jigs. The types, use, and methods of using these accessories are described below.

5.1.2.1 Service PROM Chips

The standard program to control the instrument is not well suited for some of the service operations, being limited to parameter ranges and operations which are practical to the instrument user. For service it is desirable to extend the ranges and eliminate automatic procedures not needed for service. To modify the control program, replace the standard PROM chip numbered "6" with the service chip. This chip povides for the service procedures which cannot otherwise be followed using standard software.

5.1.2.1.1 Initial Operating Conditions Used for Service

Set the following conditions, after initialization.

B.L. CORRECTION:	OFF
AUTO SET:	OFF
W and D_2:	Both OFF
HT:	MANUAL, 0 V

5.1.2.1.2 The range of the "GO TO λ" function is extended to cover 0 to 999 nm.

5.1.2.2 Pen Ray Lamp Assembly with Power Supply

The spectrum from a low pressure mercury lamp is used for wavelength calibration. The lamp can be directly attached to the W lamp holder.

Three wavelength settings are easily calibrated using mercury spectrum as follows:

a)	Total spectral output of Lamp	0.0 nm (zero-order, undiffracted)
(b)	Green Wavelength	Calibration at 546.1 nm (mid-range)
(c)	Blue Wavelength	Calibration at 871.7 nm (long wavelength end) from second order diffraction of actual 435.85 nm wavelength

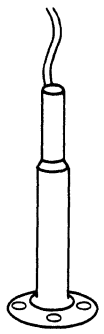


Figure 5-1 Pen Ray Lamp Assembly

5.1.2.3 Jig for Optical Axis Alignment of Sample Chamber

This jig is used for adjusting the position of the light beam in the sample chamber.

A disk with a small hole in the center is moved along the V-shaped grooves and the beams are checked for centering, relative to the hole. Decentration and angular misalignment can be detected visually in this manner.

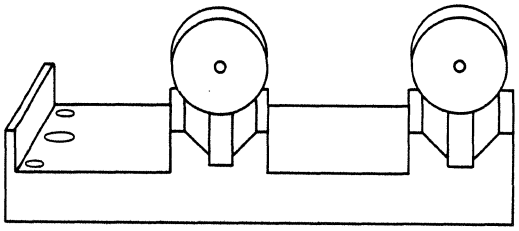


Figure 5-2. Jig for Optical Axis Alignment of Sample Chamber

5.1.2.4 Jig for Grating Lever Length Adjustment

This jig is used for adjusting the length of the wavelength drive lever. (It is seldom used because the lever length does not change under normal circumstances.)

The length is set at 110.25±0.05 nm between the diffraction grating drive axis and the roll center of the lever edge.

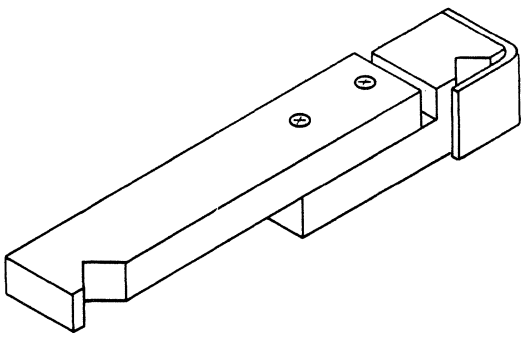


Figure 5-3. Jig for Lever Length Adjustment

5.1.2.5 Spanner Wrench for Sample Chamber Window

Used in removing and remounting the sample chamber quartz windows.

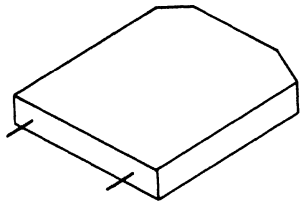


Figure 5-4. Spanner Wrench for Sample Chamber Window

5.1.3 List of Recommended Optical Spare Parts

Optical elements for replacement are supplied in subassemblies described in Table 5-1.

Table 5-1. Optical Spare Parts

Component	Description of Subassembly
Tungsten lamp	with mounting flange and Nylon electrical connector
Deuterium lamp	with Nylon electrical connector

Component	Description of Subassembly
Mirror M_1	R95 spherical mirror with mount (9410 and 9420) R80 spherical mirror with mount (9430)
M_2	Plane mirror with mount
M_3 and M_4	R500 spherical mirror with mount
M_5	Plane mirror with mount
M_6	R spherical mirror with mount
M_7, M_8, M_9, M_{10}, M_{11}	Plane mirror with holder
M_{12} and M_{13}	Spherical mirror with mount
$M_{14}$and M_{15}	Plane mirror with mount
Diffraction grating (main monochromator)	Plane grating with mount (for 9430, this grating is a ruled grating, not the regular holographic grating)
Diffraction grating (pre-monochromator)	Concave grating with mount (9430 only)
Light source window	With holder
Optical window of sample chamber	Set of 4 pieces with holder

5.2 REPLACEMENT AND ADJUSTMENT OF LAMPS

5.2.1 Preliminary Instructions and Precautions

5.2.1.1 Electrical Safety

Hazardous voltages are used in the lamp section. Therefore the instrument should be turned off and unplugged during the replacement part of this procedure.

5.2.1.2 Exposure to Radiation

When the deuterium (D_2) lamp is on, it emits ultraviolet radiation which is harmful to the eyes. Therefore, eye protection must be worn. The use of goggles which strongly attenuate the ultraviolet is recommended.

5.2.1.3 Lamp Handling

Do not handle the lamps directly with bare hands. A lamp contaminated with body oils should be rinsed in a solvent (hexane) and wiped dry (not air dried).

5.2.2 Lamp Access and Identification

5.2.2.1 Removal of the Lamp Housing Cover

The lamp housing, located at the right rear corner of the Optical Unit is shown in Figure 5-5 (top view from front).

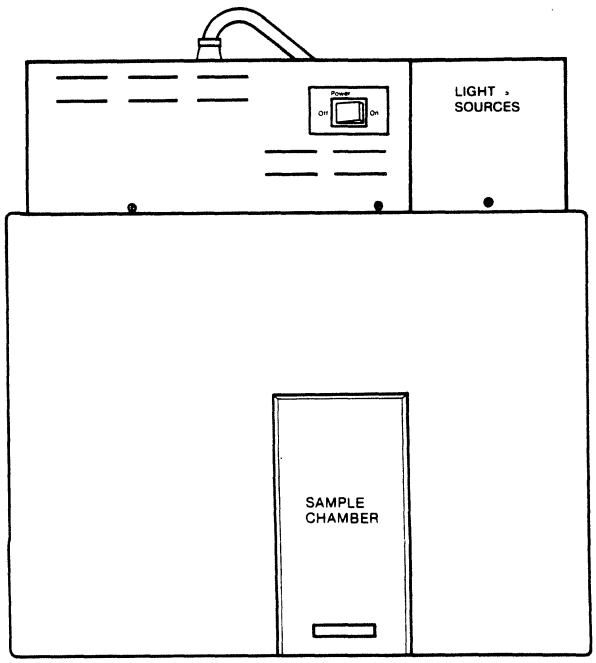


Figure 5-5. Location of Lamp Housing

To remove the cover, loosen the thumb screw and back it out several turns. Gently rock and raise the rear of it enough to clear the shoulder screws. Slide it rearward, and lift it off.

5.2.2.2 Identification of the Lamps

The location of the individual lamps within the housing is shown in Figure 5-6 (top view from rear).

The W Lamp is located close to the left side of the instrument, as indicated by "W" in Figure 5-6. It is identified by its spherical bulb, small tungsten filament, a mirror coating on a large spot on the glass behind the filament, and two electrical connections. The D_2 lamp is located to the right of the W lamp. It is identified by its cylindrical bulb clamped into a special mounting assembly, a small rectangular aperture in the metal assembly inside the bulb, and three electrical connections. The third component is the lamp selecting mirror.

5.2.3 Lamp Replacement

5.2.3.1 The W Lamp

Disconnect the electrical connector to the W lamp. Unlock the lamp by rotating it counterclockwise (CCW), and lift it out.

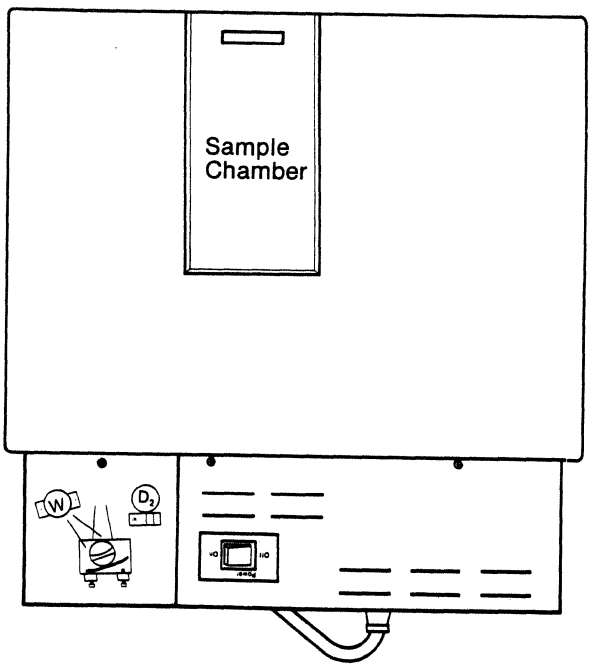


Figure 5-6. Location of Lamps

Place the new W Lamp on the holder with the mirror behind the filament, the leads down through the bottom slot of the lamp mount and out toward the mating connector. Seat it on the three locking pins and rotate it CW until it is fully locked. Connect the electrical connector.

5.2.3.2 The D_2 Lamp

Disconnect the electrical connector to the D_2 lamp. Remove the entire lamp mounting assembly by removing the one screw which holds it to the casting (see Figure 4-1) and lifting the assembly from the two locating pins.

Loosen the clamp which holds the lamp in the assembly. Slide the old lamp out and the new one in. Position it approximately the same as the old one was (with the internal aperture aimed toward mirror M_1). Leave the clamp loose enough for adjustment. Replace the assembly on the locating pins, and retighten the screw to the casting. Connect the electrical connector.

5.2.4 Optical Alignment

5.2.4.1 General

Turn the instrument on. Using the Source Control Method (7 in the 9410 and 51 in the 9420 and 30) each lamp is turned on individually (one at a time) to eliminate interference with visual inspection. Refer to Figure 5-7 for an alignment diagram of the light source optics, viewed from behind the instrument.

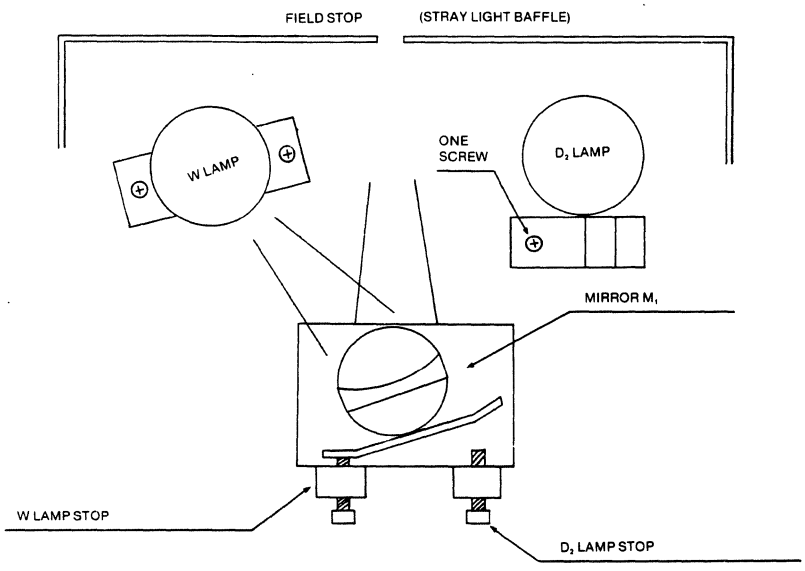


Figure 5-7. Alignment Diagram

The spherical mirror, M_1, images one or the other of the lamps onto the field stop (a rectangular opening in the wall leading to the monochromator). The "lamp stop" is a mechanical stop used to center the image horizontally at the field stop. This is done by first loosening the lower socket head screw of the clamping mechanism for the adjustment (use a 2.5 mm hex key from tool pouch provided). The upper screw is then adjusted to center the image and the lower screw is then retightened to clamp the adjustment. (The clamping mechanism affects the adjustment slightly and should be taken into account in the adjustment; the alignment should be rechecked after clamping).

5.2.4.2 Alignment of the W Lamp Optical Path

Use the Source Control Method (7 in the 9410 and 51 in the 9420 and 30) to turn on the W lamp alone. The horizontal alignment is accomplished as in 4.1.

The vertical dimension should not need adjustment.

5.2.4.3 Alignment of the D_2 Lamp Optical Path

Use Method 51 to turn on the D_2 lamp alone. Align the lamp itself as the first step in this procedure. It must be aligned in both the horizontal (angular) and vertical (axial) dimensions, based on the image position at the field stop, as in paragraph 4.1. The vertical position should be as accurate as possible. After alignment of the D_2 lamp, carefully tighten the clamp. The D_2 lamp stop (see Figure 4-1) can be used for a fine adjustment of the horizontal alignment, if necessary, as with the W lamp. Use the Source Control Method (7 or 51) to return the instrument to operation with both lamps.

5.2.4.4 Finer Adjustments

Finer adjustments are made by using the PMT detector in the single beam mode, to maximize the light level. A PMT Voltage command (Method 9 in the 9410 or 54 in the 9420 or 30) is used to set PMT voltage to a level determined experimentally to give a %T reading of about 80%. (Do not allow the voltage to be high enough to exceed a reading of 150%, since PMT damage could result.) Selection of one lamp or the other is made simply by selecting a wavelength above or below 340 nm. This alignment is made based on maximizing the %T reading. Bandwidth should be set between 2 and 5 nm. Upon completion of this alignment procedure, return the voltage level on the PMT to its usual automatic setting.

5.3 ALIGNMENT OF THE MONOCHROMATOR

The service PROM chips are necessary for this procedure (see 5.1.2.1).

(a) Turn on the W lamp and set the slit width to 1.5 mm. (Slit width of 1.5 mm corresponds to 5 nm of the spectral Band Width.)

(b) Adjust the collimator mirror (M_3) so that the beam reflected by M_3 is centered on the diffraction grating.

(c) Rotate the diffraction grating until the zero order (white light) beam from the diffraction grating falls centered on the focusing mirror (M_4). If the beam incident on M_4 is decentered vertically, correct it by means of the pitch adjusting screw on the grating mount (see Figure 5-6).

(d) Adjust M_4 so that it focuses the beam, accurately centered on the exit slit.

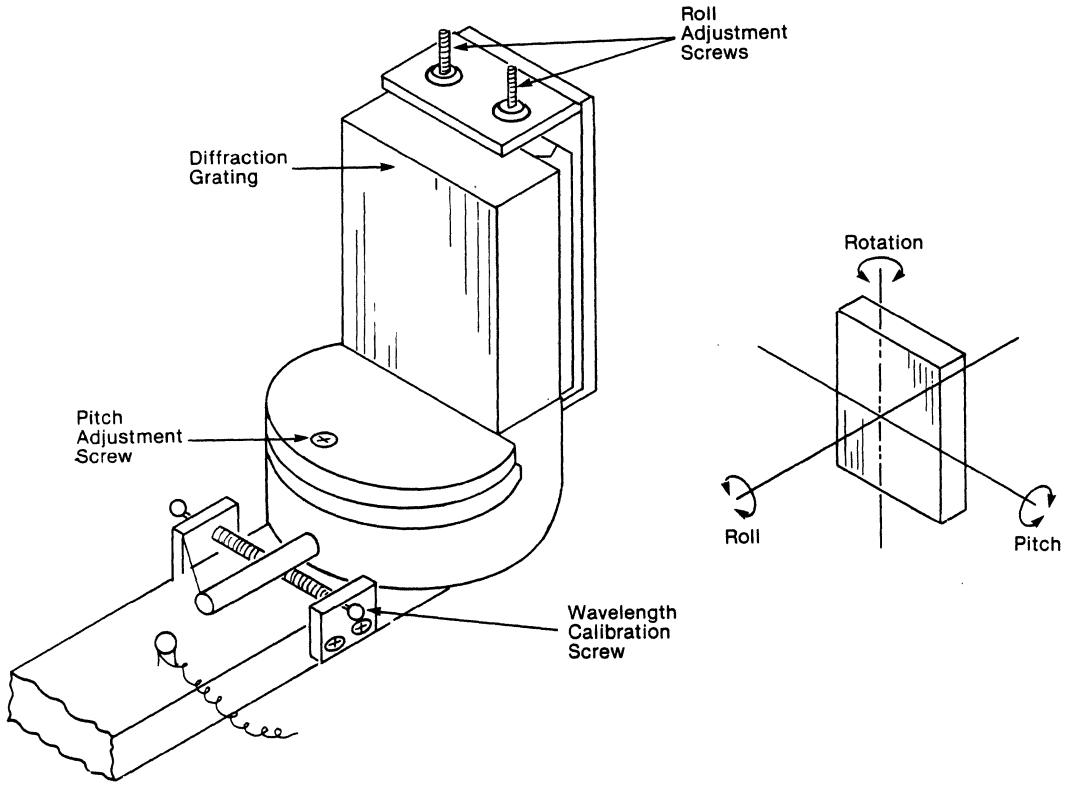


Figure 5-8. Diffraction Grating Holder

(e) Rotate the grating through the spectrum and verify that all of the visible wavelengths focused on the exit slit lie vertically centered on the slit within a tolerance of ±0.5 mm (±0.020 inch). If the spectrum is skewed, so that the error in vertical position changes as the grating is rotated, then adjust the grating groove inclination by means of the roll adjustment screws shown in Figure 5-6.

Note: Whenever the roll is corrected, step (c) should be repeated for verification. If the pitch must be recorrected, then steps (c) through (e) must be repeated until no further corrections are necessary.

5.3.1 Adjustment of the Pre-monochromator (9430 Only)

(a) Loosen the mounting screws of the M_2 mirror mount.

(b) Place a screen in front of the G_1 grating and adjust the position of the M_2 mirror mount so that the light reflected by M_2 is centered on the G_1 grating. Verify that the height of the beam reflected by M_1 is 45 mm.

(c) Tighten the M_2 mirror mount screws to clamp the adjustment.

(d) Turn off the instrument and replace PROM No. 6 with the service PROM.

(e) Turn on the instrument, and turn OFF the D_2 lamp using Method 51, so that only the W lamp is ON. Use the GO TO λ command to set the wavelength at zero.

(f) Loosen the pulley set screw on the G_1 grating shaft, to free the pulley.

(g) Rotate the G_1 grating to center the zero-order light of the G_1 grating on the entrance slit.

(h) Adjust the pitch adjusting screw on the G_1 grating holder so that the height of the image on the collimator, M_3, becomes 50 mm, using a scale in front of M_3

(i) Confirm that all of the diffracted energy at the exit slit is in good vertical alignment with the slit for each wavelength. If the alignment is not correct for some wavelength it indicates that the G_1 grating groove inclination must be adjusted. The roll adjusting screw of the G_1 grating will adjust the groove inclination.

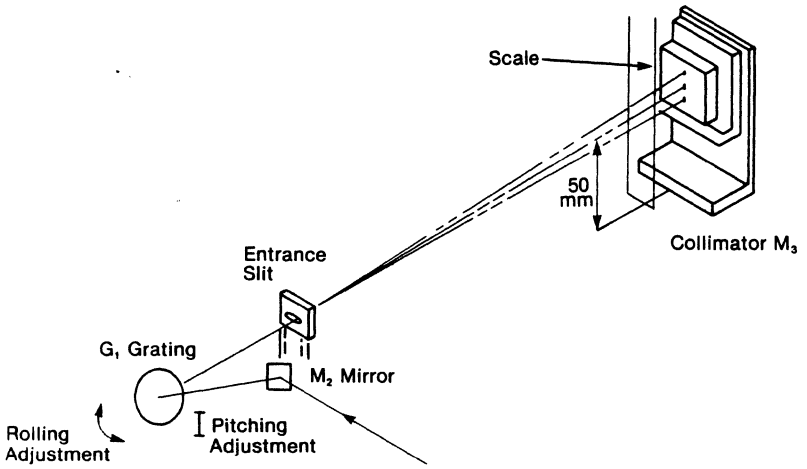


Figure 5-9. G_1 Grating Pitch Adjustment

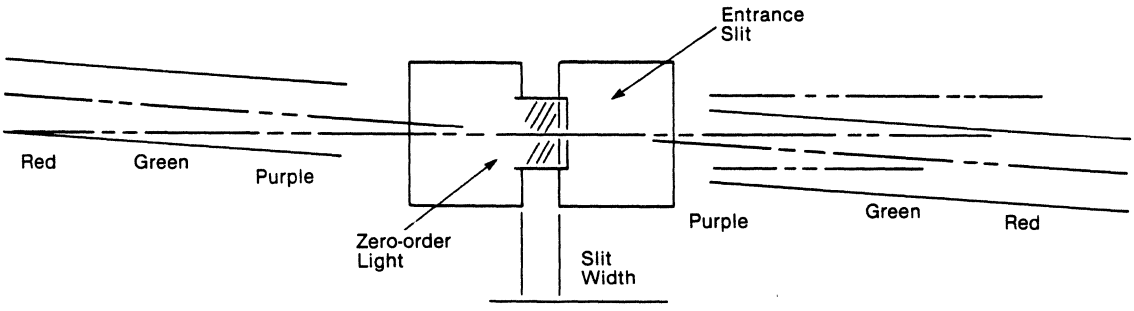


Figure 5-10. G_1 Grating Roll Error

(j) After step (i) is completed, center the zero-order light of G_1 again on the entrance slit and verify that the light is centered on the collimator M_3. If the light deviates in the vertical direction, repeat steps (g) and (h).

k) After the above adjustments are completed, rotate the G_1 grating to center the zero-order light of G_1 on the entrance slit. Clamp the G_1 grating mount by tightening the mounting screws of the G_1 grating mount.

5.3.2 Optical Coincidence of Pre- and Main Monochromators (9430 Only)

This procedure is necessary whenever any of the components are removed or aligned.

(a) Replace PROM No. 6 with service PROM. Then, turn on the instrument.

(b) Replace the W lamp with the pen-ray type Hg lamp.

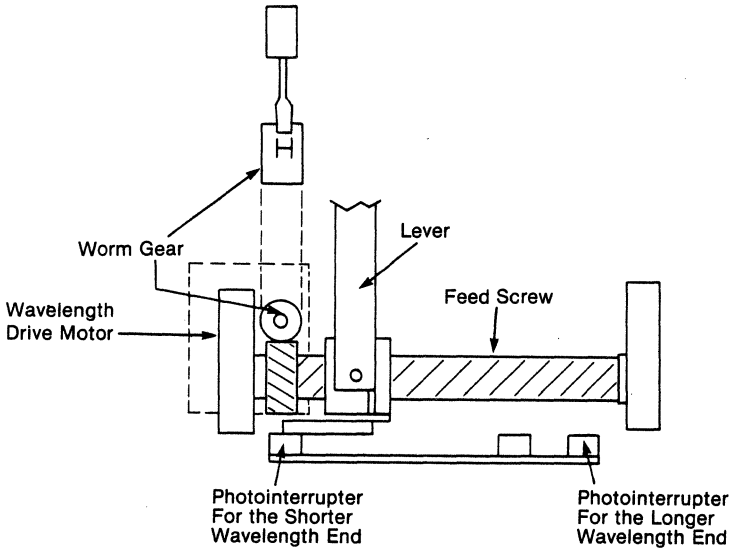


Figure 5-11. Main Monochromator Wavelength Drive Assembly

(c) Set the wavelength at 546.1 nm by means of the GO TO λ function.

(d) Set the Band Width at 2 nm.

(e) Confirm that the 546.1 nm line from the G_1 grating enters the center of the entrance slit. Confirm that the 546.1 nm line also passes through the exit slit. If the 546.1 nm line is not well centered in the exit slit, then the main monochromator must be calibrated (see section 5.5). If a small rotation of the worm gear drive of the G_2 sine bar will allow the 546.1 nm line to pass the exit slit, without noticeably disturbing the centering of the line on the entrance slit, then no further adjustment to the pre-monochromator is necessary.

(f) If the alignment of the 546.1 nm line at the exit slit does not coincide with alignment at the entrance slit, then loosen the mounting screws on the G_1 grating mount and rotate G_1 to bring the 546.1 nm line through the entrance slit.

(g) Rotate the worm gear of main monochromator wavelength drive motor so that the green light of 546.1 nm is observed passing through the exit slit (see Figure 5-9).

(h) Tighten the set screw on the G_1 grating pulley.

(i) Tighten the mounting screws on the G_1 grating mount.

5.4 ADJUSTMENT FOR MAXIMUM MONOCHROMATOR RESOLUTION

This procedure should be performed only after completion of Section 5.3. The service PROM chip is necessary for this procedure, and the service area must be darkened. Maximization of the monochromator spectral resolution is basically a precise focus adjustment, based on what is called the "Foucault Knife Edge Test."

(a) Turn on the W lamp and set the slit to 0.7 nm spectral Band Width. Do not fully close the slit, because it may be seriously damaged if it is fully closed.

(b) Place a piece of white paper somewhat behind the exit slit. Rotate the grating to put the zero order (white light) onto the exit slit (and on the paper screen). Then back it off to a setting of about 5 nm. This will place to zero-order light to one side of the slit and allow it to be moved manually across the slit.

(c) Using a thin rod to manipulate the Sine Bar end of the grating lever, slowly move the image across the exit slit. Examine the spot of light on the paper screen. If the image on the slit and the spot on the paper screen move in the opposite directions, move the focusing mirror, M_4, closer to the slit. Conversely, if the image on the slit and the spot on the screen move in the same direction, move M_4 away from the slit. If this adjustment is perfectly done, the image on the screen should appear and disappear with no apparent motion. Actually, there are two kinds of motion with this test. The technician must learn to distinguish between the motion due to the beam moving across the slit and the non-simultaneous filling of the aperture due to a focus error. Furthermore, for an adequate focus, great care must be taken both in assessing the results of the test and in moving the M_4 mount to adjust the focus. The position of the mirror mount must be set to well within ±0.010 inch for an adequate focus. There is also a tendency for the mount to move as the clamping screws are tightened. Therefore, a final test should be made after the mount is tightened, to be sure that the tightening of the screws has not introduced a focus error. The technician may also be able to devise a means of moving the grating mount without inserting a rod into the light beam, since it is a source of confusion in interpretation of the Knife Edge test. A long rod (even a straightened coat hanger) can be used between the roll adjusting screws at the top of the grating.

(d) Upon completion of this adjustment, check the positions of M_3 and M_4 by looking down from directly above the mirrors. If there is a large difference in the distance from the slit to these collimators, move the collimator M_3 by half the difference so as to correct the difference and then repeat the adjustment described above in 5.4 (c). If the error is large the procedures contained in (c) and (d) may have to be repeated until simultaneous collimation at the grating and optimum focus are achieved.

(e) Finally, repeat the tests of sections 4.1.2 and 4.1.5 to verify those specifications.

5.5 WAVELENGTH CALIBRATION

The service PROM chip is necessary for this procedure and the service area must also be darkened.

(a) Replace the W lamp with the pen ray mercury lamp and set the spectral Band Width to 0.7 nm.

(b) Set the diffraction grating drive lever and its drive screw in such a way that the center lines of the two intersect at approximately right angles when the zero order beam (full spectrum reflected light) on the exit slit.

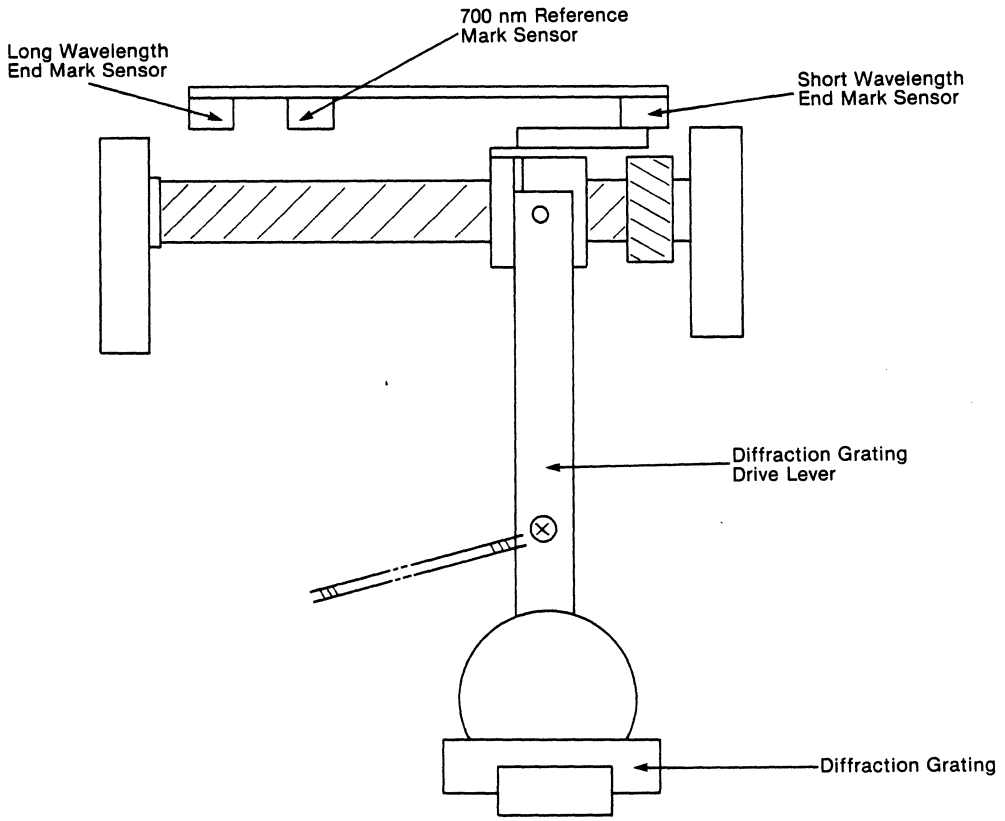


Figure 5-12. Diffraction Grating Lever and Drive Screw

(c) Place a small plane mirror behind the exit slit and position it so that the light passing through the exit slit can be easily viewed.

(d) Set the wavelength at 100.0 nm by using the key "GO TO λ."

(e) Turn the wavelength drive motor shaft manually, using a #2 Phillips screwdriver inserted axially into the worm (gear on the motor shaft, so that the zero order beam is centered exactly on the exit slit, as evidenced by a maximum of light through the exit slit. The monochromator is now set at zero nm, although the digital data display on the CRT is at 100 nm. All subsequent wavelength readings will have this offset in their value.

(f) Use the "GO TO λ" to set the wavelength near 646 nm. Be sure the filter wheel is in an open position. If not, move it by hand. By keying in various λ values, find the one that maximizes the light through the exit slit. The actual wavelength of this line is 546.07 nm, which should read 646.1 nm with the 100 nm offset. If it reads within ±0.1 nm of this value, then go to step (h). If the indicated wavelength deviates from this value by more than 0.1 nm, adjust it in accordance with step (g).

(g) If the CRT display of the wavelength reads a longer wavelength than 646.1 nm, the zero order line must be moved so as to coincide with a longer wavelength reading by 17.4 times the error. For example, if the error is +0.5 nm (reading 646.6 nm), the zero order must be moved +8.7 nm. To do this, set the wavelength to 108.7 nm by "GO TO λ" and adjust the grating angle by means of the wavelength calibration screws shown in Figure 5-6, so that the zero order beam is centered on the exit slit.

If the wavelength on the CRT display reads a shorter wavelength than 646.1 nm, the zero order line must be moved so as to coincide with a shorter wavelength reading by 17.4 times the error. For example, if the error is -0.5 nm (reading 645.6 nm), set the wavelength to 91.3 nm by "GO TO λ" and adjust the grating angle by means of the wavelength calibration screws shown in Figure 5-6 so that the zero order beam is centered on the exit slit.

(h) Set the wavelength to 100.0 nm by "GO TO λ."

(i) Turn the wavelength drive motor shaft with the #2 Phillips screwdriver to center the zero order beam on the exit slit, as indicted by a maximum in the light through the exit slit, as in step (e).

(j) Repeat steps (f) through (i) until the CRT displays a wavelength value of 646.1±0.1 nm when the 546.07 nm line is centered on the exit slit.

(k) Using the "GO TO λ" command near 972 nm, search for the maximum light through the exit slit, as in step (f). This is the second order of the grating for the 435.84 nm line, which should appear at 971.7±0.3 nm (435.84 x 2 + 100 nm) on the CRT display.

(l) If the wavelength value found in step (k) is in error by more than ±0.3 nm, the diffraction grating drive lever length may be incorrect. The correct lever length is 110.25±0.05 nm in the center to centered distance, between the grating rotation shaft and the 8 mm rod which contact the follower on the lead screw. Use the special jig for lever length adjustment (Figure 5-3), to either verify the correct length or adjust the rod position so that the grating rotation shaft and rod fit in the V-grooves correctly. Loosen the screw in the rod, and carefully retighten it, while holding both the shaft and rod seated in the V-grooves. Then repeat steps (f) through (k).

Note: The tolerance on the lever length, 0.05 mm, is only 0.0025 inches. Therefore, it must be set very carefully.

5.6 ADJUSTMENT OF PHOTOMETER SECTION

5.6.1 General Considerations

The optical elements used in an ultraviolet/visible spectrophotometer must generally be treated with more care than those used in the infrared region, especially against contamination of the surface with organic matter, because organic matter strongly absorbs the short wavelengths. Furthermore, the two light paths (Sample and Reference) in the photometer section must be balanced over the entire wavelength region. To facilitate this, the mirrors used in the dual beam part of the photometer section (from the Sector Mirror to the PMT) are paired. Each element in one path corresponds to one in the other path. The two mirrors in each pair are coated in the same production lot. If it becomes necessary to replace a mirror due to damage or contamination, then it is recommended that the complete pair of mirrors be replaced. These pairs are identified as follows:

M_8 (sector mirror) and M_9

M_{10} and M_{11}

M_{12} and M_{13}

M_{14} and M_{15}

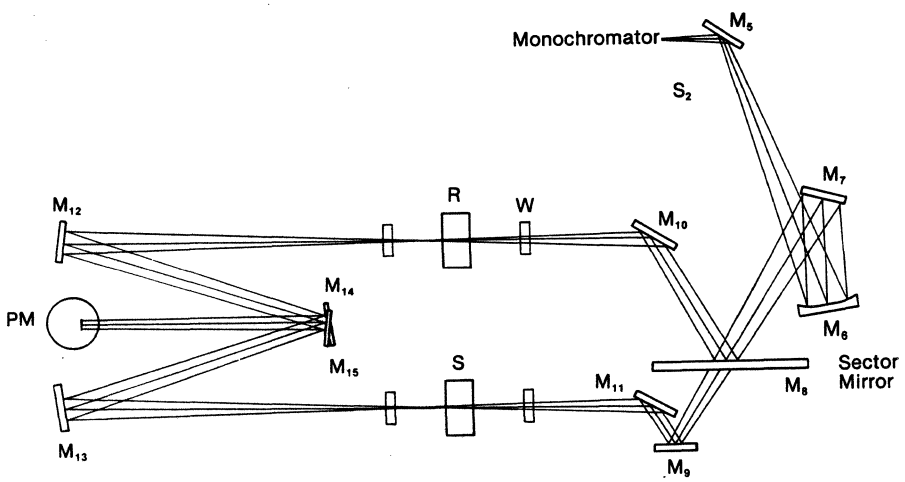


Figure 5-13. Optical arrangement of Photometer Section

Similarly, the quartz windows used at the entrance and exit of the sample chamber should be uniform in transmittance and kept free of scratches, dust, and contamination with oils or organics.

The adjustment of the photometer section requires the service PROM chips. The optical alignment jig for the sample chamber (Figure 5-2) should be used in the adjustment of components covered in 5.6.3.

5.6.2 Replacement of Sector Mirror (M_8)

If it is necessary to replace the sector mirror, follow these steps:

(a) Loosen the four set-screws on the flexible joint connecting the sector mirror and motor shafts. Slide the joint to the motor side.

(b) Remove the sector mirror assembly by loosening the two screws on the sector mirror assembly base and lifting it out carefully past the chopper sensor to avoid damage to the chopper wheels (See Figure 5-10).

(c) Remove the sector mirror from the assembly by loosening the four mounting screws on the hub.

(d) Remove any excess adhesive or foreign matter from the contact surfaces of the sector shaft and mirror. Mount the new sector mirror with the four hub screws.

(e) Reinstall the sector mirror assembly and the flexible joint.

(f) Verify that there is no interference between the chopper sensor and wheel as it is rotated by hand. If necessary, adjust the position of the sensor to prevent contact.

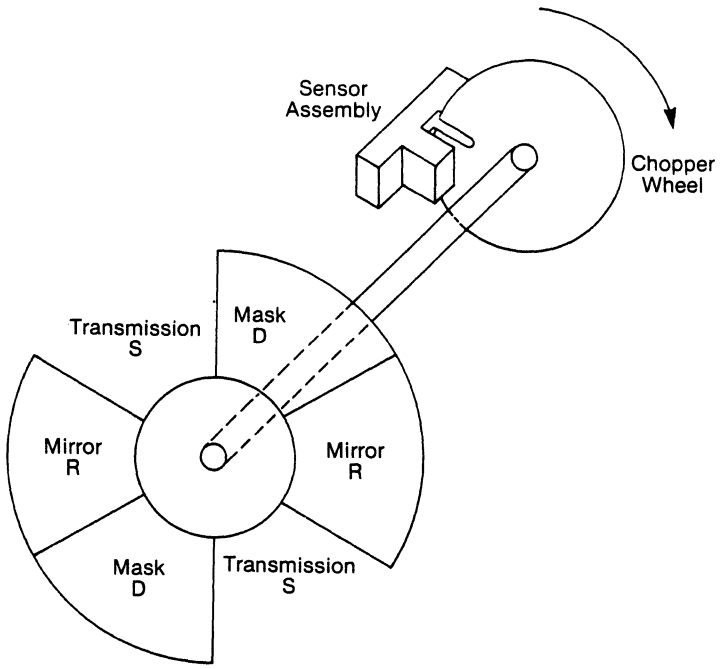


Figure 5-14. Relative Position of Sector Mirror and Sensor

5.6.3 Alignment of the Optical Components from Exit Slit to Sample Chamber

(a) Install the sector mirror and connect it with the motor shaft (see 5.6.2).

(b) Mount mirrors M_5, M_6, M_7, M_8, M_9, M_{10} and M_{11} as shown in Figure 5-9. (Mirrors M_8 and M_9, and M_{10} and M_{11} must be from the same production lot.)

(c) Rotate the diffraction grating to a position where the zero order beam is centered on the exit slit. Turn on the W lamp and set the slit at about 1.5 mm. Adjust M_5 so that the light beam from the slit of the monochromator is centered on M_6, without being shaded by M_7.

(d) Adjust M_6 so that the beam is centered on M_7.

(e) Install the optical alignment jig in the sample chamber.

(f) With the sector mirror rotated by hand to a reflecting position, adjust M_7 so that the beam is centered on M_{10}. Adjust M_{10} so that the beam passes through the center of the "REFERENCE" side of the alignment jig.

(g) With the sector mirror rotated by hand to a transmitting position, adjust M_9 and M_{11} so that the light beam passes through the center of the "SAMPLE" side of the alignment jig.

Note: Baseline flatness over the entire wavelength region is dependent upon careful adjustment of the dual path.

5.6.4 Optical Alignment of Detector Section

(a) Install mirrors M_{12}, M_{13}, M_{14} and M_{15}. (Mirrors M_{12} and M_{13}, and M_{14} and M_{15} should be from the same production lot.) The two beams should be centered on the spherical mirrors M_{12} and M_{13}.

(b) As shown in Figure 5-9, adjust M_{12} and M_{13} so that the beam reflected by M_{12} is centered on the M_{14} (the upper), and the beam reflected by M_{13} is centered on M_{15} (the lower).

(c) Adjust the M_{14} and M_{15} so that both beams from M_{14} and M_{15} completely coincide on the photocathode area of the photomultiplier (PMT).

Note: The beam alignment onto the PMT is critical. It greatly affects the stability covered in paragraph 4.2.3.

5.7 MOUNTING OF THE SLIT MARKER

The service PROM chip is necessary for this adjustment. Whenever the slit assembly or slit driving motor is replaced, readjust the slit marker position, as follows:

(a) Set the slit at about 1 mm (3.3 nm).

(b) Adjust the slit marker position as shown in Figure 5.11 and clamp it.

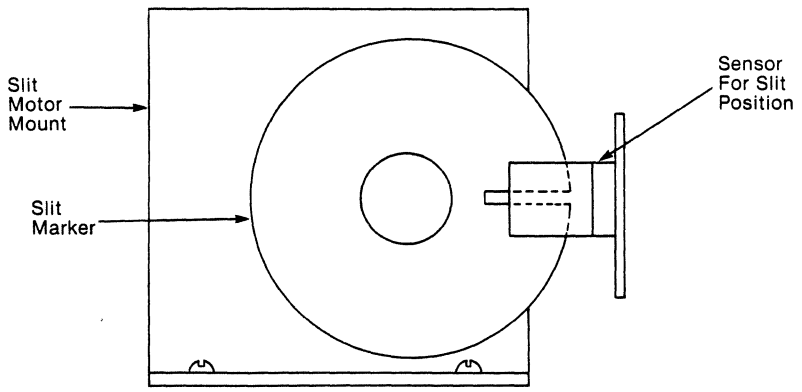


Figure 5-15. Mounting of Slit Marker

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