MODEL J-810 SPECTROPOLARIMETER

Hardware/Function Manual

JASCO Corporation

Safety Cautions

Correct operation and scheduled maintenance are essential for safe use of the instrument. Read the safety cautions in this manual and fully understand them before operation.

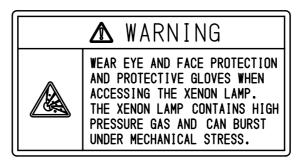
"WARNING", "CAUTION" and "Note" are used throughout this manual to call operator's attention to safety.

- (1) Meanings of safety notations
- **WARNING** : Failure to comply with this involves the possibility of death or serious personal injury. Serious personal injury means loss of sight, injury, burn (high temperature, low temperature), electric shock, fracture, intoxication, etc. which can cause sequel or require hospitalization or longterm treatment at hospital.
- **CAUTION** : Failure to comply with this involves the possibility of minor personal injury or physical damage. Minor personal injury means injury that does not require hospitalization or long-term treatment, and physical damage means damage to the instrument itself or damage to objects other than the instrument, such as buildings, properties, etc. (extended damage).
- **Note**: This contains care to be taken during operation and information that will be helpful in operation.

(2) Location of Warning Labels

The instrument is labeled with the following warnings. Use care not to damage or tear the labels. Should the labels be stained or torn, contact your local JASCO distributor with its part number.

1) WARNINGS! Handling the Xenon Lamp (①of Fig. 1)



Part number : 0822-0127A

- . The xenon lamp is made of quartz glass and is filled with high-pressure gas (5 to 10 atm. pressure, about four times when the lamp is lit).
- Twisting, bending or impact can burst the lamp, causing danger with glass fragments. Never open the lid of the light source unit if the lamp is lit or hot.
- . When handling the xenon lamp, wear protective clothing such as a thick, long-sleeved shirt, a mask, thick gloves, etc.
- . Before replacing the xenon lamp, turn the lamp off and allow it to cool for about 30 minutes.
- When replacing the xenon lamp, be careful not to give an impact to the lamp. Never twist the lamp by holding it at both ends.
- . Do not mistake the mounting direction (polarity) of the lamp. The cathode will be damaged if the lamp is mounted incorrectly, rendering the lamp inoperable.
- . Do not touch the glass portion of the xenon lamp with bare skin.
- . If the glass portion of the xenon lamp is contaminated, wipe it with clean gauze moistened with alcohol.
- . After removal and before disposal, place the xenon lamp in its case, and store the case in a safe place. If the case is not available, carefully wrap the lamp in foamed plastic or other protective wrapping, and store it in a same place.
- . When disposing of the xenon lamp, carefully wrap it in a cloth, smash it with a hammer, and dispose of it as hazardous material.
- WARNING! Fuse Rating (2) in Fig. 1)



Part number : 0822-0102A

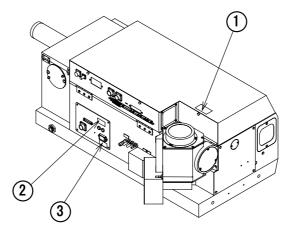
Use only fuses of the designated rating to protect both the operator and the equipment from fire and other hazards. When replacing the fuse, turn OFF the " Power" switch and unplug the power cable from the outlet to avoid electric shock. WARNING! Grounding (③in Fig. 1)



Part number : 0822-0109A

If the instrument is operated without being grounded properly, the operator may be subjected to electric shock. Correctly ground the instrument using the grounding terminal on the switchboard. Do not use gas or water pipes for grounding, because these pipes are often made of non-conductive material.

150W Light Source, Small Sample Chamber



450W Light Source, Large Sample Chamber

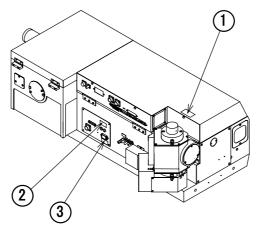


Fig. 1

Introduction

This manual is intended to serve as a guide for using the Model J-810 spectropolarimeter, both for experienced and first-time operators.

Prior to operating the instrument, the operator should carefully read and thoroughly understand the contents of these manuals. In addition, the "Measurement", "Analysis" and "CD-ORD Measurement Method" instruction manuals should be understood in full. These three manuals should be kept at your hand at all time during operation of the instrument. After operation, keep these manuals in your file. Should the manuals be lost, contact your local JASCO distributor for an additional copy.

Installation Requirements

Install the instrument in a location where the following conditions are satisfied. The instrument should be installed in a room that is maintained at a constant temperature and humidity, because the spectropolarimeter is sensitive to atmosphere fluctuations.

- . Room temperature 20 $\pm 5^{\circ}$ C
- . Humidity lower than 70%
- . Not exposed to direct sunlight
- . Not in the proximity of harmful or corrosive gas
- . Not exposed to a high-intensity light source
- . Not in the direct path of air currents emanating from air conditioners or other equipment
- . Relatively free of vibration
- . Not in the proximity of a high-intensity magnetic or electromagnetic field
- . For the 450W light source, within the proximity of a water supply (flow rate : 2 λ

/min, pressure : 0.5 \sim 2.0 kg/cm²). Cooling water piping : 10 m in length, inside diameter : 12 mm

)
Note : Do not use water at more than 2.0 kg / cm^2	pressure

. Within the proximity of equipment that supplies nitrogen gas. (flow rate : more than 3 λ /min). Nitrogen gas tube : 3 m in length, inside diameter : 9.5 mm.

. Within the proximity of nitrogen gas ventilator.

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1. Overview and Specifications

1.1 Overview

1.1.1 Principles of operation

When linearly-polarized light passes through an optically-active substance, its two circularly-polarized components (right and left circularly-polarized beams of light) travel at different speeds, and are absorbed in differing degrees by the substance. Thus, the light passing through the substance is elliptically polarized, and the substance is said to have "Circular Dichroism (CD)". The magnitude of circular dichroism is usually expressed in terms of molecular elliptically [θ], which is determined according to the following formula :

$$\left[\theta\right] = \frac{4500}{\pi} (\varepsilon_L - \varepsilon_R) \log_e 10 \tag{1}$$

where, ϵ_L and ϵ_R are the molecular extinction coefficients for the right and left circularlypolarized beams of light. The difference ($\Delta\epsilon$) between ϵ_L and ϵ_R is determined using the following formula :

$$\Delta \varepsilon = \varepsilon_L - \varepsilon_R = \frac{1}{LC} \log_{10} \left(\frac{I_R}{I_L} \right)$$
(2)

$$\left[\theta\right] = \frac{4500}{\pi LC} \log_e 10 \log_{10} \left(\frac{I_R}{I_L}\right)$$
(3)

In equation (2) and (3), L represents the thickness (cm) of the absorbing layer, and C represents the molar concentration, I_R and I_L represent the intensities of the right and left circularly-polarized beams of light, respectively, after passing through the substance. Theoretically, the molecular ellipticity can be derived using equation (3). However, in practice, determining [θ] with a high degree of accuracy is very difficult using equation (3), because the value of $I_R.I_L$ is nearly 1. To avoid this difficulty, we substitute the following quantities.

$$I_A = \frac{1}{2} (I_R + I_L)$$

$$S = I_R - I_L$$
(4)
(5)

Since $S/2I_A$ is less than 1, equation (3) can be re-expressed as follows, by substituting the expressions from equations (4) and (5) :

$$\begin{bmatrix} \theta \end{bmatrix} = \frac{4500}{\pi LC} \log_e 10 \log_{10} \left(\frac{1 + \frac{S}{2I_A}}{1 - \frac{S}{2I_A}} \right)$$

$$= \frac{4500}{\pi LC} \log_e 10 \left(\frac{S}{I_A} \log_{10} e \right)$$
(6)

Thus, the ratio between I_A and S can be approximated with an accuracy sufficient for practical application.

Given that E_A and E_S represent the output voltages of the photomultiplier tubes corresponding to light intensities I_A and S respectively, $S/I_A = E_S/E_A$. By substituting this expression, equation (6) can be expressed as follows.

$$\left[\theta\right] = \frac{4500}{\pi LC} \log_e 10 \left(\frac{E_s}{E_A} \log_{10} e\right) \tag{7}$$

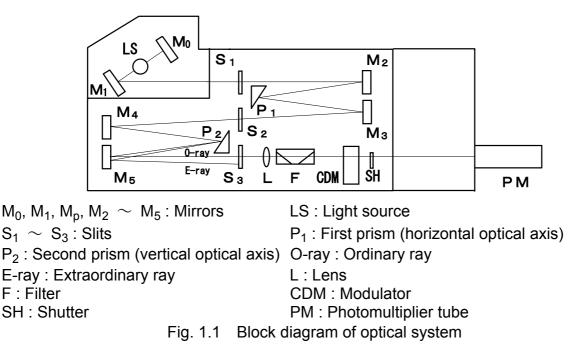
Here, if E_S can be amplified independently of E_A , equation (7) can be expressed as follows.

$$\left[\theta\right] = \left(\frac{4500}{\pi LC} \log_e 10\right) \left(\frac{E_s G}{E_A}\right) \left(\frac{\log_{10} e}{G}\right)$$
(8)

where G represents the amplification factor of E_S . Since the value E_SG can be brought very close to the value of E_A by choosing an appropriate value for G, [θ] can be determined with a high degree of accuracy.

1.1.2 Optical system

Fig. 1.1 shows the optical system of the Model J-810 spectropolarimeter.



A xenon lamp is used as the light source. The light emitted from the xenon lamp is converged by the M_1 mirror into the S_1 entrance slit. The optical system between the S_1 entrance slit and the S_2 intermediate slit is referred to as the first monochromator, and the optical system between the S_2 intermediate slit and the S_3 exit slit is referred to as the second monochromator. Such an optical system, comprised of two monochromators, is known as a double monochromator. The capability of a double monochromator reducing stray light makes it indispensable for CD measurement.

The instrument uses crystal prisms (P_1 and P_2) that have different axial orientations, so that the light that passes through the monochromator is not only monochromated, but also linearly polarized, and oscillates in the horizontal direction.

This linearly-polarized light is modulated by the modulator into right and left circularlypolarized beams of light. The modulator subjects quartz to mechanical stress in order to produce circular polarization in the crystal, based on the principle of the Piezo effect. When a sample with circular dichroism is placed in the sample chamber, the intensity, I, of the transmitted light changes as shown in Fig. 1.2 :

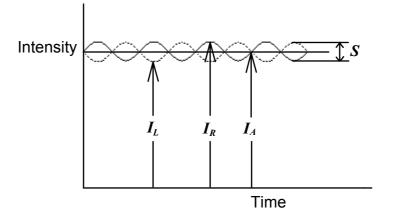


Fig. 1.2 Intensity of transmission light

The relationship between the minimum and maximum intensities and the right and left circularly-polarized light depends on whether the value E_R is smaller or larger than the value E_L . In Fig. 1.2, the solid line represents the case when E_R is greater than E_L , and the dotted line represents the case when E_R is smaller than E_L . For the definitions of I_A

and S, refer to Section 1.1.1 "Principles of operation".

When light with intensity fluctuations such as that shown in Figure 1.2 is incident upon the photomultiplier tube, the output signal consists of DC components equivalent to I_A , and AC components equivalent to S.

1.1.3 Electrical system

The output signal from the detector (PM photomultiplier tube) consists of an AC component electrically modulated by the modulator and a DC component that represents the average intensity of the transmission light. The CD value can be derived from the ratio between the DC component and the AC component. This instrument varies the PM voltage in order to maintain a constant DC component, and utilizes the AC component as the CD signal. Therefore, once the AC signal is calibrated using a standard sample, the correct CD value can be obtained.

Fig 1.3 shows a block diagram of the electrical system. Since the AC and DC components can be discussed independently, they are described separately in reference to Figure 1.3.

The DC component is separated between the preamplifier and the CD amplifier, and is compared with the reference voltage in order to control the voltage of the PM power supply. This voltage is also applied to the PM detector, changing the PM sensitivity. The AC component is converted to a digital signal after being amplified by the preamplifier and the CD amplifier.

The main unit and the personal computer communicate through the RS-232C interface. All parameters of the main unit are designated from the personal computer. The CD signal and PM voltage are communicated to the personal computer through the RS-232C interface after being converted to digital signal and stored in the buffer memory.

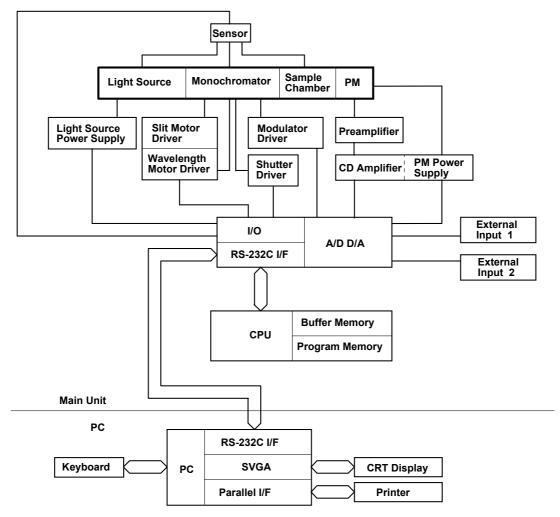


Fig. 1.3 Block diagram of electrical system

1.2 Specifications

Light source :	150W air-cooled xenon lamp, or 450W water-cooled xenon lamp	
Detector :	Head-on photomultiplier tube	
Modulator :	Piezoelastic modulator	
Measurement wavelength range : 163 to 900 nm (standard detector)		
	163 to 1100 nm (optional detector)	
Wavelength accuracy :	\pm 0.2 nm at 163 to 180 nm	
	±0.1 nm at 180 to 250 nm	
	±0.3 nm at 250 to 500 nm	

	±0.8 nm at 500 to 800 nm
<u>+</u>	±2.0 nm at 800 to 1100 nm
Wavelength repeatability : ±	±0.05 nm at 163 to 250 nm
5	±0.1 nm at 250 to 500 nm
	±0.2 nm at 500 to 1100 nm
1	0.01 to 15 nm
	1 to 3000 μm
•	0.5 msec to 32 sec Continuous scan
	Step scan (Fixed response and auto response system)
Scanning speed : t	o 10000 nm/min (continuous scan)
	0.025 to 10 nm (continuous scan)
	0.1 to 100 nm (step scan) 0.5 msec to 60 min (time change)
	$\pm 10, 200, 2000 \text{ mdeg}$
	0.0005 mdeg (at ± 10 mdeg full scale)
	0.01 mdeg (at ±200 mdeg full scale)
	0.1 mdeg (at ±2000 mdeg full scale)
Stray light :	Not more than 0.0003% (200 nm)
RMS noise :	185 nm : 0.050 mdeg (150W light source)
5	0.045 mdeg (450W light source) 200 nm : 0.040 mdeg (150W light source)
-	0.035 mdeg (450W light source)
5	500 nm : 0.035 mdeg (150W light source)
/	0.035 mdeg (450 light source)
	(spectral bandwidth 1nm, response 16 sec) 0.03 mdeg/hr
-	spectral bandwidth 1 nm, response 32 sec, wavelength 290
	nm)
	Single beam measurement
	Photometric range : 0 to 5 Abs Photometric accuracy : ±0.01 Abs
	(0 to 1 Abs, checked using NIST SRM 930D filter)
	Two channels (input range : -1 to 1 V DC)
	Opens and closes in front of sample
•	Small sample chamber dimensions :
	140mm wide \times 300mm deep \times 130mm high _arge sample chamber dimensions :
	305mm wide \times 420mm deep \times 270mm high
	Sample stage can be dismounted and remounted and
	accepts various accessories.
	Equipped with constant temperature water inlet/outlet ports
	t: Atmosphere in the light source unit, monochromator unit, and sample chamber is displaced using dry nitrogen gas.
	$20 \pm 5^{\circ}$ C
•	Less than 70%
2	Main unit (small sample chamber) :
=	

	Main unit (large sample chamber) :
	1270mm wide $ imes$ 570mm deep $ imes$ 410mm high
Weight :	Main unit (small sample chamber): 87 kg
	Main unit (large sample chamber):106 kg
Power requirements :	100, 115, 200, 220, 230, 240 V, 50/60 Hz
	270 W (150 W light source)
	670 W (450 W light source)

2. Unpacking and Installation

Note: Hold the monochromater bench when you move the main unit.

2.1 Unpacking

After unpacking the instrument, check the parts received against the list of components (Table 2.1). If any part is missing or damaged, contact your local JASCO distributor.

Component	Q'ty	Remarks
J-810 main unit	1	
Modulator element	1	
Detector unit	1	
Cable	1 set	
Nitrogen gas inlet tube	1	3 m
Nitrogen gas outlet tube	1	
Nitrogen gas tube band	2	
Cooling water tube	1	10 m for the 450W light source
Cooling water tube band	3	For the 450W light source
Sample chamber window	1	
Inner sample chamber	1	For large sample chamber
Cell holder	1	
Standard samples	1 set	Ammonium d-10-camphor sulfonate, etc.
Tools	1 set	
Floppy disk	1 set	For setup, data, etc.
Instruction manual	1 set	For hardware, software, etc.
Tube for accessories	1 set	For accessories

Table 2.1 List of Components for J-810 Spectropolarimeter

2.2 Installation Requirements

Install the instrument in a location where the following conditions are satisfied. The instrument should be installed in a room that is maintained at a constant temperature and humidity, because the spectropolarimeter is sensitive to atmosphere fluctuations.

- . Room temperature $20 \pm 5^{\circ}C$
- . Humidity lower than 70%
- . Not exposed to direct sunlight
- . Not in the proximity of harmful or corrosive gas
- . Not exposed to a high-intensity light source
- . Not in the direct path of air currents emanating from air conditioners, or other equipment
- . Relatively free of vibration
- . Not in the proximity of a high-intensity magnetic or electromagnetic field
- . For the 450W light source, within the proximity of a water supply (flow rate : 2 λ /min,

pressure : 0.5 \sim 2.0 kg/cm²). Cooling water tube : 10 m in length, inside diameter : 12 mm

Note: Do not use water at more than 2.0 kg / cm^2 pressure.

- . Within the proximity of equipment that supplies nitrogen gas. (flow rate : more than 3 λ /min).
- . Nitrogen gas tube : 3 m in length, inside diameter : 9.5 mm.

2.3 Reassembly

Note: Reassembly of the instrument is performed by your local JASCO distributor.

2.3.1 Removing the cushion from the main unit

CAUTION: When removing the cushion, be careful not to give an impact to the cam.

The cam and lever on the bottom of the instrument are separated by a cushion and secured with a rubber band to protect the cam from damage and the wavelength from shifting during transit.

Remove the cushion and rubber band after installing the main unit.

(1) Remove the side cover from the main unit.

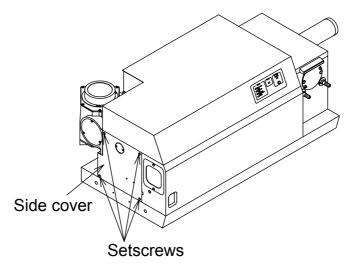


Fig. 2.1 Removing the side cover

(2) Remove the cushion and rubber band.

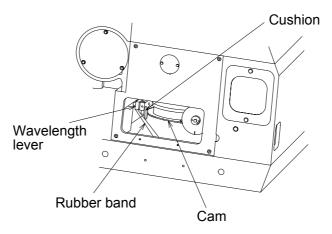


Fig. 2.2 Removing the cushion and rubber band

2.3.2 Installing the modulation element

The modulation element has been removed from the main unit before shipment to prevent damage in transit. Install the modulator element in the main unit after installing the main unit.

(1) Remove the electrical system cover from the main unit.

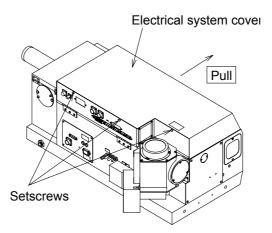


Fig. 2.3 Removing the electrical system cover

(2) Open the monochromator lid.

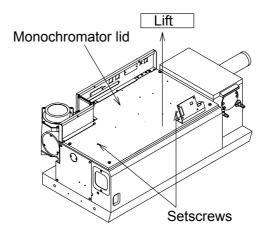


Fig. 2.4 Opening the monochromator lid

(3) Mount the modulator element in the holder and mount it in the main unit.Solder the leads to the terminals.

CAUTION : Be careful not to damage the modulator element or break the leads.CAUTION : See to it that the leads do not come into contact with the holder, cover or with each other.

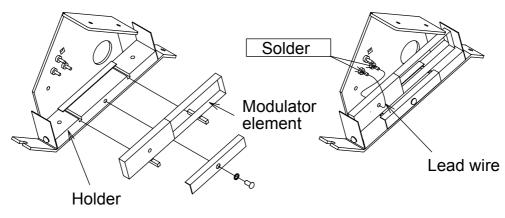


Fig. 2.5 Mounting the modulator element

2.3.3 Installation the detector unit

- **CAUTION** : Handle the detector unit with great care. Do not give an impact to the detector unit.
- **CAUTION** : Do not loosen any screws, except for the lock screw.

CAUTION : Do not expose the detector window to intense light.

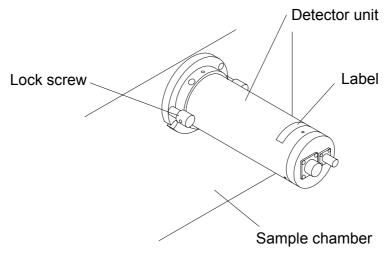


Fig. 2.6 Installation of detector unit

Install the detector unit on the main unit according to the following procedure.

- (1) Remove the cover from the detector mounting hole located in the right side panel of the main unit
- (2) Remove the cover from the detector unit
- (3) Gently mount the detector unit on the main unit, and secure it using the lock screw.

CAUTION : Install the detector so that the label faces upward.

2.3.4 Connecting the cables and tubes

Connect the cables and tubes according to the following procedure :

- (1) Connecting the cables
 - 1) Using a digital voltmeter, confirm that the supplied line voltage corresponds to the voltage shown on the rating plate.

CAUTION : The line voltage must be confirmed. An outlet can provide an incorrect voltage due to faulty wiring.

- 2) Confirm that the "Power" switch on the power supply unit is turned OFF.
- 3) Plug the cables according to Fig. 2.7.

CAUTION: Ground the grounding terminal of the power cable.

CAUTION: For details regarding the wiring and connections of the personal computer and printer, refer to their respective instruction manuals.

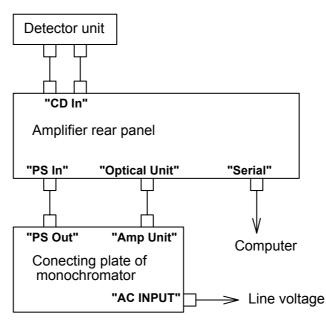


Fig. 2.7 Connection of cables

(2) Connecting the cooling water tubes (450W light source)

CAUTION : Bind the tube at the water faucet and the water inlet/outlet ports of the light source unit, using the supplied tube bands.

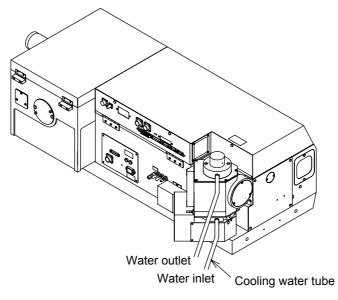


Fig. 2.8

- 1) Connect the "Water Inlet" of the light source cooling water flow sensor to the faucet using the tube.
- 2) Connect the "Water Outlet" of the light source cooling water flow sensor to the "Water Inlet" of the light source unit.
- 3) Connect the "Water Outlet" of the light source unit to the water drain port.
- 4) Feed cooling water to confirm that the system is free from leakage.
- (3) Connecting the nitrogen gas tube

Connect the nitrogen gas cylinder (flow meter) to the nitrogen gas inlet.

Note : Bind the tube with the tube band, if necessary.

(4) Connecting the nitrogen gas or leak water outlet port.

The outlet tube for nitrogen gas or leaked circulation water is provided at the bottom of the sample chamber. Connect the tube to the water outlet port, if necessary.

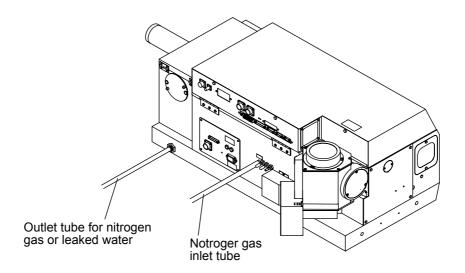


Fig. 2.9

3. Names of Functions of Components

3.1 Overall View

150W light source. Small sample chamber

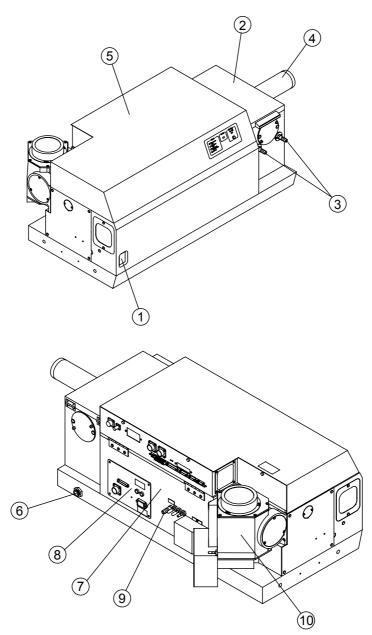


Fig. 3.1 Overall view (150 kW light source. Small sample chamber)

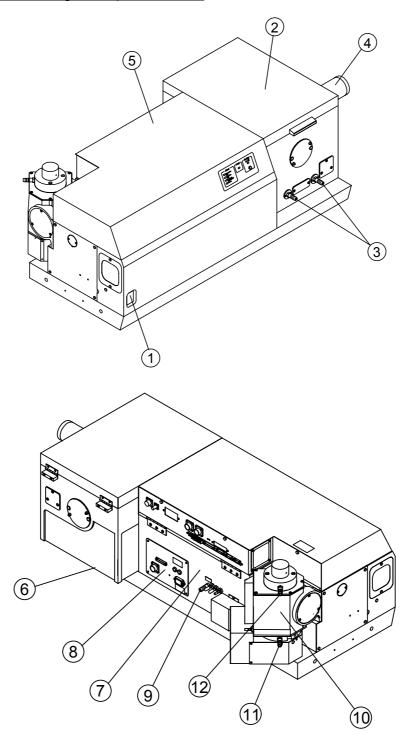


Fig. 3.2 Overall view (450kW light source. Large sample chamber)

Component	Function
① "Power" switch	Power switch of the main unit.
②Sample chamber	Set sample.
③Constant temperature water ports	Inlet and outlet ports of constant temperature water.
④Detector unit	Houses the photomultiplier tube and preamplifier.
5 Amplifier unit	Houses the amplifier and other elements.
6 Sample chamber leak water outlet	Outlet for the water leaking from sample chamber.
⑦Monochromator unit	Houses the monochromator and modulator.
8 Connecting plate	Connected to the electrical system.
Initrogen gas inlet	Admits nitrogen gas to displace the air in the monochromator.
10 Light source unit	Houses the light source.
①Light source cooling water inlet	Cooling water inlet to the light source.
Light source cooling water outlet	Cooling water outlet from the light source.

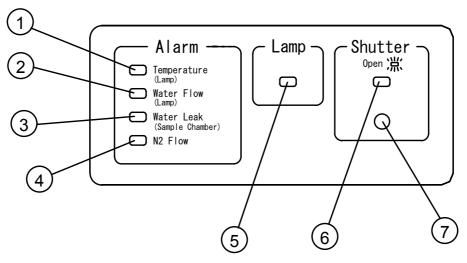


Fig. 3.3 Front panel of amplifier unit

Component	Function
① "Temperature (Lamp)" LED	Lights if the light source temperature becomes abnormally high. The light source is automatically turned off if an alarm is displayed.
② "Water Flow (Lamp)" LED	Lights if the cooling water to the 450W light source is insufficient.
③ "Water Leak (Sample Chamber)" LED	Lights if water leaks in the sample chamber. The use of the optional constant temperature water stop valve stops water leak automatically.
④ "N2 Flow" LED	Lights if the nitrogen gas flow rate is insufficient. This alarm is effective when the optional PC-controlled flowmeter or the flowmeter with sensor is used.
5 "Lamp" LED	Lights when the light source is turned ON.
6 "Shutter" LED	Lights when the shutter is open.
⑦ "Shutter" button	Opens/closes the light shield shutter.

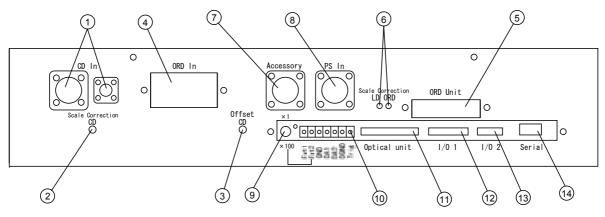


Fig. 3.4 Rear panel of amplifier unit

Component	Function
① "CD In" connector	Connect to the connector on the detector unit.
② "Scale Correction ID"	Trimmer for adjusting the CD scale using a standard sample.
③ "Offset CD" trimmer	Trimmer for adjusting offset of CD.
④ "ORD In" connector port	Connect the connector for optional ORD unit.
 ORD Unit" connector port 	Connect the connector for optional ORD unit.
 6 "Scale Correction LD/ORD" adjustment mounting port 	Port for mounting the optional LD/ORD scale adjustment.
⑦ "Accessory" connector	Connector for optional accessories.
⑧ "Ps In" connector	Connect to the "Ps Out" connector on the rear panel of the monochromator unit.
③ "X1/X100" selector switch	Changes over the "Ext2" terminal input voltage gain. Set it at X100 to amplify the gain by a factor of 100.
III/O terminals	
"Ext1" terminal	Input terminal for analog signal (-1 to 1 VDC)
"Ext2" terminal	Input terminal for analog signal (-1 to 1 VDC)
"GND" terminal	Grounding terminal (for analog signal)
"DA1" terminal	Output terminal for analog signal (0 to 1 VDC)
"DA5" terminal	Output terminal for analog signal (0 to 5 VDC)
"DGND" terminal	Grounding terminal (for digital signal).
"Trig" terminal	Trigger signal input terminal.
1 "Optical Unit" connector	Connect to the "Amp Unit" connector on the rear panel of the monochromator unit.
1/O 1" connector	Connect to an optional accessory.
13 "I/O 2" connector	Connect to an optional accessory.
(1) "Serial" connector	Connect to the connector for communication of the personal computer.

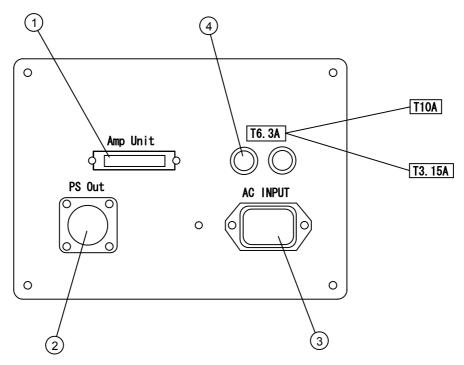
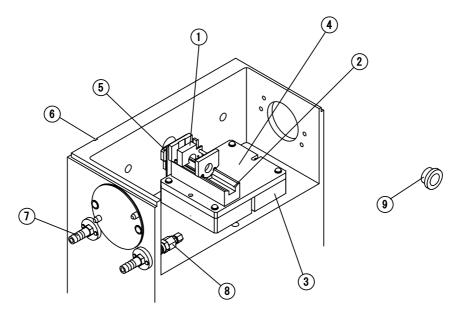


Fig. 3.5 Rear panel of monochromator unit

Component	Function
① "Amp Unit" connector	Connect to the "Optical In" connector on the rear panel of the amplifier unit.
② "PS Out" connector	Connect to the "PS In" connector on the rear panel of the amplifier unit.
③Receptacle	Power inlet receptacle
④Fuse	
"T3.15A" label	For 200 VAC line voltage.
"T6.3A" label	For 100 VAC (150W light source) or 200 VAC (450W light source) line voltage.
"T10A" label	For 100 VAC line voltage.

3.3 Sample Chamber

Small sample chamber



Large sample chamber

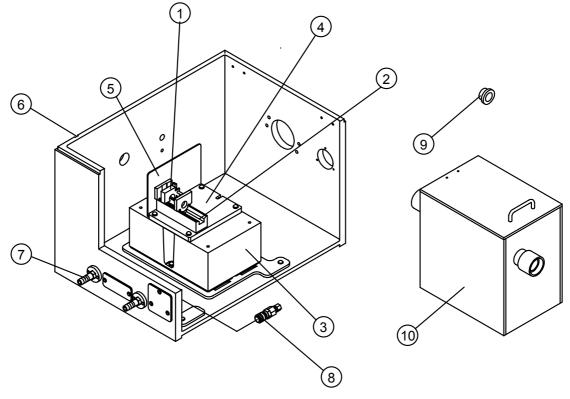
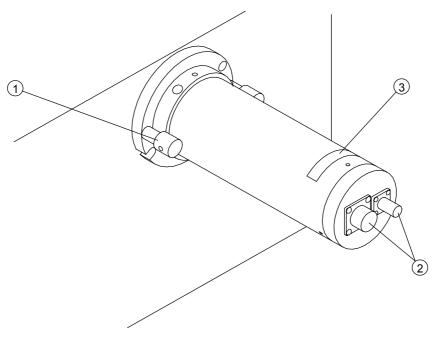


Fig. 3.6 Sample chamber

Component	Function
①Cell holder	Set the cell.
2 Cell holder mount	Set the cell holder.
③Sample stage sub-base (1)	Remove this when a medium-sized accessory like Peltier type thermostatted cell holder is mounted.
④Sample stage sub-base (2)	Remove this when a small-sized accessory like sample changer is mounted.
5 Light shield plate	Changes over the beam diameter to 8 mm or 13 mm according to the cell to be used.
6 Detector protective switch	Turns OFF the switch and sets the voltage applied to the detector to zero when the sample chamber lid is opened.
⑦Constant temperature water inlet/outlet ports	
⑧Joint	Removable joint for constant temperature water.
Window plate	Attached to the sample chamber window when measuring a sample that emits harmful gas to protect the monochromator unit.
Inner sample chamber	Sample chamber to displace nitrogen gas more efficiently.

3.4 Detector Unit





Component	Function
①Lock screw	Secures the detector unit to the main unit.
2 Connector	Connect to the "CD In" connector located on the rear panel of the amplifier unit.
③Label	Indicates the wavelength range. Install the detector unit on the main unit with this label facing upward.

4. MAINTENANCE

4.1 Light Source Check and Replacement

The service life of the xenon lamp is 300 to 500 hours, but it differs considerably from one light source to another. It is therefore difficult to predict life expectancy from operating hours. It is generally predicted from noise on the measured data. Compare the current data with the data on delivery (data obtained with a new light source) to make decision.

Note : Noise may also appear if the line voltage fluctuates abruptly.

Observe the following guidelines in order to maximize the service life of the xenon lamp.

- . If the light source is not used for an extended period, turn it off. However, if the light source will be inactive for an hour or less, leave it on Frequently turning the light source on and off will shorten its service life.
- . Always feed cooling water to the light source (450W light source).

<Procedure>

WARNING : Carefully read the "Safety Cautions" at the beginning of this manual.

- (1) Turn OFF the "Power" switch located on the main unit.
- (2) For the 150W light source, remove the light source cover and then remove the anode holder and the light source. For the 450W light source, remove the anode holder, and then loosen the cathode fixing screw with a screwdriver through the hole on the side cover to remove the light source.

Note : The 150W light source is attached to the anode holder.

- (3) For the 150W light source, remove the anode holder and cathode adapter from the light source, and attach them to a new light source.
- (4) Mount a new light source in the position of the old light source.

CAUTION : Confirm that the glass protrusion of the Xe lamp does not face the M_0 and M_1 mirrors.

CAUTION : Do not mistake the polarity of the Xe lamp.

(150W Light source)

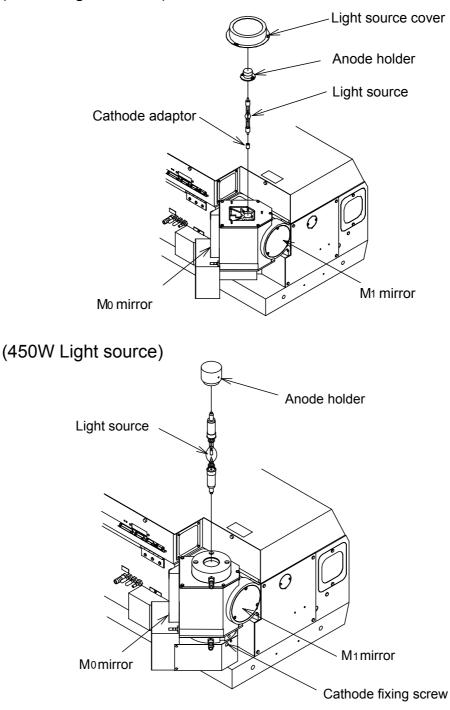


Fig. 4.1

- (5) Tighten the cathode fixing screw (for the 450W light source)
- (6) Mount the anode holder (for the 450W light source).
- (7) Start up the instrument.
- (8) Start up the [Data Monitor] program.
- (9) Select the [Parameter...] and [Data Mode...] commands from the [Setting] menu and designate the monitoring parameters shown in Fig. 4-2.

Monitor - Parameter	Photometric mode	×
Sensitivity: Standard[100mdeg ¥ Besponse: 1 sec ¥ Band width: 1 mm Manual Shinkfith: pre OK Cancel	Num. of channel 2 3 Channel #1: CD Channel #2 HT Channel #2 External 1 Channel #2 External 2	OK Cancel

Fig. 4.2 Setting parameters (replacement of light source)

(10) Select the [Move Wavelength...] command from the [Setting] menu and set the wavelength at "546.1 nm".

Goto Wavelength		×
<u>₩</u> avelength	546.1	nm
OK.	Cancel	1

Fig. 4.3 Setting wavelength (replacement of light source)

(11) Remove the cover from the M_0 and M_1 mirrors. The adjustment screw shown in Fig. 4.4 will be visible.

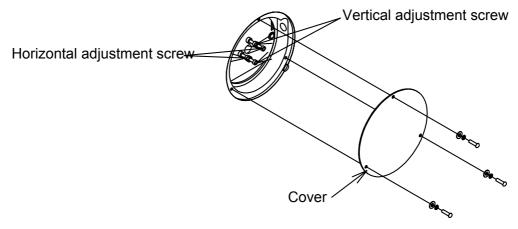


Fig. 4.4 Mirror adjustment screw

- (12) Adjust the adjustment screws for the M₁ mirror to minimize the value of channel 2 (HT voltage) when the sample chamber is empty.
- (13) Adjust the adjustment screws for the M₀ mirror to minimize the value of channel 2 (HT voltage) when the sample chamber is empty.

Note : Normally, the minimum value is 200 to 300 volts.

(14) Re-install the covers for the M_0 and M_1 mirrors.

4.2 Energy Check

The energy of the instrument will decrease over time due to the deterioration of the mirrors and other optical components. This decrease in energy is most apparent in the

shorter wavelength region (250 nm and less). Check the energy about once a year. The M_0 and M_1 mirrors should be replaced every two years, and the M_2 through M_5 mirrors should be replaced every five years.

Note : If an energy decrease is observed in the long wavelength region, improper optical alignment or a other trouble is suspected.

<Procedure>

- (1) Start up the [Data Monitor] program.
- (2) Select the [Parameter...] and [Data Mode...] commands from the [Setting] menu and designate the monitoring parameters shown in Fig. 4.5.

Monitor - Parameter 🛛	Photometric mode	×
Sensitivity: Standard(100mdeg ¥ Besponse: 1 sec ¥ Band width: 1 nm Manual Chine Str. OK. Cancel	Num of channel 2 2 Channel #1: CD 2 Channel #2 HT 2 Channel #2 External 1 2 Channel #4 External 2 2	

Fig. 4.5 Setting measurement parameters (energy check)

(3) Select the [Wavelength ...] command from the [Setting] menu and set the wavelength at "300 nm"



Fig. 4.6 Setting wavelength (energy check at 300 nm)

- (4) Confirm that the value of channel 2 (HT voltage) is 180 to 260 volts when the sample chamber is empty.
- (5) Select the [Move Wavelength...] command from the [Setting] menu and set the wavelength at "300 nm".

Goto Wavelength	×
Wavelength	200.0 mm
OK.	Cancel

Fig. 4.7 Setting wavelength (energy check at 200 nm)

(6) Confirm that the value of channel 2 (HT voltage) is 500 volts or less when the sample chamber is empty.

4.3 Wavelength Accuracy Check and Adjustment

Note : Before checking wavelength accuracy, warm up the instrument for about one hour after turning the light source ON.

Note : Use neodymium glass as the sample.

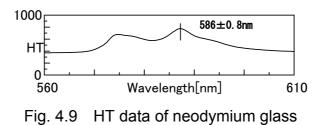
<Procedure>

- (1) Start up the Spectrum Measurement program.
- (2) Select the [Parameter...] command from the [Measurement] menu, and designate the measurement parameters shown in Fig. 4.8.

Spectrum Measurement	t - Parameter 🛛 💌	Spectrum Measurement - Parameter 🛛 😹
Parameters Data M Sensityty: Sjart End Data Pitch: Scanning Mode: Spanning Speed Besponse: Band Width: Scannalation:	ode Data File Option Standard(100mdeg) * 510 rm 560 rm 0.025 rm 20 rm/min * 0.25 rec * 1 rm Magual	Parameters Data Mode Data File Option jijun. of channel 2
OK I	Carcel	OK Cancel Open. Save

Fig. 4.8 Measurement parameters (wavelength accuracy check)

- (3) Mount neodymium glass in the sample chamber.
- (4) Select the [Start] command from the [Measurement] menu in order to perform measurement.
- (5) Using the spectrum analysis program, verify that the peak wavelength of Channel 2 (Fig. 4.9) of the measurement data is 586 \pm 0.8 nm. If the peak wavelength does not fall within this range, adjust the instrument according to the following procedure.



<Adjustment procedure>

Make adjustment using the adjustment screws on the wavelength lever located at the bottom of the main unit.

(1) If the main unit is mounted in the optional cabinet, the wavelength cam and

wavelength lever will be visible from below, as shown in Fig. 4.10. If the main unit is mounted on a table or bench, bridge the instrument across two tables or benches to permit access to the adjustment screws.

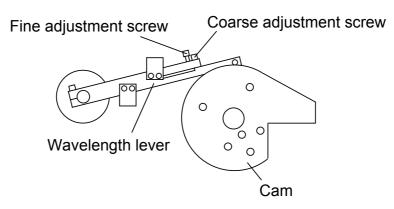


Fig. 4.10 Adjustment screws

- (2) Correct the wavelength by adjusting the fine and coarse adjustment screws located on the wavelength lever. Turning the screw clockwise shifts the HT data to shorter wavelengths, and turning the screw counterclockwise shifts the data to longer wavelengths.
- (3) Measure the HT data of the neodymium glass sample to check the wavelength.

Note : If the instrument is moved, gently move it back to its original position, and check wavelength accuracy again.

4.4 CD Scale Check and Adjustment

- **Note** : Before checking the CD scale, warm up the instrument for about one hour after turning the light source ON.
- **Note** : Use a 0.06% (w/v) aqueous solution of ammonium d-10-camphor sulfonate as the sample.

<Procedure>

- (1) Start up the [Spectrum Measurement] program.
- (2) Select the [Parameter...] command from the [Measurement] menu, and designate the measurement parameters in Fig. 4.11.

Spectrum Measurement - Parameter 🛛 🔀	Spectrum Measurement - Parameter
Spectrum Meanurement - Parameter Image: Control of the second s	Parameters: Data Mode Data File (Option) [jun: of channel: 2
Accumulation: 1 OK Cancel Open Save	OK Carcel Open. Save

Fig. 4.11 Measurement parameters (CD scale check)

- (3) Fill the 10mm light path cell with 0.06% aqueous solution of ammonium d-10camphor sulfonate (solvent : distilled water), and mount the cell in the sample chamber.
- (4) Select the [Start] command from the [Measurement] menu to make measurement.
- (5) Using the spectrum analysis program, confirm that the peak value of Channel 1 (Fig. 4.12) is 190.4 \pm 1 mdeg (291.0 nm). If the peak value does not fall within this range, adjust the instrument according to the following procedure.

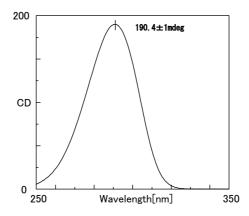


Fig. 4.12 CD spectrum of 0.06% ammonium d-10-camphor sulfonate

<Adjustment procedure>

Use the "Scale Correction CD" knob located on the rear panel of the amplifier unit (Fig. 3.4).

(1) Select the [Move Wavelength...] command from the [Control] menu and designate the wavelength shown in Fig. 4.13. Click on the <OK> button.

Goto Wavelength		×
<u>₩</u> avelength	291.0	nm
OK.	Cancel	

Fig. 4.13 Setting wavelength (CD scale adjustment)

- (2) Turn the "Scale Correction CD" knob located on the rear panel of the amplifier unit (Fig. 3.4) until the CD value comes within the reference range.
- (3) Measure the CD spectrum to check the CD scale.

4.5 Test Signal Check

The test signal is used to check if the electrical system is operating normally. A test signal can be generated by setting the photometric mode at "Test Signal".

<Procedure>

- (1) Start up the [Data Monitor] program.
- (2) Select the [Parameter...] and [Data mode...] commands from the [Setting] menu and designate the measurement parameters as shown in Fig. 4.14.

Sensitivity.	Standar	d(100mdeg 💌
Besponse:	1 sec	
Band width:	1	nm 🗆 Manual
SP width.		

Photometric mode			×
Num. of channel	2 3		0K.
Channel #1:	Test signal	•	Cancel
Channel #2	HT	٠	
Crownel #2	External 1	Ŧ	
Drame #4	External 2	٣	

Fig. 4.14 Setting measurement parameters (Test signal check)

- (3) Verify that the measurement value of Channel 1 falls within 18 (\pm 10%) mdeg.
- (4) After that, set the "Photometric mode" at "CD".

5. TROUBLESHOOTING

If the instrument does not operate properly, the following causes are suspected.

- . Erroneous operation
- . Deterioration of consumable components
- . Failure of instrument

The following table describes basic corrective actions for specific symptoms. If the difficulty cannot be corrected by performing these actions, failure of the instrument is suspected. In this case, contact your local JASCO distributor with detailed information about your difficulty, including the model name, serial number, and date of manufacture of your instrument.

Symptom	Check	Corrective action
Power cannot be turned ON	Is the power cable plugged in to the outlet?	Correctly plug in the cable.
	Is the fuse for the power supply unit intact?	Replace the fuse.
The light source does not light.	Is the check box "Turn ON light source at start" in the "System Setting" dialog box checked?	Turn ON the light source in the "Light source control" dialog box.
	Is the cooling water supplied to the 450W light source sufficient?	Increase the cooling water flow rate.
	Is the cathode fixing screw tight, (450W light source) and are the anode holder and lamp is contact with each other?	Tighten the cathode fixing screw. Adjust the contact plate of the anode holder.
	Is a sparking sound heard?	Replace the lamp.
HT voltage does not increase.	Is the shutter open?	Open the shutter in the "Shutter control" dialog box.
	Is the photometric mode set correctly?	Set the measurement mode at "CD" (not "Test signal").
	Is the "HT voltage setting" in the "Detector sensitivity" dialog box set correctly?	Set it at "Auto" (not "Manu").
	Is the sample chamber lid completely closed?	Completely close the lid.
HT voltage has risen and will not lower.	Is a sample in the sample chamber?	Remove the sample.
	Is the spectral bandwidth setting too small?	Increase the spectral bandwidth.
	Is the cable correctly connected to the detector unit and to the connector located on the back panel of the amplifier unit?	Correctly replug the cable.

	Is the wavelength set to a value at which the detector is not sensitive?	Set the wavelength to a value at which the detector is sensitive.
	Is the nitrogen gas flow rate high enough when the wavelength is set below 180 nm?	Increase the nitrogen gas flow rate.
	Is the "HT" switch on the sub- panel of the amplifier unit in the "Auto" position?	Set the "HT" switch to "Auto".
Noise is high	Is the spectra bandwidth setting too small?	Increase the spectra bandwidth.
	Does the sample have high light absorption?	Decrease the sample concentration, or shorten the light path of the cell.
	Is noise detected in the HT voltage?	Replace the Xe lamp.
	Is the HT voltage at below 250 nm too high?	Adjust the M_0 and M_1 mirrors.
	Any noise source that generates electromagnetic waves nearby?	Remove the noise source from the proximity of the instrument.
	Is any source of mechanical vibration nearby?	Remove the source of vibration.
	Does line voltage vary abruptly.	Use stabilized line voltage.
The baseline curves sharply.	Is the curvature within ±10 mdeg when the baseline is not corrected?	Perform baseline correction.
CD value is displayed although sample is not optically active.	Is the sample fluorescent?	Decrease sample absorbance to 2 or less.
	Is the sample a film or liquid crystal?	False CD signal from the sample is suspected.
	Does the cell contain any optically active residue?	Prepare a new sample.
The displayed CD value is smaller than normal, or no display appears.	Is the "HT voltage setting" in the "Detector sensitivity" dialog box set correctly?	Set it at "Auto" (not "Manu").
	Is the photometric value 18±2 mdeg when the photometric mode is "Test signal"?	Failure of the electrical system or modulator element is suspected.
Repeatability of CD values is low.	Is the instrument warmed up sufficiently?	Before performing Cd measurement, warm up the instrument for approximately one hour after the light source has lit.

	Is the sample deteriorated by the light emitted from the light source? Is the variation of the peak value of aqueous solution of ammonium d-10-camphor sulfonate (distilled water, 10 nm cell) at 291.0 nm within 2 mdeg/hr?	Use the shutter function or narrow the spectrum bandwidth. Normal.
	Are room temperature and humidity variations normal?	Maintain room temperature within 20 \pm 5°C, and humidity below 70%.
	Is the instrument blown with draft from an air conditioner or other?	Install the instrument in a position outside of the direct path of air currents.
	Is the scanning speed too high?	Slightly lower the scanning speed.
	Is noise too high?	Increase response. Or, refer to the "Noise is high" symptom.
Wavelength repeatability is low.	Is the instrument warmed up sufficiently?	Measure wavelength repeatability after warming up the instrument for approximately one hour after the light source is lit.
	Are room temperature and humidity variations too high?	Maintain room temperature within 20 \pm 5°C, and humidity below 70%.
No communication with the computer.	Is the RS-232C cable connected properly?	Reconnect the cable correctly.
	Is the communication port setting in agreement with the wiring?	Correctly set the communication ports.