

Jasco J-810 Spectropolarimeter

Operation instructions

Turn the system on

Please, follow exact instructions for turning the system on. Not following the procedure may result in damage of the equipment.

1. Turn on N_2 gas cylinder (Main valve). The flowmeter is located near sample compartment and should read at least ~35 SCFH measured by the top of the floating metal cylinder. Wait for 15 minutes.

If the flow is less or bigger than 35 SCFH, adjusted by the knob on the flow meter. In case you are not able to increase flow to sufficient level by this knob, you may try to increase the incoming pressure by the control valve close to the nitrogen tank.

Please, check the flow every 3-5 minutes during the whole time of your measurements. Please, do not leave the instrument operating unattended for more than 3-5 minutes.

- 2. **Turn on the Jasco J-810 power switch** located at the lower left. You will hear an audible sounds. The shutter LED will turn green.
- 3. Start the Spectra Manager Program.
 - Select Spectrum Measurement.



• A self-diagnosis will be performed and the Xe lamp should automatically turns ON. The LED lamp indicator on the instrument turns green. It is recommended to wait for about 10min to warm the lamp up before proceeding.

4. Spectra Measurement menu.

When selecting the [Spectra Measurement] menu, the following window appears:

🙀 Spectra Measurement - J81	0/B011660750						- C	3	×
<u>File Measure Control Vie</u>	w <u>H</u> elp								
😁 260.0 nm 🛛 🖊	Acc. 0/3	0.08 mdeg	286.1 v						
🕒 📼 📴 📮 💷	🛼 🚉 🖳	💌 🐺 🚱 📀	👌 🗷 🖂 🔤 📽 🏢 🐘						
	×	1							-
Information									
Item	Contents	-							
🗆 🖅 J810	idle	1000							
234 Serial No.	B011660750	0.5							
Cell units	Not attached								
🗉 😼 Instrument State									
PMT voltage	Auto								
Light source	On	CD [mdeg] 0							
Chutter	Open								
🗉 👜 Sample Information									
Sample Name		0.5							
Conc.		-0.5							
Solvent									
) Comment									
Parameters				1		1			7
Photometric mo	CD								
Photometric mo	HT	1000							- I
_] Sensitivity	Standard								
]D.I.T.	1 sec								
Bandwidth	1.00 nm	800-							
_ Measurement ra	260 - 190 nm								
_ Data pitch	1.U nm								
i Scanning speed	50 nm/min	600 -							
Start mode	Immediately								
Chutter control	Manual	HT [V]							
CD detector	PMT	400 -							
	3 times								
Baseline	Nothing	-							
	ritoning	200-							
		0				· · · · · · · · · · · · · · · · · · ·	 		
		190	200		220	240			260
	>	1.4.1.4.1			vvavelength [nm]				_
Ready							NU	М	

File menu:

- Save data. Saves experimental setup data, measurement parameters, and set terms as a .jws format file.
- Analysis Send. Transfers experimental data to the [Spectra Analysis] window, which starts up automatically when [Analysis Send] is used.
- **Open Baseline.** Opens baseline data file, which are .cdsl format files.
- Save Baseline. Saves baseline data to disk as .cdsl format files.
- **Open Parameters.** Opens parameters which are set in the [Parameter Settings] dialog box. The file format is .cdsp.
- Save Parameters. Save parameters as a .cdsp format file.
- Exit. Exits the [Spectra Measurement] window.

Measure menu

- **Parameters.** Sets parameters, turns [Baseline] measurements and auto-shutter control ON/OFF, edits comments and saves and loads measurement parameters and data.
- **Sample.** Starts measurement of sample.
- **Baseline.** Starts [Baseline] measurement.
- **Cancel.** Stops blank and sample measurement.

Control menu

- Move Wavelength. Moves the current wavelength to desired value.
- **CD Detector.** Sets detector (CD/ORD/LD/UV) sensitivity.
- Light Source. Turns the light source ON/OFF. Displays/clears the light source operating time.
- **Shutter.** Opens or closes the shutter (when installed).
- Auto Zero. Zeroes the photometric value of ORD-M (when installed).
- Accessory. Registers compatible accessories.

5. Measurement menu / Parameters.

Sets parameters, turns shutter ON/OFF for blank measurements and auto-shutter drive, edits comments and saves and loads measurement parameters and data onto or from disk. The parameters dialog box is made up of four dialog boxes: [General], [Control], [Information] and [Data]. They can be changed by clicking the appropriate tab at the top of the box. Clicking the tab [Data], for example, when the [Parameters Settings] dialog box is active displays the [Data] dialog box. The dialog boxes are described below.

General	Control	Information Data			
Photon	netric mod	de	Start:	260	nm
Channe	els Num:	2	End:	190	nm
Cha	nnel #1:	CD ~	Data pitch:	1.0 nm 🗸 🗸	
Cha	nnel #2:	HT ~	Start mode:	Immediately	~
Cha	nnel #3:	External.1 💦 🗸 🗸	Scanning mode:	Continuous	~
Cha	nnel #4:	External.2 \sim	Scanning speed:	50 nm/min	~
Sensitivit	y:	Standard v			
D.I.T. :		1 sec 🗸	Accumulation/Re	epeat	
Band wid	dth:	1.00 nm	Accumulations:	3	
🔲 Slit w	idth:	100 um			

- Photometric mode.
- Channel Num. Maximum of four channels. Four types of data can be acquired simultaneously.
- Channel #1 The selectable range varies with an optional accessory. CD: Circular dichroism signal, which is given by ~AC/DC with 50 kHz. CD/DC: This is the same with CD mode when DC is set to 1 V. This mode is useful when the DC varies (i.e., HT is fixed at an arbitrary value) with the sample absorption.
- **Channel #2.** Selectable range: HT: Photomultiplier tube voltage, DC: Photomultiplier tube DC voltage, and Abs: calculated absorption value using the HT value.
- Sensitivity. Selects the sensitivity from the drop-down list. Low Up to 1000 mdeg Standard Up to 200 mdeg, High Up to 5 mdeg.
- **D.I.T.** Digital integration time per data. When the scan mode is set to **Step** (Auto Response), designate a range (in this case, the effective value extends to msec order).

- **Band Width.** Sets the spectrum band width. Range: 0.01 nm 15 nm, which is can be specified to two decimal places.
- Slit Width. Sets the slit width. Can be entered by inserting a check mark in Slit Width. Range: 30 μ m to 3000 μ m (integer values only).
- Start. Measurement start wavelength (long wavelength end). Range: 163 nm 1100 nm.
- End. Measurement end wavelength (short wavelength end). Range: 163 nm 1100 nm.
- **Data Pitch.** Selects the data acquisition interval from the drop-down list. Effective value extends to ms order.
- **Start Mode.** This depends on the external trigger, which is intrinsic to an accessory. Immediate: Performs a measurement without an external trigger. Wait for trigger: Sets whether measurement is to be started by an external trigger or not.
- Scanning Mode. Selects continuous scan or step scan as the scanning mode. Continuous mode: Performs "continuous scanning". Step mode: Performs "step scanning". Step (Auto Response) mode: Performs "step scanning" in which the DIT is variable.
- Scanning Mode. Selects continuous scan or step scan as the scanning mode. Continuous mode: Performs "continuous scanning". Step mode: Performs "step scanning". Step (Auto Response) mode: Performs "step scanning" in which the DIT is variable.
- Scanning Speed. Selects a wavelength scanning speed. Can be entered when the scan mode is set to Continuous.
- Accumulation/Repeat. Sets the number of the accumulations for a measurement. Can be entered by inserting a check mark. Range: 1 999 times. Cycle Times sets the number of measurements, which defines the number of the spectra acquired. Can be entered by inserting a check mark when Accumulation is off. Range: 1 999 times. Cycle Interval sets the time between measurements. This is displayed when a number is entered in the Cycle Times box. Range: 0 15000 sec.
- **Open** Loads the setting parameters from the disk. Clicking this button displays the **Open dialog box**. Selecting a baseline data file and clicking the **Open** button loads the baseline data into the memory. The data is in a .cdsp format file.
- Save. Saves the parameters in a .cdsp format file.
- **Default**. Reverts to the default parameters.

Control dialog box. Turns baseline correction ON/OFF and auto shutter driving. If baseline correction is turned on, the baseline data will be subtracted from the sample spectra .

Parameter Settings	×
General Control Information Data	
Correction	
○ None	
<u>Shutter is opened and closed automatically</u> CD <u>D</u> etector : PMT	
<u>Open</u> <u>S</u> ave <u>D</u> efault <u>OK</u> Cano	el

- **Baseline.** Turns the baseline term ON, which results in a spectra measurement where the horizontal axis is time.
- None. Turns the correction OFF.
- Auto shutter is automatically opens the shutter just before measurement starts. Uncheck this function.

arameter	Settings		×
General	Control	Information Data	
<u>S</u> am	ple name:	:	
Оре	rator:		
<u>D</u> ivi	sion:		
Corr	nme <u>n</u> t:		
C <u>o</u> n	centration	κ mol/L ~	
Sol <u>v</u>	ent:		
	<u>D</u> isplay the	e [Comment] dialog box before measurement	
		Open Save Default OK	Cancel
		<u>D</u> efault OK	Cancel

Information dialog box. Set sample name, operator, division, comment, solvent, etc.

Parameter Setting	s	\times
General Contro	Information Data	
Save data		
Auto save		
Save jn:	Browse	
<u>F</u> ormat:	Date-No. \sim	
<u>S</u> tring:		
Print	to Spectra Analysis	
Print meas	ured data	
<u>T</u> emplate:	Browse	
	Open Save Default OK Cano	cel

Data dialog box. Sets automatic saving, transfers to Spectra Analysis.

- Save Data. Sets whether measured spectrum is to be saved automatically or not. If a check mark is put in the [Auto Save] box, enter a dialog box where a filename and directory are defined. This is useful for the repeatable measurement.
- Save in. Puts the file and directory name. The Browse button is useful for assigning the address.
- **Format.** Date-No. displays date and file number. Sample-No. displays sample name and file number.

6. Making measurements

• Click or select **Parameter** from **Measure** menu. The following dialog box appears.

Parameter Settings				×
General Control	Information Data			
Photometric mod	de	Start:	260	nm
Channels Num:	2 🛉	End:	190	nm
Channel #1:	CD ~	Data pitch:	1.0 nm 🔍 🗸	
Channel #2:	HT ~	Start mode:	Immediately	~
Channel #3:	External.1 ~	Scanning mode:	Continuous	~
Channel #4:	External.2 \vee	Scanning speed:	50 nm/min	\sim
Sensitivity:	Standard 🗸 🗸 🗸			
D.I.T. :	1 sec 🗸	Accumulation/Re	epeat 1	
Band width:	1.00 nm	Accumulations:	3	
Slit width:	100 um			
	Open Save	Default	OK	Cancel

Parameters currently displayed in the [Parameters] dialog box are commonly used for checking CD accuracy using *d*-10-ammonium camphorsulfonic acid (NH4-CSA) in a pure water solution.

- Set Sensitivity. Usually, standard mode is employed, which is effective up to ± 200 mdeg. For measuring small CD values, the low mode is the most effective. On the other hand, for large CD values, greater than ± 200 mdeg, the high mode should be employed.
- Set **Start Mode**. If the external trigger is not required such is the case when using stopped flow accessories, use the immediate mode.
- Set **Start** and **End**. Enter the upper wavelength into the **Start** text box, and the lower wavelength into the **End** text box.

- Set Scan Mode. Almost all measurements can be performed in Continuous mode. In rare cases, gas phase CD measurements for example, "step scanning" should be employed. When selecting Step (Auto Response), the limits for the D.I.T. value must be set.
- Set Scan Speed.
- Set **Band Width**. Input the half-width value in the **Band Width** text box.
- Set **D.I.T.** This is restricted by the **Scanning Speed** and by the **Band Width**. Supposing that the **Scanning Speed** and the **Band Width** have the values given like in figure. In this case, a single scan takes 1.2 sec/nm. So a band width of 1.0 nm is reasonable if the **D.I.T.** is set to be 1 sec or less. But a **D.I.T.** of over 1.2 sec is often preferred because the liquid phase broadens the CD spectrum considerably. An appropriate **D.I.T.** value should be selected by considering such broadening with the specified **Scanning Speed** and **Band Width**.
- Set Data Pitch.
- Set Accumulation Times. The cycle time can also be specified.
- Set the Control parameters and the values of Information and Data.
- 7. Measuring the baseline.

Clicking the **Baseline** from the **Measure** menu or the following button:



Click **Measure** and the sample spectrum is acquired and the progress of measurement is displayed on the screen.

8. Measuring the sample.

Clicking the **Sample** from the **Measure** menu or the following button:



The measured spectrum is automatically will be send to **Spectra Analysis** if this option has been selected in **Data** tab of **Parameter Settings**.

If this option has not been checked, the data can be sent to **Spectra Analysis** via **File** / **Send to Spectra Analysis**.

Parameter	Settings		×
General	Control	Information Data	
Save	data		
	ito save		
Save	jn:		<u>B</u> rowse
<u>F</u> orm	at:	Date-No. \sim	
<u>S</u> tring	J:		
Send Se Primt □ Er	end <u>d</u> ata to int measur	o Spectra Analysis ed data	
<u>T</u> emp	olate:		B <u>r</u> owse
		<u>Open</u> <u>S</u> ave <u>D</u> efault	OK Cancel

9. Variable temperature program.

To use the Peltier Temperature Control Device which is already mounted inside sample compartment do the following:

- Switch on the water circulation at the computer tower.
- Turn ON Jasco PFD-425 Temperature control device that is next to monitor.
- Follow to menu Control, then Accessory. Select available accessory Jasco Peltier Type (Single RTE off).

Spectra	a Manager				
Program	Application View	<u>H</u> elp			
lnstr	🐙 Spectra Measurem	ent - J810/B0116	660750		
	<u>File Measure Con</u>	rol <u>V</u> iew <u>H</u> e	lp		
Mov	e Wavelength	Acc.	0/3	0.36	mdeg 284.7 v
CD E Ligh	Detector t Source	10P 🥞	B., D.,	P 🖓 🚱	😵 <u>२ -</u>) 🗷 🖂 📟 📽 🋲 🐘
Shut	ter		×		1
ORD	-M Zero Clear	ion			
Cala		Co	ontents		- 1
Selec	ct Accessory		idle		
	1234 Serial No.	B011	1660750	(0.5
- 🧶 Ir	Cell units	Not a	attached		
- 👬 C	🗆 🗟 Instrument S	ate			
- 👯 C	PMT volt	age /	Auto		
🚟 d .	🙀 🔄 Light source		On	CD [mdeg]	
23 F) Shutter	C	Dpen		
	🗉 💩 Sample Infor	nation			
	🗋 Sample N	ame			0.5
	Conc.			-(-0.5

• Select available accessory - Jasco Peltier Type (Single RTE off).

Name	Model	Status	S
🗹 🎒 Peltier Type FDCD Cell Holder	PFD-425S	Not Con	A00
<			>
Name	Model	Status	S

• After selecting the Peltier thermostatted cell holder, the following window will appear:



• After the initialization, the additional section in measurement program will appear:

Control: -10	20.	00 C	Holder 110
Current:	34. 34.	36 C 00 C	Holder * Cell
ltem		Value	8
Cell Length(mr Remark	n)	10.00	00
			Initializa

Temperature tab:

Parameter S	ettings					×
Temperatu	re Start/End	General Contr	ol Information	Data		
Start temp	perature: 20		Reverse H	old time: 0	sec	
	Sampling(C)	Target(C)	Ramp Rate(C/mir	Wait(sec)		
1	1.0	30	1.0		0	
2						
Control	sensor		Monitor senso	r		
● H	older 🔿 Ce	I	Hold	ler 🔿 Cell		
🗹 Halt te	emperature ramp	ing during measu	irement.			
	Open	Save	Default	ОК	Cance	el 🛛

- Sets the initial and target temperatures. Temperature increases during course of measurement. Temperature range: -10.0°C 110.0°C. Temperature can be specified to the first decimal place.
- **Sampling.** Selects the data acquisition interval. Range: $0.1^{\circ}C 10.0^{\circ}C$.
- **Ramp Rate**. Sets the temperature increasing and decreasing rate (temperature/time). The units °C/minute or °C/hour can be selected. Range: 0 10°C/min and 0 600°C/hr. It can be specified to the sixth decimal place.
- Wait. Sets the delay time, which is the time before the measurement commences after the target temperature has been reached. Range: 0 sec 3600 sec, where only integers can be entered.
- Reverse. After the completetion of measurement will make reverse measuremen.

Start/Endl tab:

arameter Sett	ings					×
Temperature	Start/End	General Contro	I Informatio	n Data		
Start condi	tion					
🖲 Кеер	within +/-	0.1 C of the temperature	ne target arature for	5	sec	
	the target ten	nperature 3	ti	mes		
End condit	on n to start temp	perature				
<u> </u>	end temperat	ure				
🔿 Chan	ge to specified	d <u>t</u> emperature	20.0	С		
	Open.	. <u>S</u> ave	<u>D</u> efaul	t 🚺	ок	Cancel

• Start Condition. Two criteria for attaining the target temperatures are presented.

CASE I: Determines the temperature range (°C) and the time (seconds) after the temperature enters the specified temperature range. The ranges are $0 - 10^{\circ}$ C and 0 - 60 sec, respectively. They can be specified to the first decimal place and as an integer, respectively.

CASE II: Determines the number of crossing points (times) with the target temperature. Range: 0 - 10 times.

General tab:

Parameter Settings	÷				\times
Temperature Star	t/End Gener	al Control	Information	Data	
Photometric mod	le 2 🛋		Wavelength:		_
Channel # <u>1</u> :	CD	~	1	Wavelength [nm] 260.0	
Channel # <u>2</u> :	HT	\sim	2		
Channel # <u>3</u> :	External.1	\sim			
Channel # <u>4</u> :	External.2	\sim			
Sensiti <u>v</u> ity:	Standard	~			
<u>D</u> .I.T. :	1 sec 🛛 🗸				
Band width:	1.00	nm			
Slit width:	100	um			
	0pen	<u>S</u> ave	<u>D</u> efault	OK Cano	el

- **Wavelength**. Wavelength of interest at which the variable temperature curve will be measured.
- Channel Num. Maximum of four channels. Four types of data can be acquired simultaneously.
- **Channel #1** The selectable range varies with an optional accessory. CD: Circular dichroism signal, which is given by ~AC/DC with 50 kHz. CD/DC: This is the same with CD mode when DC is set to 1 V. This mode is useful when the DC varies (i.e., HT is fixed at an arbitrary value) with the sample absorption.
- **Channel #2.** Selectable range: HT: Photomultiplier tube voltage, DC: Photomultiplier tube DC voltage, and Abs: calculated absorption value using the HT value.
- Sensitivity. Selects the sensitivity from the drop-down list. Low Up to 1000 mdeg Standard Up to 200 mdeg, High Up to 5 mdeg.

- **D.I.T.** Digital integration time per data. When the scan mode is set to **Step** (Auto Response), designate a range (in this case, the effective value extends to msec order).
- **Band Width.** Sets the spectrum band width. Range: 0.01 nm 15 nm, which is can be specified to two decimal places.
- Slit Width. Sets the slit width. Can be entered by inserting a check mark in Slit Width. Range: 30 μ m to 3000 μ m (integer values only).
- **Open** Loads the setting parameters from the disk. Clicking this button displays the **Open dialog box**. Selecting a baseline data file and clicking the **Open** button loads the baseline data into the memory. The data is in a .cdsp format file.
- Save. Saves the parameters in a .cdsp format file.
- **Default**. Reverts to the default parameters.

Control tab:

Parameter Sett	ings						×
Temperature	Start/End	General	Control	Information	Data		
Correction							
● <u>N</u> one		0	Blan <u>k</u>				
<u>S</u> hutter is	opened and	l closed au	itomatical	y			
			1				
	<u> </u>	<u> </u> <u>S</u>	ave	<u>D</u> efault		OK	Cancel

• Turns the blank correction ON/OFF and auto shutter driving. If blank correction is turned on, the blank data will be subtracted from the sample spectra .

Information tab:

Parameter Setti	ngs						×
Temperature	Start/End	General	Control	Information	Data		
<u>S</u> ample n	ame :]	
Operator :]	
<u>D</u> ivision :]	
Comme <u>n</u> t	:]	
Concentra	ation :	r	nol/L	\sim			
Sol <u>v</u> ent :]	
Displa	y the [Comm	ent] dialog	g box befo	ore measureme	ent		
	(<u>O</u> pen	<u>S</u>	ave	<u>D</u> efault		OK	Cancel

• Set sample name, operator, division, comment, solvent, etc.

emperature	Start/End	General	Control	Information	Data		
Cause data							
Auto saj	ve 						
Savejn:						Browse	
<u>F</u> ormat:	Date-N	lo.		~			
String:							
Send to ana	alysis ata to Stand	ard <u>A</u> nalys	is				
Send to ana	alysis ata to Stand	ard <u>A</u> nalys	is				
Sendto ana	alysis ata to Stand	ard <u>A</u> nalys	is				
Send to ana	alysis ata to Stand	ard <u>A</u> nalys	is				

- Save Data. Sets whether measured spectrum is to be saved automatically or not. If a check mark is put in the [Auto Save] box, enter a dialog box where a filename and directory are defined. This is useful for the repeatable measurement.
- Save in. Puts the file and directory name. The Browse button is useful for assigning the address.
- Format. Date-No. displays date and file number. Sample-No. displays sample name and file number.
- Send to analysis. If this option has not been checked, the data can be sent to Spectra Analysis via File / Send to Spectra Analysis.

10. Shutdown Procedure

Please, follow exact instructions for turning the system off. Not following the procedure may result in damage of the equipment.

• Turn off the Xe lamp by going to Control / Light Source.



• Click on the "light box" to switch off the light source:



Make sure the LED lamp indicator on the instrument has switched off.

- If in use, turn off the water Circulator and the temperature control device.
- Close Spectra Analysis program.

- Close **Spectrum Measurement** (Variable Temperature or any other program that was in use).
- Close **Spectra Manager**. The following window will appear and drivers will be terminated:

🔊 Spectra Manager	- 10- es 1						- D ×
Program View Help Instrument Statument Statument Viidation Interval Scan Measurement Temperature Measurement Temperature Interval Measurement Temperature (Navelength Scan Measurement Analysis Statument Interval Data Analysis ORD-E Data Correction Statument CD Multivariate SEE File Converter II	Instrument J810	Model Name J-810	Serial No. B011650750	Condition Sleep	Username	Terminaling the drivers	
Keady							

- Only after the drivers have been terminated, turn off the CD instrument.
- Wait for 15 minutes then shut OFF the main value of N₂ tank.