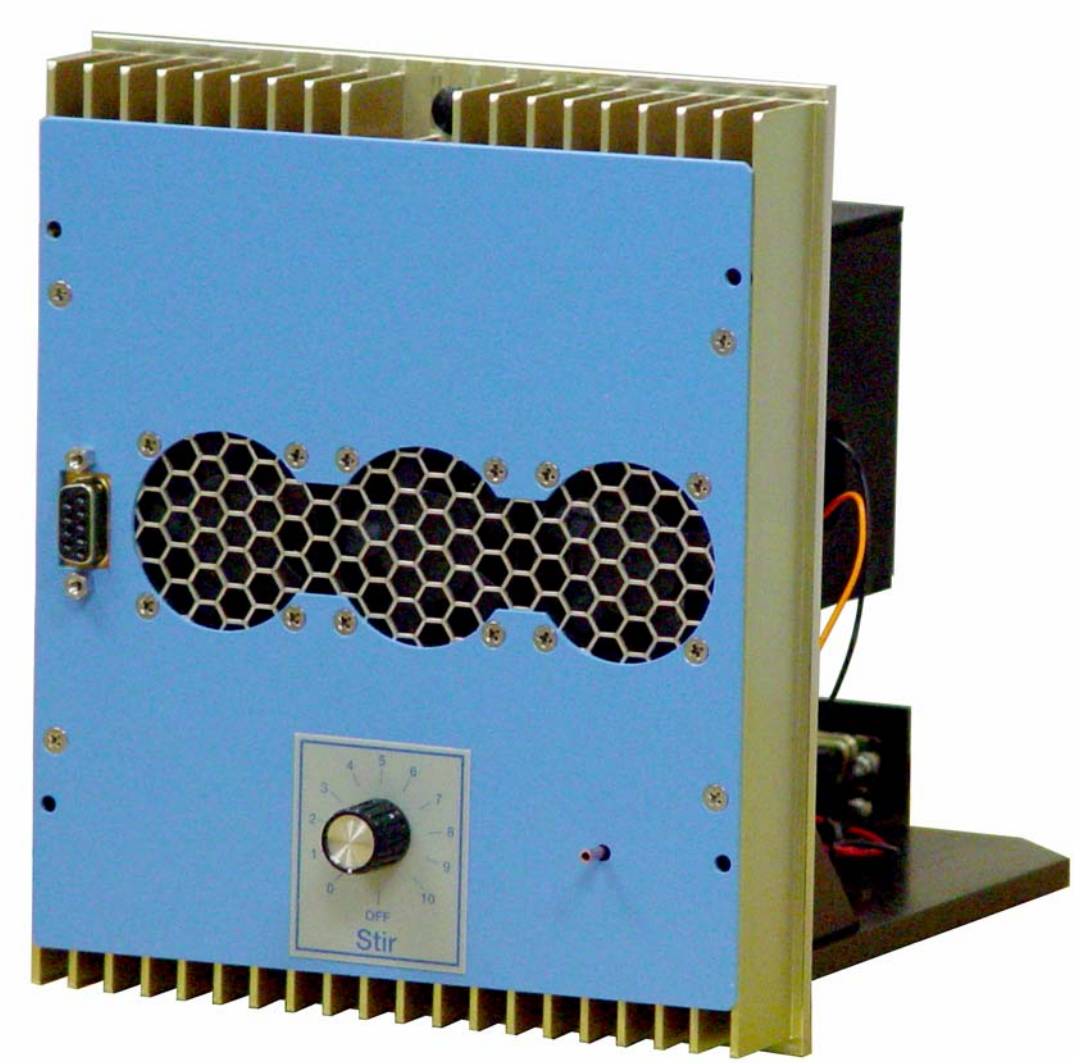
HORIBA Scientific

Peltier Sample Cooler
F-3004

Operation Manual
Part number J81049 rev. C

F-3004 Peltier Sample Cooler



Operation Manual v. 2.0

www.jobinyvon.com

August 2006

Part Number 81049

Table of Contents

0: Introduction

The Peltier effect

Jean Peltier discovered a thermoelectric effect in 1834, in which electricity flowing through certain materials causes a temperature gradient across those materials. Specifically, Peltier noticed that an electric current moving through two different conductors can cause emission or absorption of heat. That is, when electricity flows through a substance such as bismuth telluride (Bi_2Te_3), heat is moved across the material, creating a warmer side and a colder side. Modern research on the "Peltier effect" shows that the charge carriers in doped semiconductors push heat in the direction of their motion. Therefore, reversing the current's direction changes the warm side to the cool side, so that the Peltier device may be used to heat a sample as well as cool it.

Peltier devices are used to cool integrated circuitry, semiconductor photodetectors, and even food in picnic coolers. Their advantages over conventional cooling units are: no moving parts, small size, no refrigerant fluids, no vibration or mechanical noises, and high stability and long life. Their major disadvantage is their relative inefficiency. The Peltier Sample Cooler is an automated temperature controller for samples inside the Spex® series of spectrofluorometers. Thermoelectric heating and cooling controls the sample temperature faster than conventional water baths. A magnetic stirring system to keep your sample homogeneously mixed, and a port for nitrogen gas purging (to reduce condensation on a chilled sample), are included in the Peltier Sample Cooler.

Disclaimer

By setting up or starting to use any HORIBA Jobin Yvon product, you are accepting the following terms:

You are responsible for understanding the information contained in this document. You should not rely on this information as absolute or all-encompassing; there may be local issues (in your environment) not addressed in this document that you may need to address, and there may be issues or procedures discussed that may not apply to your situation.

If you do not follow the instructions or procedures contained in this document, you are responsible for yourself and your actions and all resulting consequences. If you rely on the information contained in this document, you are responsible for:

- Adhering to safety procedures
- Following all precautions
- Referring to additional safety documentation, such as Material Safety Data Sheets (MSDS), when advised

As a condition of purchase, you agree to use safe operating procedures in the use of all products supplied by HORIBA Jobin Yvon, including those specified in the MSDS provided with any chemicals and all warning and cautionary notices, and to use all safety devices and guards when operating equipment. You agree to indemnify and hold HORIBA Jobin Yvon harmless from any liability or obligation arising from your use or misuse of any such products, including, without limitation, to persons injured directly or indirectly in connection with your use or operation of the products. The foregoing indemnification shall in no event be deemed to have expanded HORIBA Jobin Yvon's liability for the products.

HORIBA Jobin Yvon products are not intended for any general cosmetic, drug, food, or household application, but may be used for analytical measurements or research in these fields. A condition of HORIBA Jobin Yvon's acceptance of a purchase order is that only qualified individuals, trained and familiar with procedures suitable for the products ordered, will handle them. Training and maintenance procedures may be purchased from HORIBA Jobin Yvon at an additional cost. HORIBA Jobin Yvon cannot be held responsible for actions your employer or contractor may take without proper training.

Due to HORIBA Jobin Yvon's efforts to continuously improve our products, all specifications, dimensions, internal workings, and operating procedures are subject to change without notice. All specifications and measurements are approximate, based on a standard configuration; results may vary with the application and environment. Any software manufactured by HORIBA Jobin Yvon is also under constant development and subject to change without notice.

Any warranties and remedies with respect to our products are limited to those provided in writing as to a particular product. In no event shall HORIBA Jobin Yvon be held liable for any special, incidental, indirect or consequential damages of any kind, or any damages whatsoever resulting from loss of use, loss of data, or loss of profits, arising out of or in connection with our products or the use or possession thereof. HORIBA Jobin Yvon is also in

no event liable for damages on any theory of liability arising out of, or in connection with, the use or performance of our hardware or software, regardless of whether you have been advised of the possibility of damage.

Safety summary

The following general safety precautions must be observed during all phases of operation of this instrument. Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture and intended use of instrument. HORIBA Jobin Yvon assumes no liability for the customer's failure to comply with these requirements. Certain symbols are used throughout the text for special conditions when operating the instruments:

Warning:

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or similar that, if incorrectly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met. HORIBA Jobin Yvon Inc. is not responsible for damage arising out of improper use of the equipment.

Caution:

A CAUTION notice denotes a hazard. It calls attention to an operating procedure, practice, or similar that, if incorrectly performed or adhered to, could result in damage to the product. Do not proceed beyond a CAUTION notice until the indicated conditions are fully understood and met. HORIBA Jobin Yvon Inc. is not responsible for damage arising out of improper use of the equipment.

Warning:

Hot! This symbol warns the user that hot equipment may be present, and could create a risk of fire or burns.

Read this manual before using or servicing the instrument.

Note:

General information is given concerning operation of the equipment.

1: Requirements & Installation

Requirements

Computer requirements

- Dedicated serial COM port
- FluorEssence™ software
- Windows® XP or higher
- 973005 or equivalent PC computer

Electrical requirements

- 115 VAC or 230 VAC ±15% (switch selectable), 60 or 50 Hz
- 25 W average with output off; 55 W average with output on

Physical Requirements

- Controller must be within 36″ (90 cm) of Peltier Sample Cooler (communications cable length)

Installation

1 Unpack and inspect all components.

a The following Peltier Sample Cooler components should be included:

Quantity	Description	Part Number
1	Peltier Sample Cooler	400313
1	Sample-cooler cover	
1	Control unit with 50-Ω BNC terminator	LFI-3751
1	Peltier Sample Cooler Operation manual	81049
1	*Wavelength Electronics™ User's Guide* with extra parts	
1	9-pin-male-to-9-pin-female cable	400367
1	9-pin-to-25-pin RS-232 cable	35506
1	Power cord (115 V or 220 V)	98015 or 98020
4	#4-40 × 1¼" screw for FluoroMax® installation	600105
4	#4-40 × 1½" screw for Fluorolog® installation	600104
1	Magnetic stirring bar	53044
1	Certificate of Calibration for control unit	
1	3.5" diskette (only if Peltier Sample Cooler was purchased separately form the spectrometer)	

b Inspect all the components for signs of damage that may have occurred during transit. If damage is evident, do not continue with the installation. Notify HORIBA Jobin Yvon and the shipper at once.

Note: *Many public carriers do not recognize claims for concealed damage reported later than 15 days after delivery. For a shipping damage claim, inspection by the carrier agent normally is required. Therefore, the original packing material should be kept as evidence. While HORIBA Jobin Yvon is not liable for damage occurring during transit, the company will extend every effort to aid and advise.*

2 Read this instruction manual thoroughly before installing the Peltier Sample Cooler.

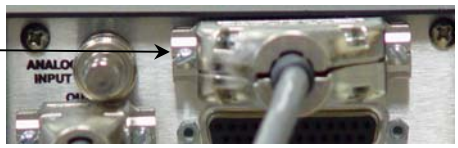
3 Install the Peltier Sample Cooler:

a Remove the existing sample holder from the sample compartment of the spectrofluorometer.

b Install the control unit as directed in the *Wavelength Electronics™ User's Guide* (Chapter 1, "Preparing the Temperature Controller for Use," steps A through F).

c Make sure that the 50-Ω BNC terminator is attached to the ANALOG INPUT on the back of the controller.

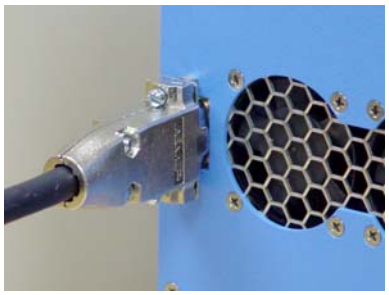
d Connect the 25-pin–9-pin adapter cable's 25-pin female end to the RS-232 OUTPUT on the back of the control unit.



e Attach the 9-pin female end of the the 25-pin–9-pin adapter cable to the COM2 port on the host computer.

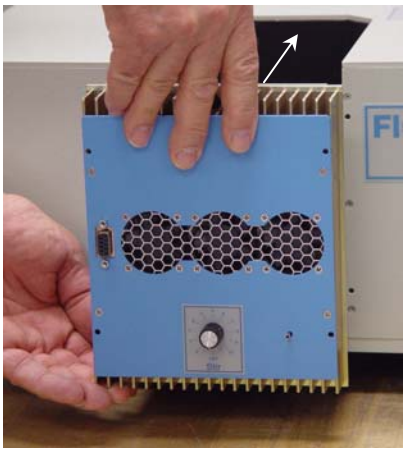
f Connect the 9-pin-female–9-pin-male's female end to the OUTPUT jack on the back of the control unit.

g Attach the 9-pin-female–9-pin-male adapter cable's male end to the jack on the front of the Peltier Sample Cooler.



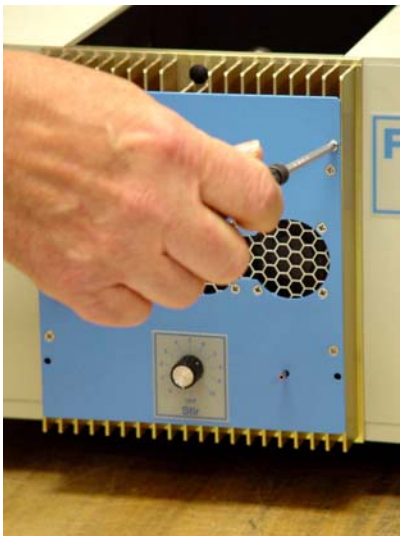
h Insert the power cord into the jack on the control unit. Plug the other end of the power cord into a wall receptacle.

i Place the Peltier Sample Cooler into the sample compartment.



***Note**: Be sure that the communications jack inside the sample cooler is firmly seated in the connection within the sample compartment.*

j Attach the Peltier Sample Cooler to the front panel of the sample compartment using four #4-40 screws.



***Note**: For installation in a Fluoro-Max®, use the shorter screws; for installation in a Fluorolog®, use the longer screws.*

k *(optional)* If condensation may be a problem for the chilled sample, attach a dry-nitrogen gas line to the small metal purging inlet on the front of the sample cooler.

4 Install the software on the host computer.

(only if the Peltier Sample Cooler was purchased separately from the spectrofluorometer)

5 If the Peltier was installed separately, add it to your instrument configuration.

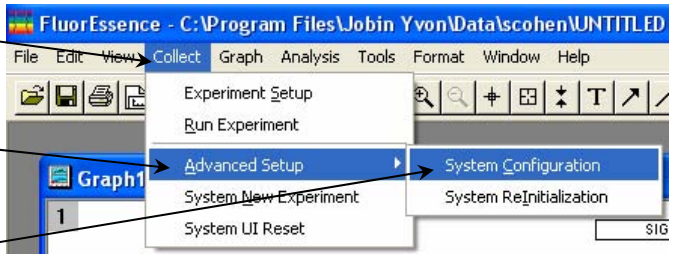
a Start the instrument and computer, according to the instrument's and computer's instruction manuals.

b Click on the FluorEssence™ icon in Windows®.

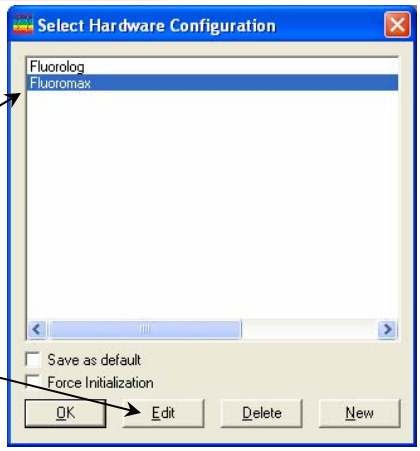
The instrument initializes, then the **FluorEssence** window appears. If there are any difficulties, see the chapter on troubleshooting.

c Choose the Collect menu.

d Choose Advanced Setup, then System Configuration.



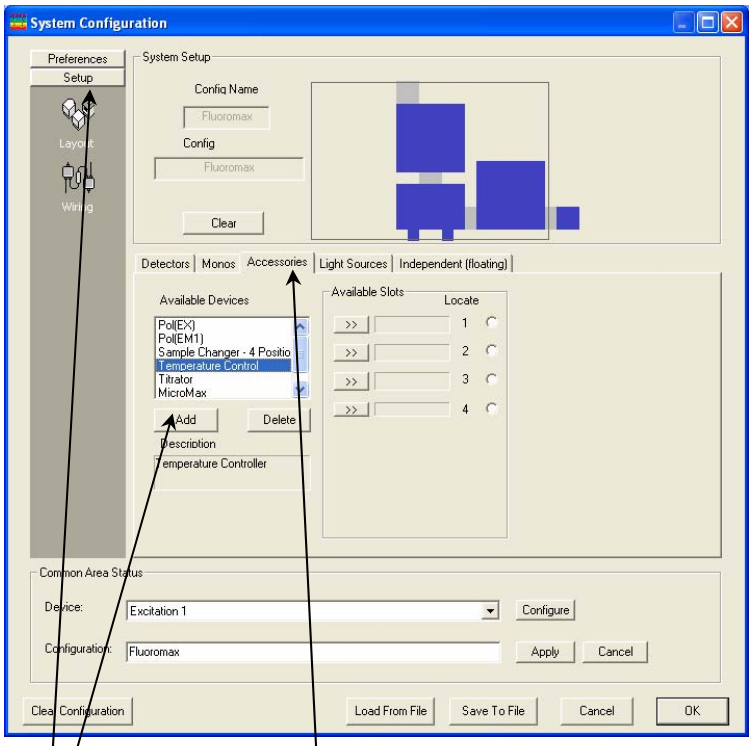
If you haven't chosen a specific instrument configuration, the **Select Hardware Configuration** window appears:



e Choose the desired instrument configuration to which you wish to add the Peltier device.

f Click the Edit button.

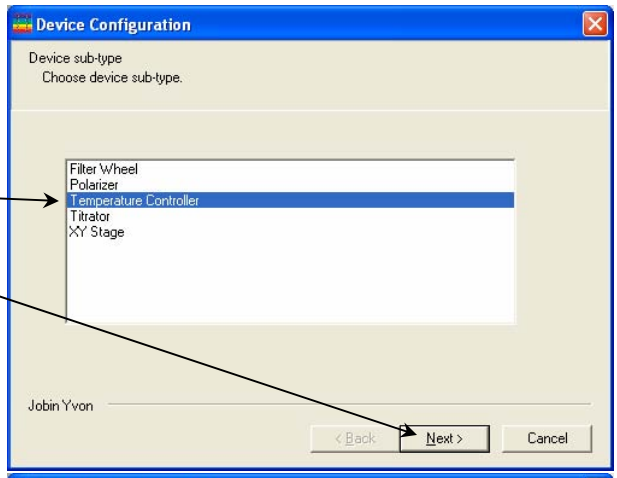
The **System Configuration** window appears.



g Choose the Setup button and the Accessories tab.

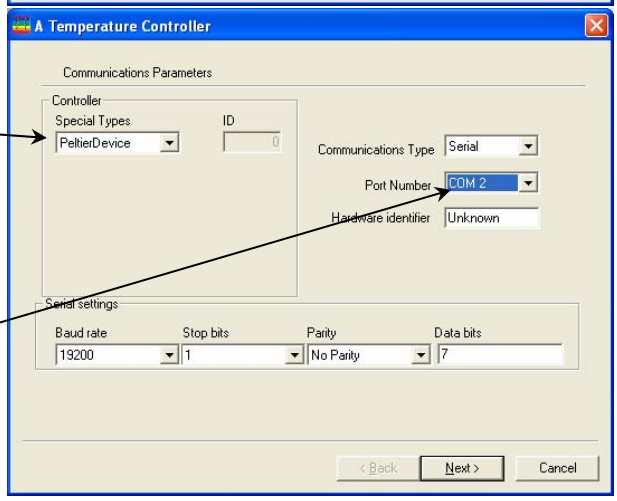
h Choose the Add button.

The **Device Configuration** window appears:



i Choose Temperature Controller, then click the Next > button.

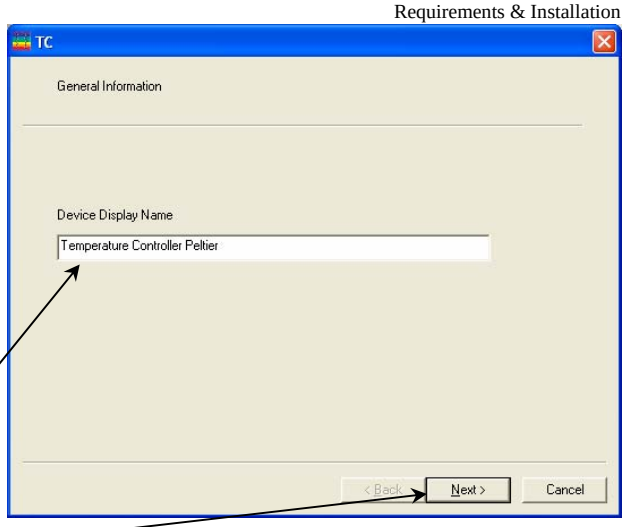
The **A Temperature Controller** window appears:



j From the Special Types drop-down menu, choose PeltierDevice. From the Port Number drop-down menu, choose COM 2.

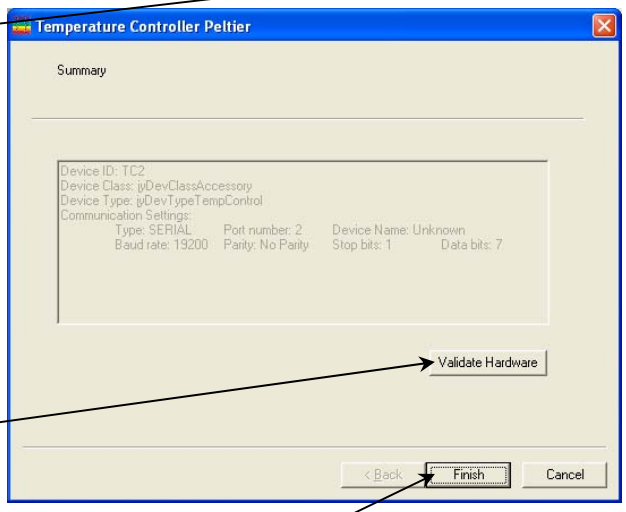
k Usually the instrument is attached to COM 1, so try COM 2 or higher. Leave the other parameters as they are. Click the Next > button.

The **TC** window appears:



l Enter a name, say Temperature Controller Peltier, for the Peltier device, then click the Next > button.

The **Temperature Controller Peltier** (or whatever name you gave the Peltier device) window appears, with a Summary of its parameters:



m If the Peltier Device is actually connected, click the Validate Hardware button.

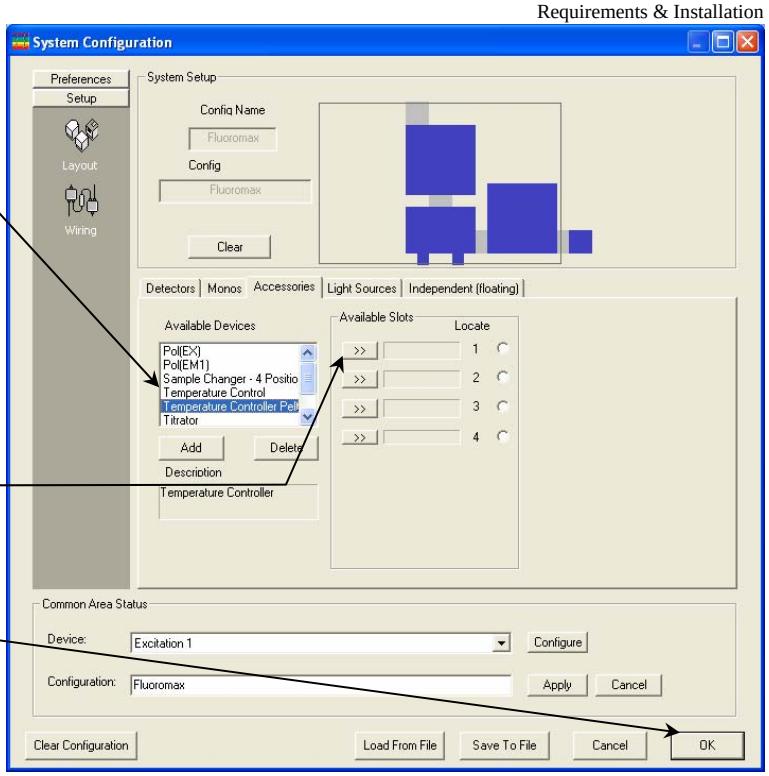
The software attempts to establish communication with the Peltier Device. If the communication attempt fails, an error message appears. Verify that the correct COM port is used and that all cables are firmly attached.

n Click the Finish button.

The window closes, and the Peltier device appears in the Available Devices list:

o Choose the Peltier device. Click the next available >> button to add the Peltier device to the Available Slots list.

p Click the OK button to complete the session.



6 Confirm that the upper and lower temperature limits are set correctly.

a Switch on the controller's POWER button.

b Turn knob to SET T.
This setting adjusts the temperature limits.

c Push the TEMP LIMIT button once.
Starting with the upper temperature limit, the upper and lower temperature limits are displayed alternately.

d Push the TEMP LIMIT button once to release this display.

2: System Operation

Introduction

There are three major items not controlled from the computer:

- Magnetic stirrer
- Nitrogen purge port
- *PID* settings and other advanced parameters pertaining to the Peltier device.

To adjust the advanced Peltier-device parameters

The following parameters are adjusted on the front panel of the control unit:

- maximum and minimum allowable temperatures,
- maximum and minimum allowable electric current to the Peltier device, and
- *PID* settings for rate of temperature change and sensitivity of the control unit to the thermal inertia of the sample holder,

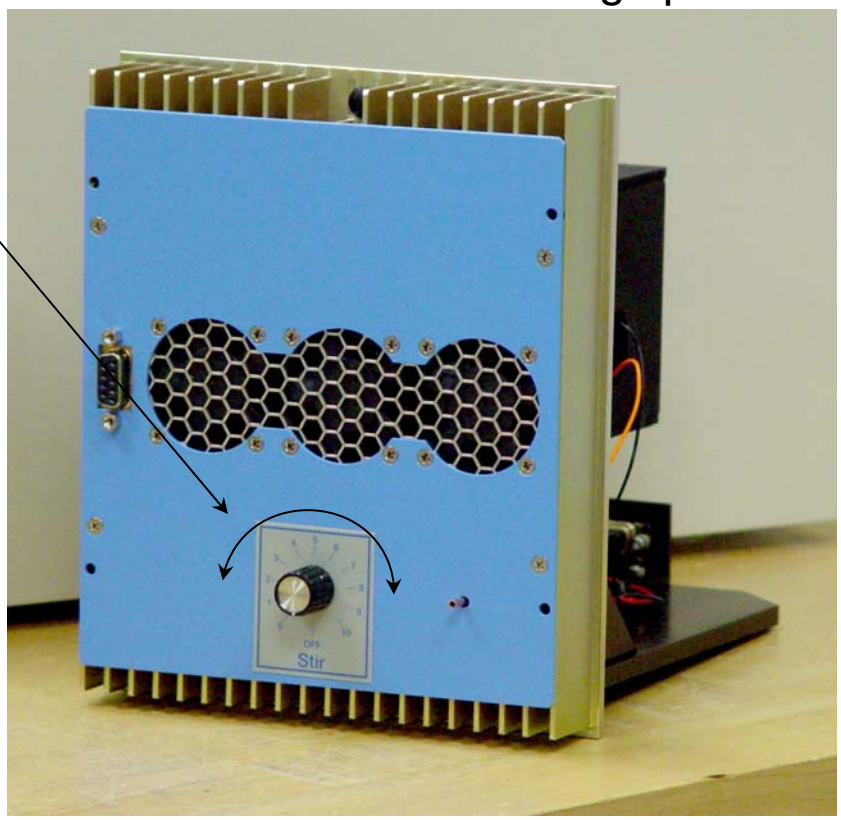
Consult the *Wavelength Elecronics™ User's Guide* for details on how to adjust these parameters.

Note: *HORIBA Jobin Yvon has set the* PID *values for an optimum response from the Peltier device. We do not recommend changing these default values.*

To adjust the magnetic stirrer

1. Place the magnetic stirring bar in the sample cuvette.
2. Place the cover on the Peltier Sample Cooler.
3. Close the lid of the spectrofluorometer's sample compartment.
4. Turn the knob on the front of the Peltier Sample Cooler from OFF to the desired stirring speed.

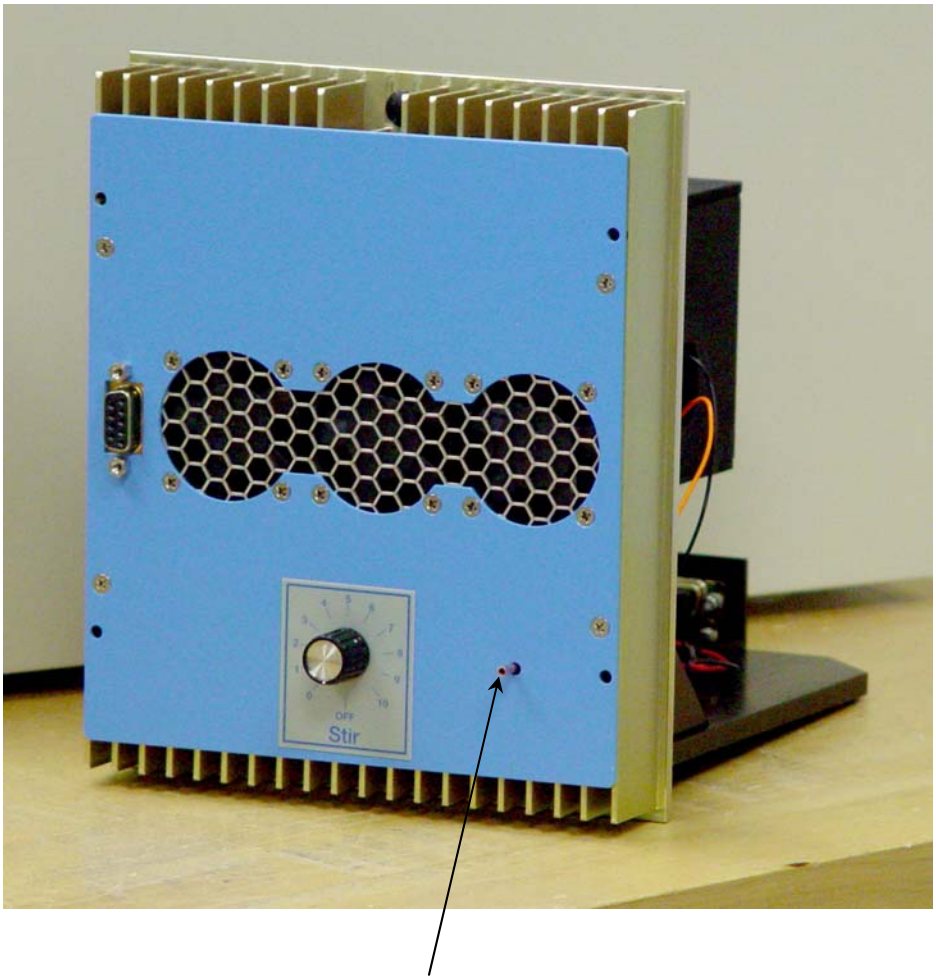
"0" is the slowest speed, and "10" is the fastest speed.



Warning: *The sample, cuvette, and cuvette holder may be hot. There is a risk of burning your fingers if the hot areas are inadvertently touched.*

To purge the sample holder

Purging the sample holder with dry nitrogen gas is usually done when the sample temperature is below the ambient dew point, i.e., in a cold, humid environment. Purging with dry nitrogen prevents water condensation on the cuvette, improving spectroscopic scan quality.



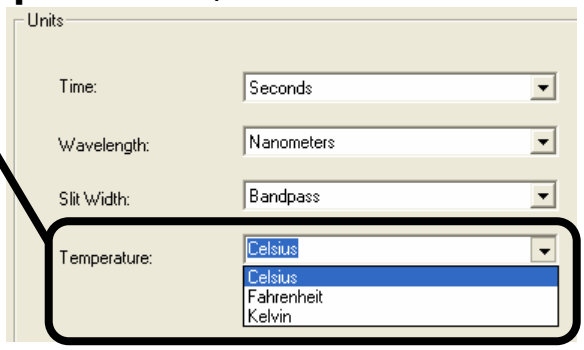
1. Attach the dry-nitrogen gas line to the purging port.
2. Start the gas.

Warning: *Pressurized-gas cylinders pose risks of explosion. Read all Materials Safety Data Sheets (MSDS) that come with the nitrogen gas.*

To adjust the basic Peltier Sample Cooler parameters

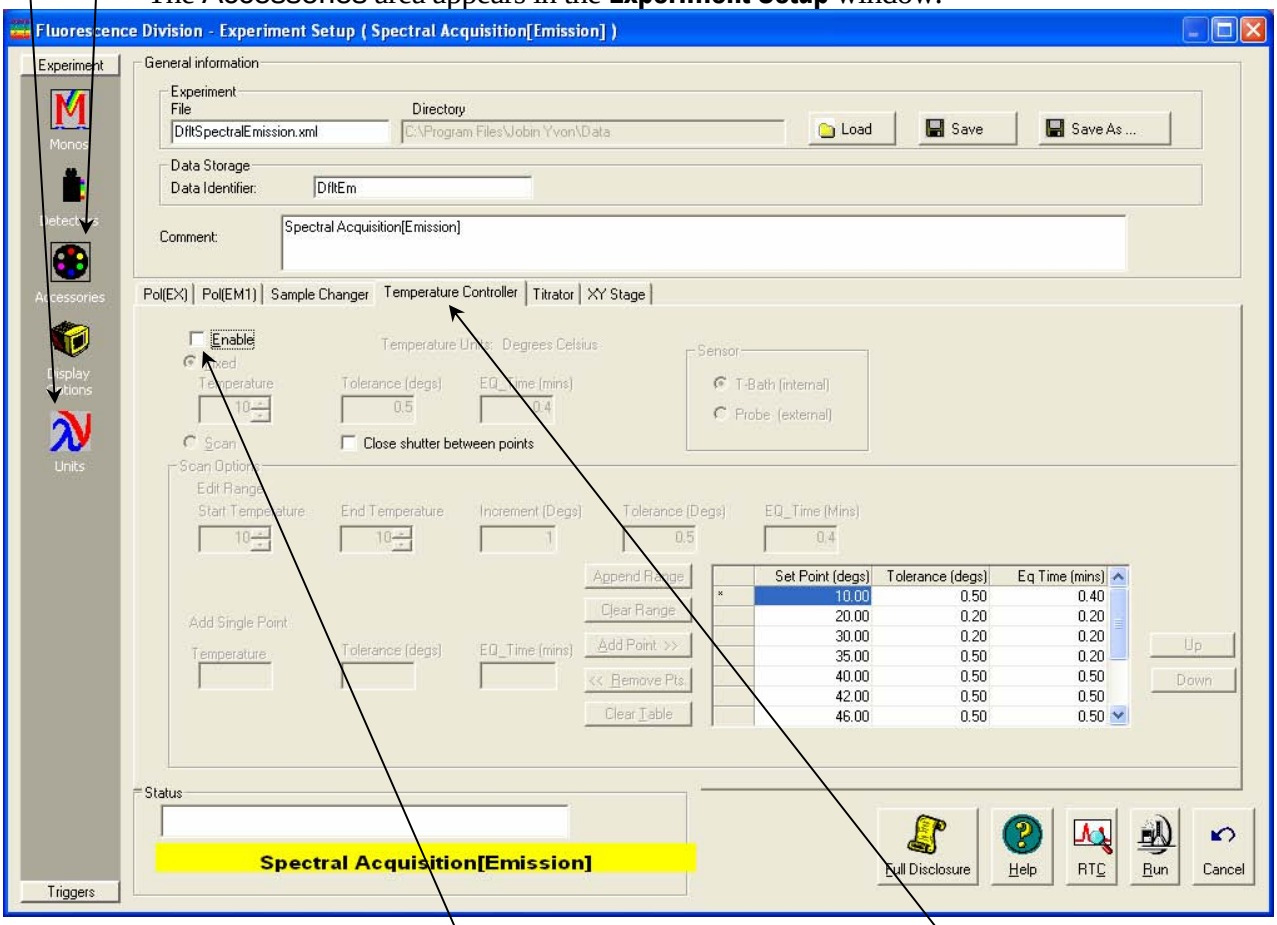
1 In the **Experiment Setup** window, choose the Units icon.

Choose the desired temperature unit (Celsius, Fahrenheit, or Kelvin) from the drop-down Temperature list. The default value is Celsius.



2 Choose the Accessories icon.

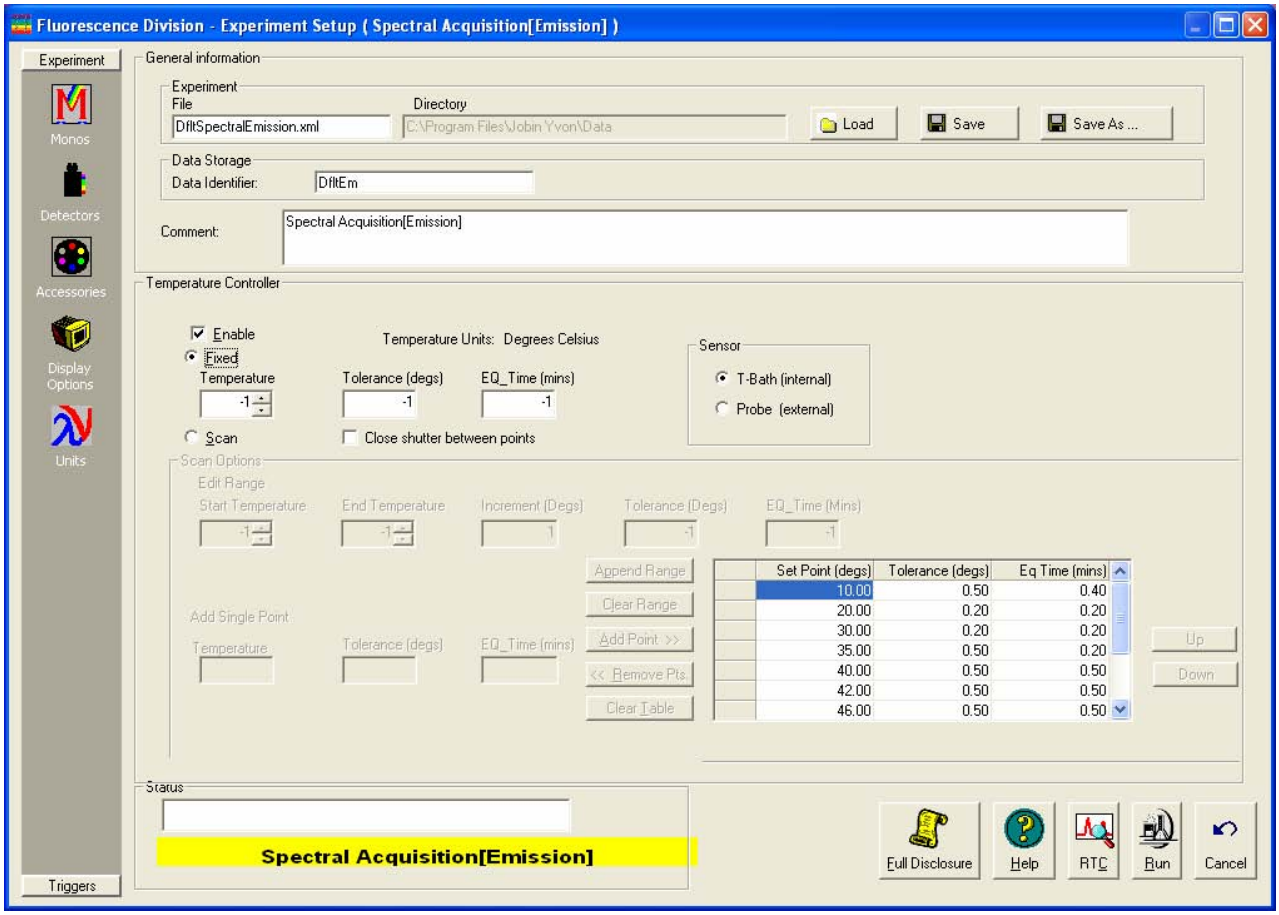
The Accessories area appears in the **Experiment Setup** window.



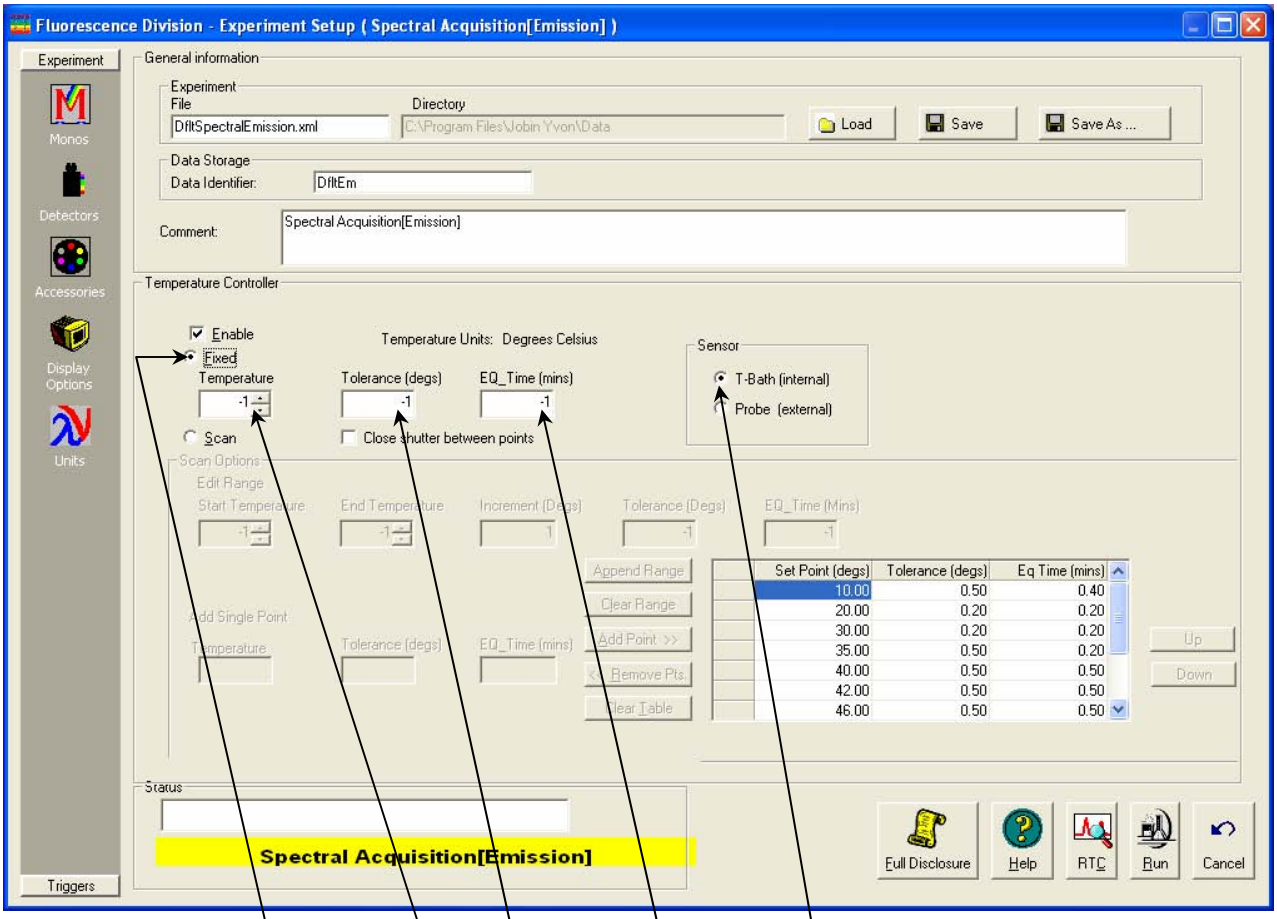
a If you have multiple accessories, click on the appropriate tab to view the Peltier device's parameters.

b Check the Enable checkbox to activate the Peltier device.

Some of the parameters become active, as shown below:


Fluorescence Division - Experiment Setup (Spectral Acquisition[Emission])
Experiment
Monos
Detectors
Accessories
Display Options
Units
Triggers
General information
Experiment
File
DfltSpectralEmission.xml
Directory
C:\Program Files\Jobin Yvon\Data
Load
Save
Save As ...
Data Storage
Data Identifier:
DfltEm
Comment:
Spectral Acquisition[Emission]
Temperature Controller
Enable
Fixed
Temperature
Tolerance (degs)
EQ_Time (mins)
Temperature Units: Degrees Celsius
Sensor
T-Bath (internal)
Probe (external)
Scan
Close shutter between points
Scan Options
Edit Range
Start Temperature
End Temperature
Increment (Degs)
Tolerance (Degs)
EQ_Time (Mins)
Append Range
Clear Range
Add Point >>
<< Remove Pts
Clear Table
Add Single Point
Temperature
Tolerance (degs)
EQ_Time (mins)
Set Point (degs)
Tolerance (degs)
Eq Time (mins)
10.00 0.50 0.40
20.00 0.20 0.20
30.00 0.20 0.20
35.00 0.50 0.20
40.00 0.50 0.50
42.00 0.50 0.50
46.00 0.50 0.50
Up
Down
Status
Spectral Acquisition[Emission]
Full Disclosure
Help
RTC
Run
Cancel

To hold the sample's temperature constant:



1. Be sure the Fixed radio button is active.
2. Enter the desired sample temperature in the Temperature field.
3. Enter the Tolerance (in degrees).

   The Tolerance controls how widely the temperature may vary around the fixed sample-temperature.
4. Enter the equilibration time (in minutes) in the EQ_Time field.

   The equilibration time controls how fast the Peltier device reaches the fixed temperature. Fractions of minutes are allowed.
5. Be sure the T-Bath (internal) radio button is active in the Sensor area.

Note: *Values are updated approximately every 5 seconds.*

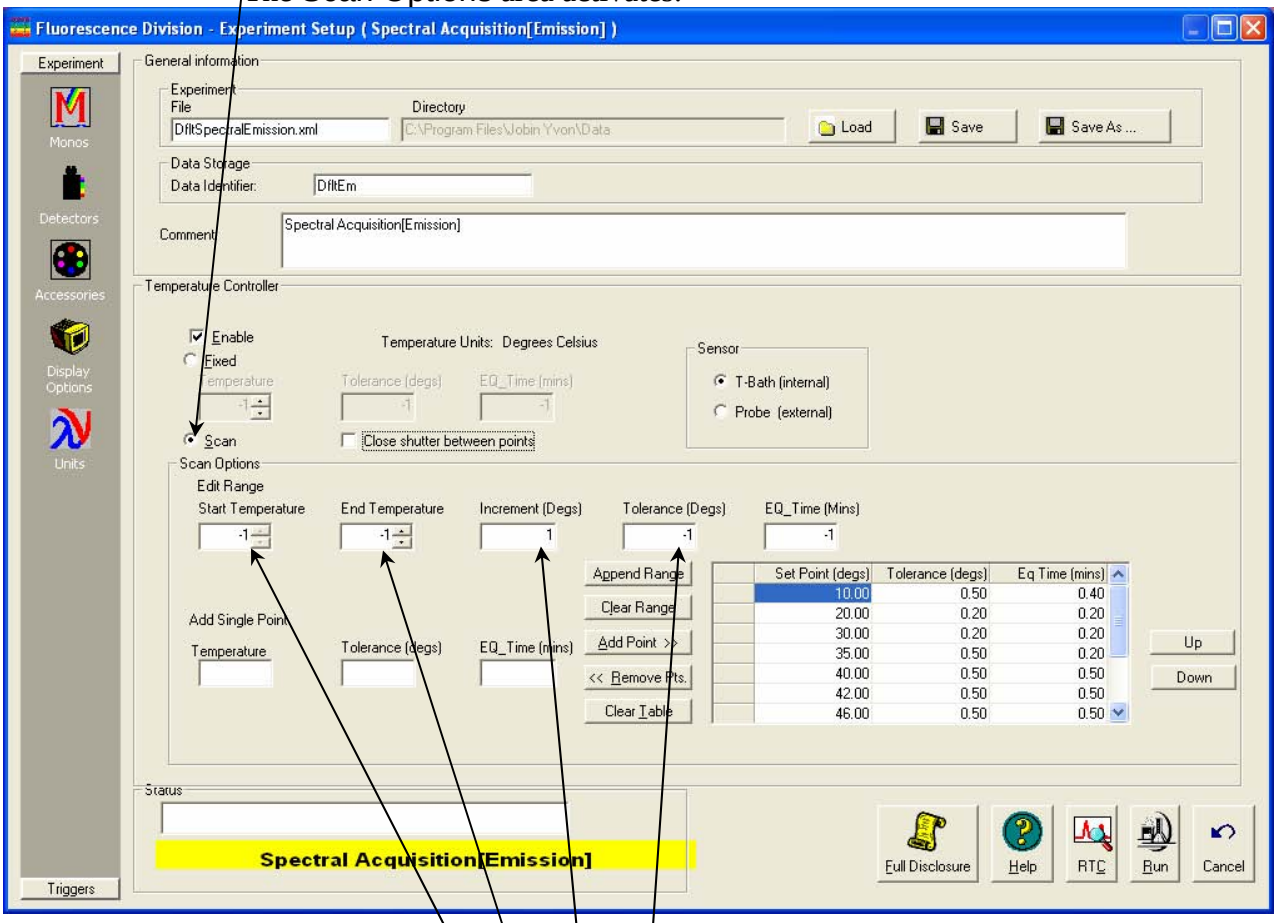
The Peltier device does not use the Probe (external) sensor.

6 If you want the automatic shutter to close between recording data points, check the Close shutter between points checkbox.

To scan the sample through a temperature range:

1 Click the Scan radio button.

The Scan Options area activates.



2 Enter the desired temperature range.

a Enter the Start Temperature.

b Enter the End Temperature.

c Enter the Increment in degrees.

The Increment controls the temperature difference between the steps of the range.

d Enter the Tolerance in degrees.

The Tolerance controls how widely the temperature may vary around the fixed sample-temperature.

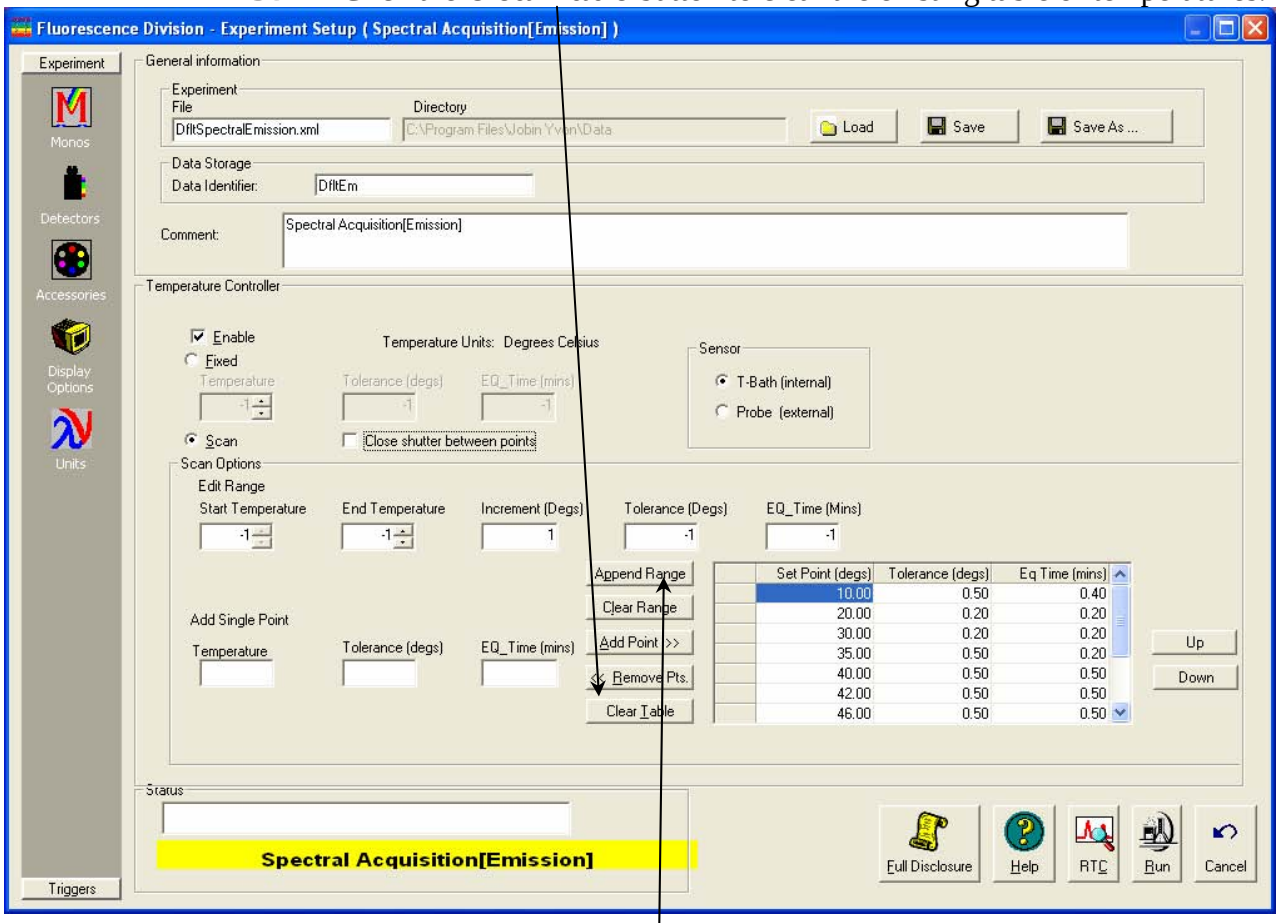
e Enter the equilibration time (in minutes) in the EQ_Time field.

Note: *Values are up-dated approximately every 5 seconds.*

The equilibration time controls how fast the Peltier device reaches the fixed temperature. Fractions of minutes are allowed.

3 Create a table of the desired temperatures.

a Click the Clear Table button to clear the existing table of temperatures.

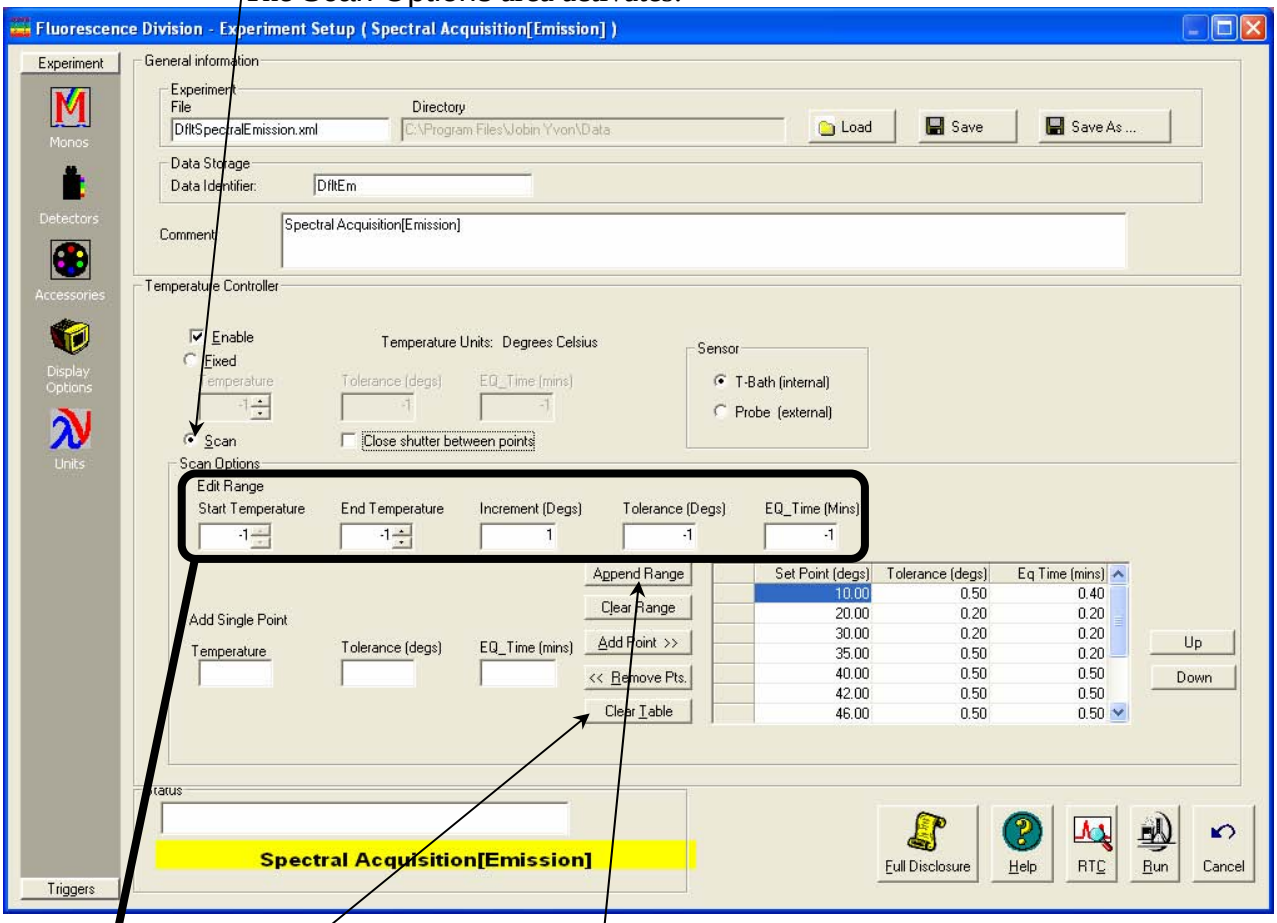


b Click the Append Range button to place the new temperatures in the table.

To set up a custom series of temperatures:

1 Click the Scan radio button.

The Scan Options area activates.



2 Click the Clear Table button to remove the existing entries in the table.

Note: *Values are updated approximately every 5 seconds.*

3 Create a new temperature sequence by editing the Start Temperature, End Temperature, Increment, Tolerance, and EQ_Time (equilibration time) fields.

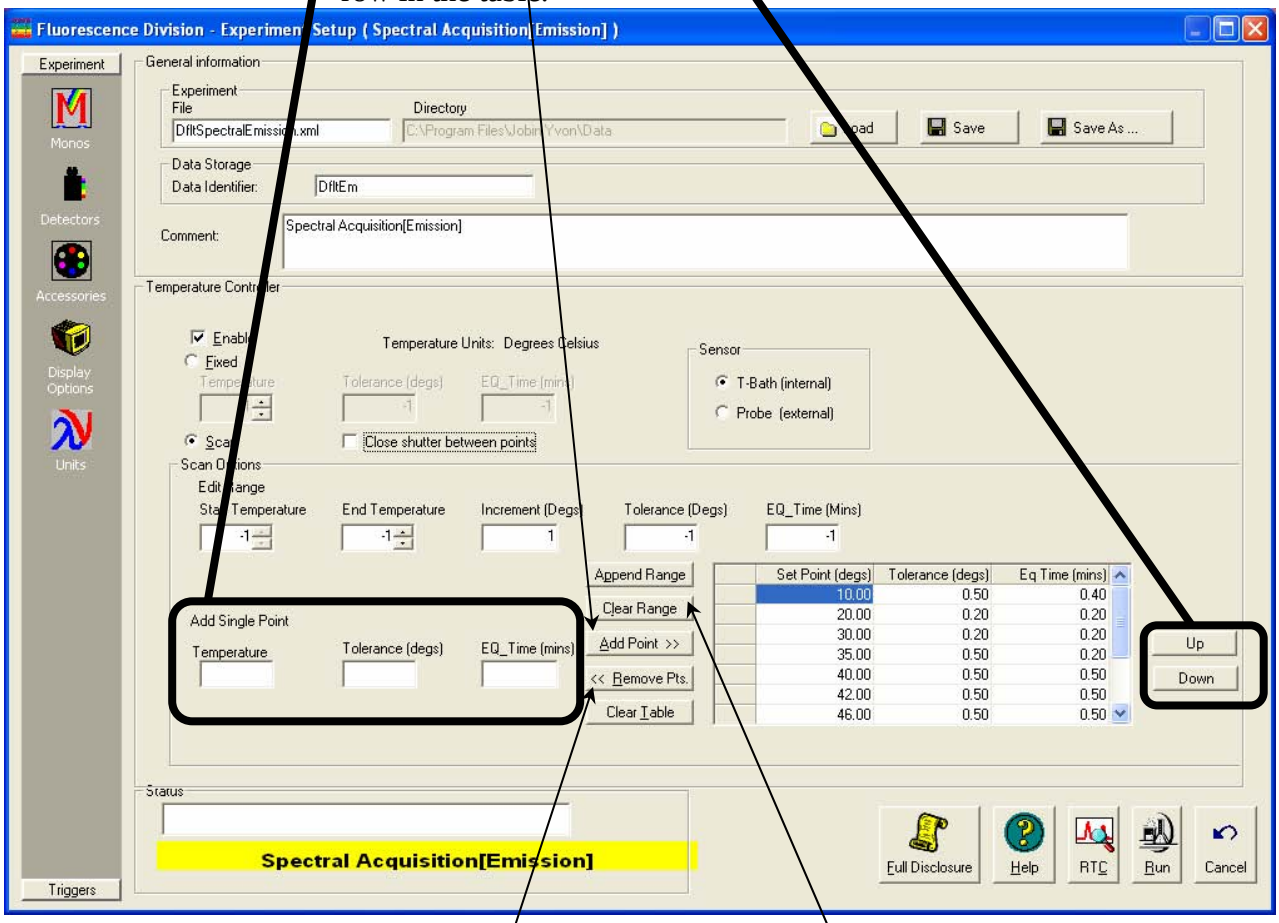
4 Click the Append Range button to place this sequence in the table.

5 Edit the table as necessary:

a Add a single point, if desired, by entering a particular Temperature, Tolerance, and EQ_Time (equilibration time).

b Click the Add Point >> button to add this single point to the table.

c Click the Up button and Down button to move the point to the desired row in the table.

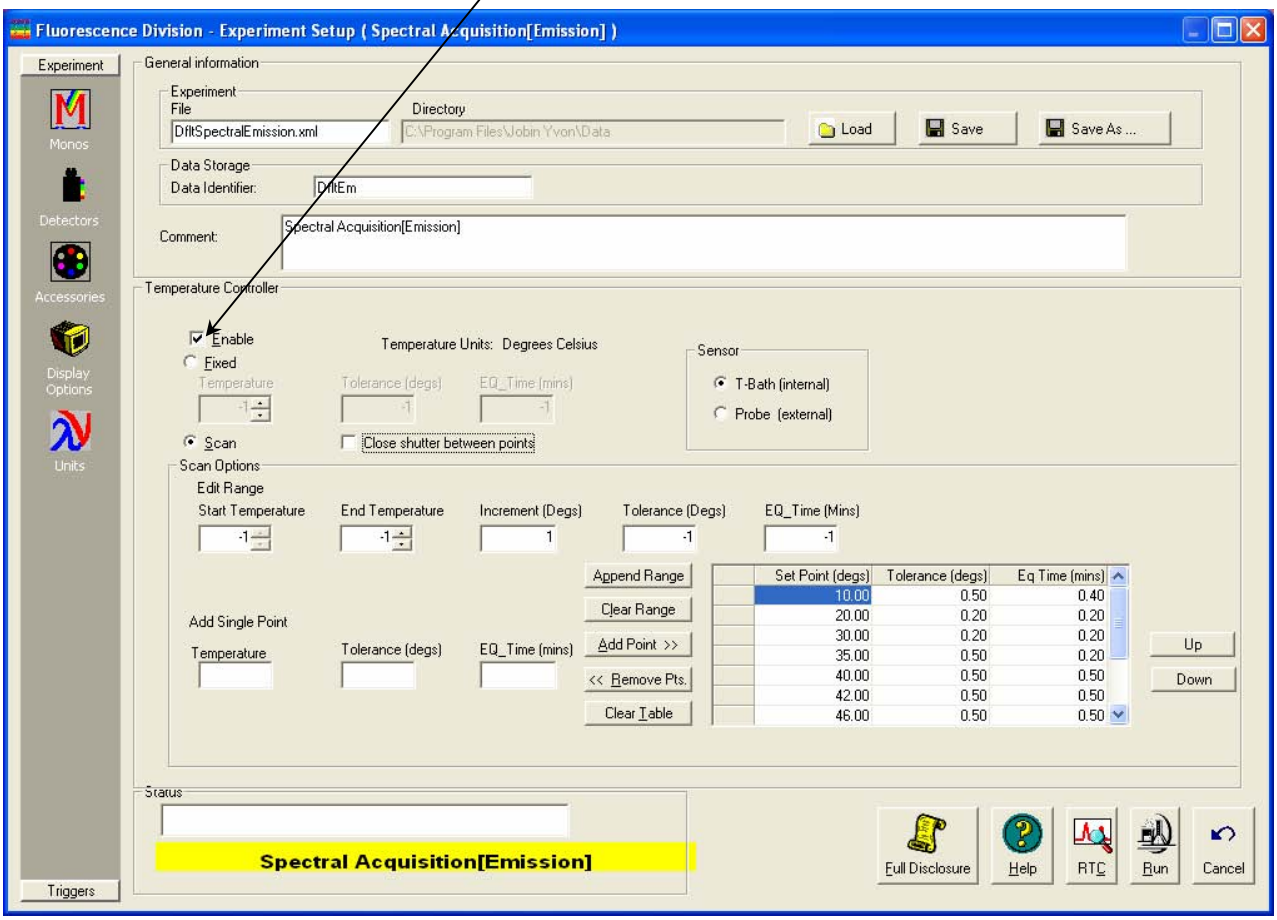


d To remove a row from the table, select the row and click the << Remove Pts. button.

e To remove a large section of the table, select the section and click the Clear Range button.

To start or stop the heating or cooling cycles

1 Choose the Enable checkbox to start the Peltier device.



2 Uncheck the Enable checkbox to stop the Peltier device.

Note: *If the Enable button does not engage and start operation of the Peltier, check connections and settings to the Peltier device.*

About *PID* parameters

P = Proportional Gain

The "Error Voltage" is the difference between set-point voltage (the desired temperature) and measured thermocouple voltage (actual temperature). The proportional gain, *P*, supplies a current proportional to the Error Voltage. If *P* is large, the Peltier device approaches the desired temperature rapidly. This, however, may overshoot the desired temperature. As the Error Voltage drops to 0 when the actual temperature reaches the set-point, so does *P*, cutting off heating or cooling. Thus over- and undershoots of the set-point may happen without adjusting the next two parameters listed below.

I = Integrator

The integrator charges up based on how close the Error Voltage is to 0. Rapid charging (small integrator time-constant, or Integrator Time Const.) occurs if the Error Voltage is large; slow charging (large time-constant) occurs if the Error Voltage is small. At Error Voltage = 0, no charging occurs, and the output current is run via the integrator. Try to keep the time-constant large enough to compensate for the thermal inertia of the sample holder and Peltier device, otherwise the temperature may oscillate.

D = Differentiator

The Differentiator (or Differentiator Time Const.) uses the change in Error Voltage with time, d*V*/d*t* (the derivative). The faster the Error Voltage changes (the larger the slope of d*V*/d*t*), the larger the "opposing" current. Thus, the Differentiator opposes radical changes in the sample's temperature.

Note: *HORIBA Jobin Yvon has set the* PID *values for an optimum response from the Peltier device. We do not recommend changing these default values. Consult the* Wavelength Electronics™ User's Guide *for details on adjusting PID parameters.*

To run the Peltier device

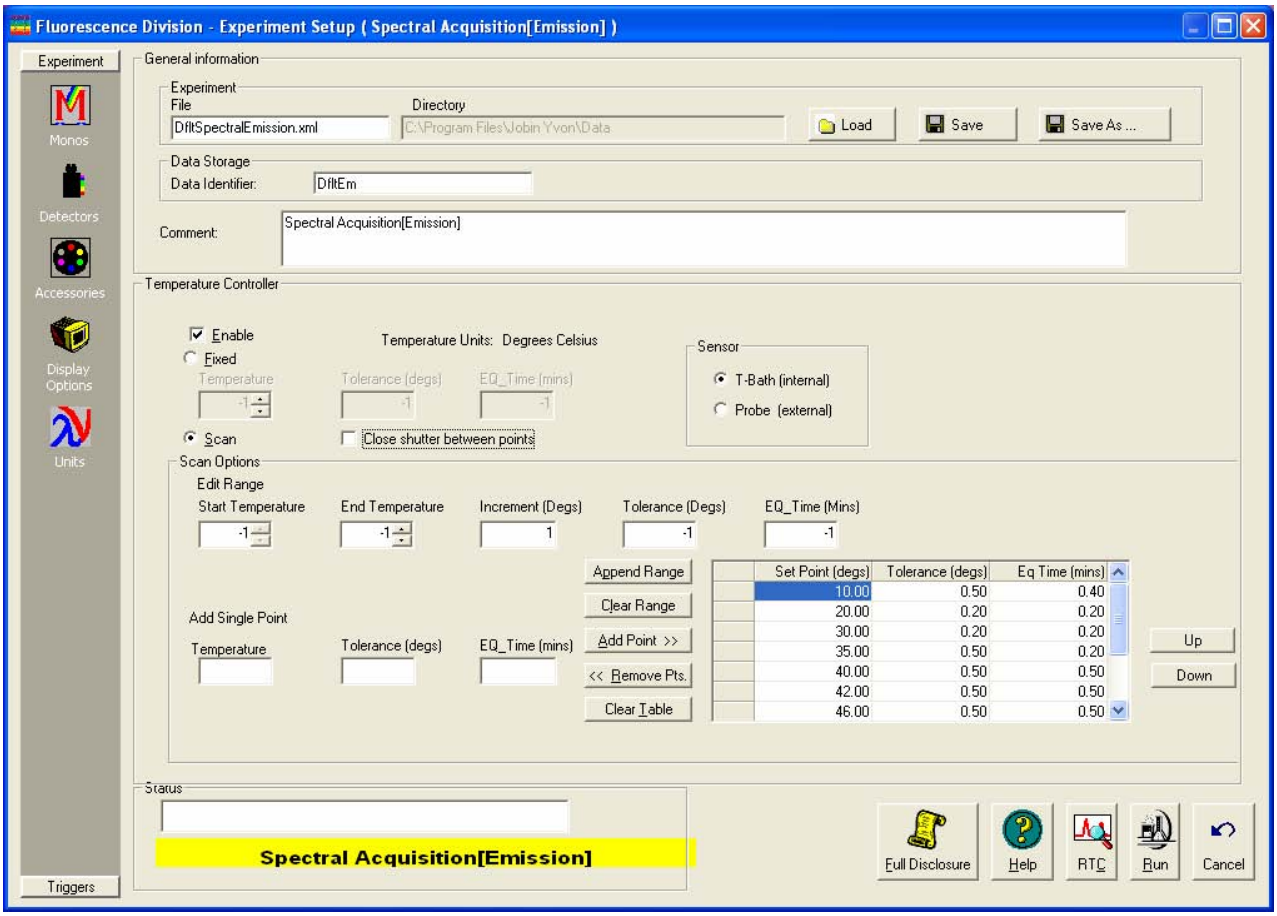
Real time control (RTC)

1 Set up the Peltier device.

a Load a configuration that includes the Peltier device.

b Adjust all parameters for the Peltier device in the **Experiment Setup** window.

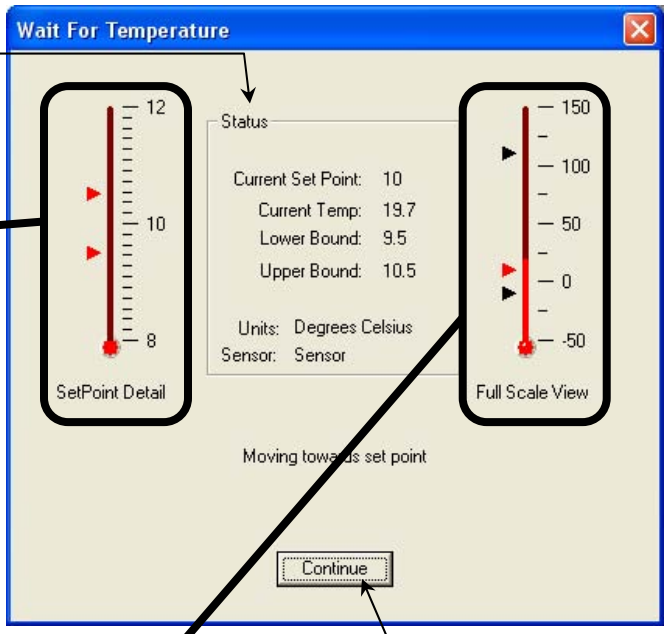
c Click the RTC button.



If the Peltier device has not yet reached its set-point, the **Wait For Temperature** window appears:

The status of the Peltier device appears in the Status area.

A close-up view of current set-points appears on the SetPoint Detail thermometer. The Lower Bound is the lower triangle; the Upper Bound is the upper triangle.



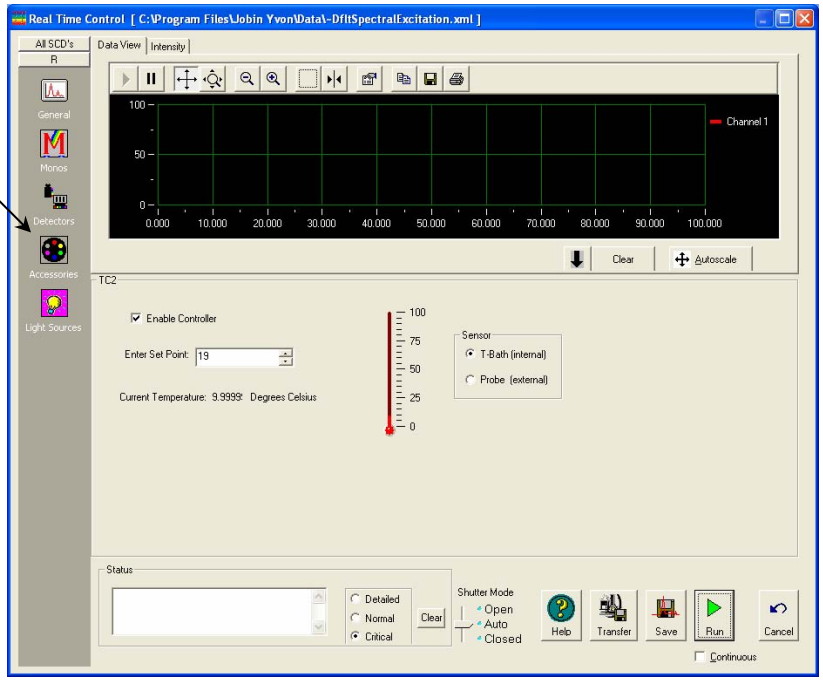
Note: *The Upper and Lower Bounds are half the Tolerance above and below the "Temperature" set-point.*

A full-range thermometer appears in the Full Scale View. Maximum and minimum possible set-points are black triangles.

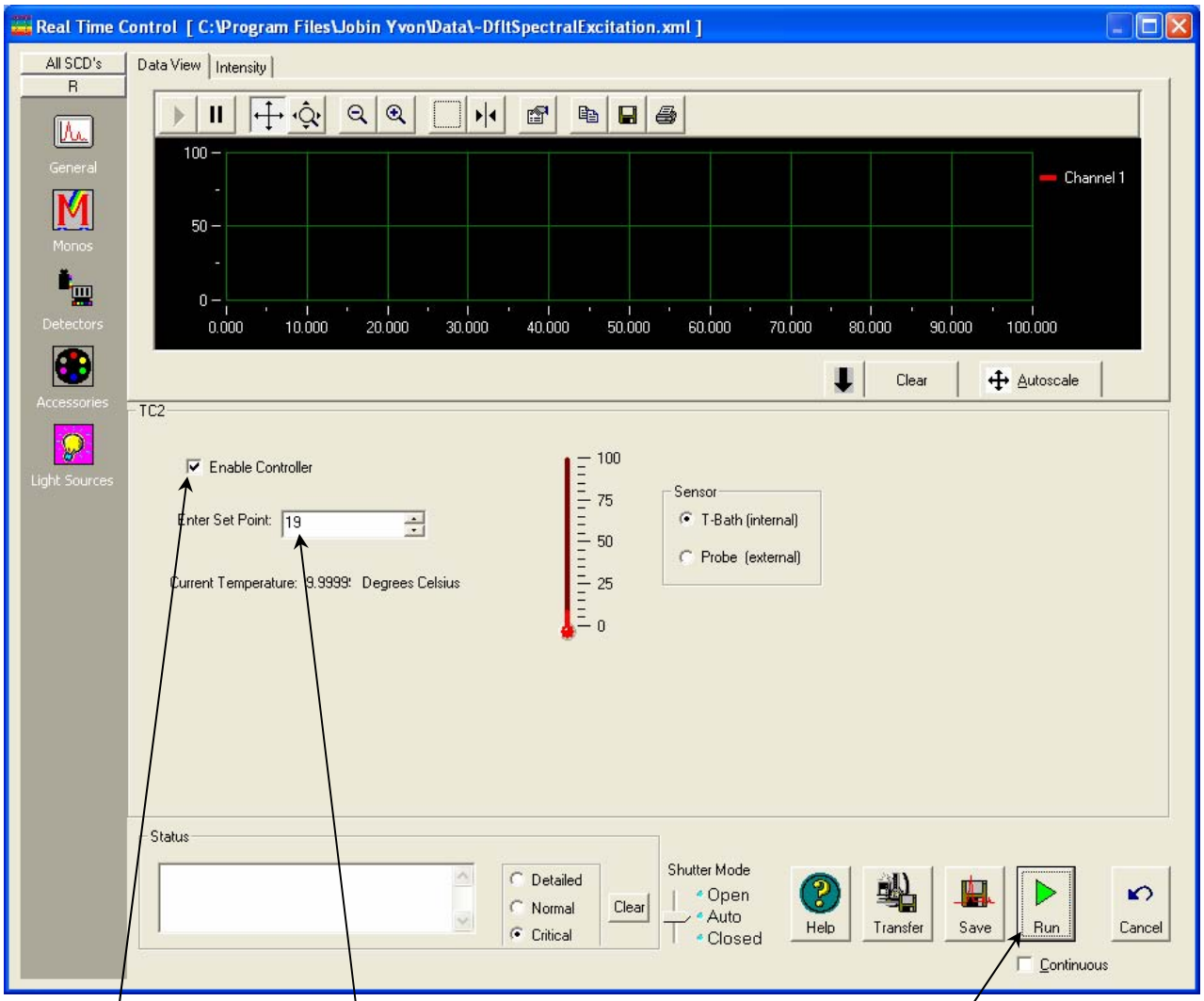
2 Click the Continue button.

The **Real Time Control** window opens.

3 Click the Accessories icon to view the Peltier device's parameters.



4 Modify the Peltier devices parameters as desired, while viewing the real-time data.



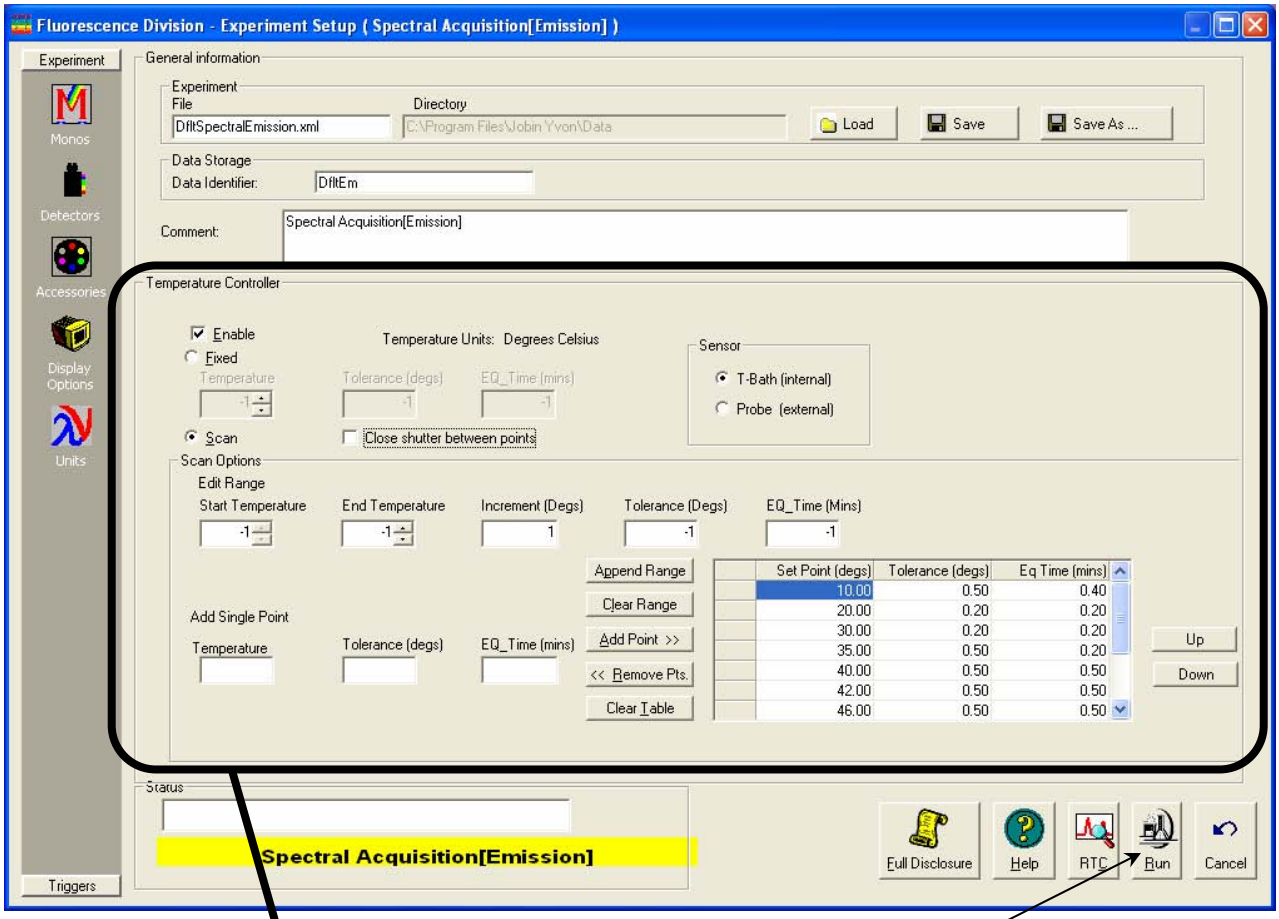
a Click the Enable Controller checkbox to activate the Peltier device.

b Change the set point in the Enter Set Point field.

The present temperature is given digitally and graphically.

5 Click Run to run an experiment.

Normal Experiment Setup



1 Set up all parameters as discussed earlier in this chapter.

2 Click the Run button.

The experiment using the Peltier device starts.

3: Troubleshooting

Consult the *Wavelength Electronics™ User's Guide* for any problems that may occur with the control unit. Below are listed some problems that might occur:

Problem	Possible Cause	Suggested Remedies
Control unit does not turn on.	Disconnected power cord	Connect power cord.
	Faulty wall receptacle	Check wall receptacle.
	Fuse is blown	Check and replace fuse.
Peltier Sample Cooler cannot connect to controller (communications error).	Faulty communications cable	Verify that you are using the correct cable as listed on the Bill of Materials.
	Improperly connected cable	Verify that the cables are firmly connected to their sockets.
	Defective COM port	Call SPEX® Fluorescence Service Department.
	COM port settings are corrupted	Call SPEX® Fluorescence Service Department.
Peltier Sample Cooler does not operate.	No Peltier accessory is available	Select configuration with Peltier in the **System Configuration** window.
	Communications cables have bad connection	Check communications cables.
		Check connection between spectrofluorometer and Peltier Sample Cooler inside sample compartment.
		Check all software settings.

Further assistance...

Read all software and accessory manuals before contacting the Spex® Fluorescence Service Department. Often the manuals show the problem's cause and a method of solution. Technical support is available for both hardware and software troubleshooting. Before contacting the service department, however, complete the following steps.

1. If this is the first time the problem has occurred, try turning off the system and accessories. After a cool-down period, turn everything back on.

2. Make sure all accessories are properly configured, and turned on as needed.

3. Following the instructions in *System Operation*, run a xenon-lamp scan to make sure the system is properly calibrated. Print the spectrum for each and note the peak intensities.

4. Check this chapter to see if the problem is discussed.

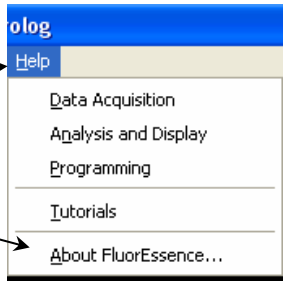
5. Visit our web site at www.jobinyvon.com/fluor/fluor.htm to see if the question is addressed in the **Systems** or **FAQs** sections of the site.

6. Try to duplicate the problem and write down the steps required to do so. The service engineers will try to do the same with a test system. Depending on the the problem, a service visit may not be required.

7. If an error message appears in FluorEssence™, write down the exact error displayed.

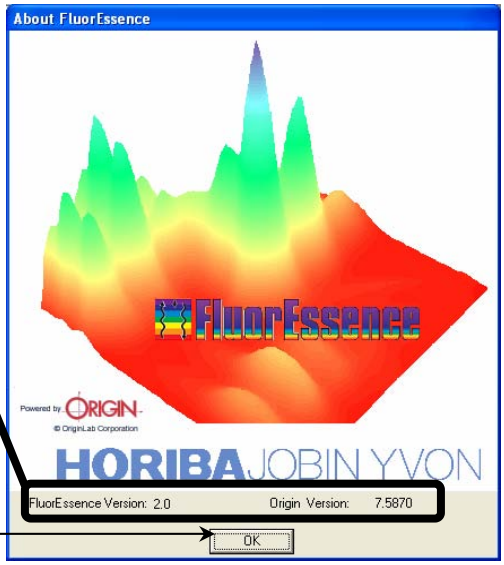
8. Determine FluorEssence™'s version number.

   Choose the Help menu.
   Choose About FluorEssence....



The **About FluorEssence** window opens. Near the bottom are the FluorEssence™ and Origin® version numbers.

Click OK to close the window.



9 Write down the software's version numbers, along with the purchase dates, model numbers, system configuration, and serial numbers of the instrument and its accessories.

10 Call the Spex® Fluorescence Service Department at (732) 494-8660 × 160.

Be prepared to describe the malfunction and the attempts, if any, to correct it. Have serial and version numbers of all software and equipment handy, along with all relevant spectra (sample, polarization ratio, xenon-lamp scan, emission calibration, etc.).

4: Specifications

Computer requirements

- 973005 or equivalent PC computer with RS-232 port

Software requirements

- Windows® XP
- FluorEssence™ 2.0 or higher

Operating specifications

- Ambient environment for controller: 0–55°C
- Minimum Peltier temperature for samples = –10°C
- Maximum Peltier temperature for samples = +110°C
- Display update to software ~ 0.2 Hz (every 5 s)

Electrical and physical specifications

- 115 VAC or 230 VAC ± 15% (switch selectable), 60 or 50 Hz
- 25 W average with output off; 55 W average with output on
- $^{3}\!/_{32}$″ (2.4 mm) O.D. inlet for dry-nitrogen purging
- Controller dimensions are 4¼″ (10.8 cm) W × 7″ (17.8 cm) H × $12^{11}\!/_{16}$″ (32.2 cm) D (allow 2¾″ [7 cm] extra clearance depth for cables)
- Peltier Sample Cooler dimensions are 6¼″ (15.9 cm) W × 7½″ (19.1 cm) H × 9″ (22.9 cm) D

5: Index

U

V

W

HORIBA
Scientific

3880 Park Avenue, Edison, New Jersey 08820-3012, USA
www.horiba.com/scientific

[Design Concept]

The HORIBA Group application images are collaged in the overall design.
Beginning from a nano size element, the scale of the story develops all the way to the Earth with a gentle flow of the water.