HORIBA Scientific

Peltier Sample Cooler
F-3004

Operation Manual
Part number J81049 rev. D

F-3004 Peltier Sample Cooler

Operation Manual rev. D

www.HORIBA.com/scientific

JOBIN YVON Technology

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Part Number J81049

Table of Contents

1: 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 HORIBA Scientific 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.

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- 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 Instruments Incorporated, 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 Instruments Incorporated 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 Instruments Incorporated's liability for the products.

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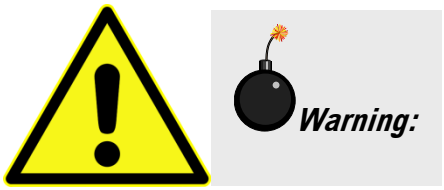
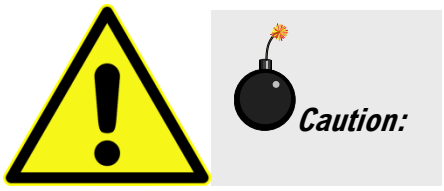
Safety summary

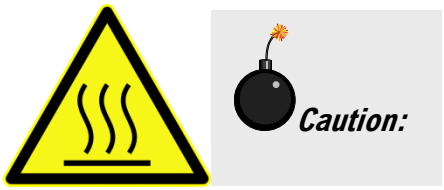
The following general safety precautions must be observed during all phases of operation of this instrument. Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture and intended use of instrument. HORIBA Instruments Incorporated 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:



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 Instruments Incorporated is not responsible for damage arising out of improper use of the equipment.



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 Instruments Incorporated is not responsible for damage arising out of improper use of the equipment.



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.

General information is given concerning operation of the equipment.

2: Requirements & Installation

Requirements

Computer requirements

- USB port
- FluorEssence™ software
- Windows® XP or higher
- J973005 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	J401233
1	Sample-cooler cover	
1	Controller Unit	J990116
1	Peltier Sample Cooler Operation manual	J81049
1	Power cord (115 V or 220 V)	J98015 or J98020
4	#4-40 × 1½" screw for FluoroMax® installation	J600105
4	#4-40 × 1¾" screw for Fluorolog® installation	J600104
1	Magnetic stirring bar	J53044
1	Certificate of Calibration for control unit	
1	9-to-15-pin D-sub Peltier Control cable	J401223
1	6′ USB cable	J980033

Caution: *The Peltier Sample Cooler is a delicate instrument with sensitive components. Mishandling may seriously damage the system.*

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 Instruments Incorporated and the shipper at once.

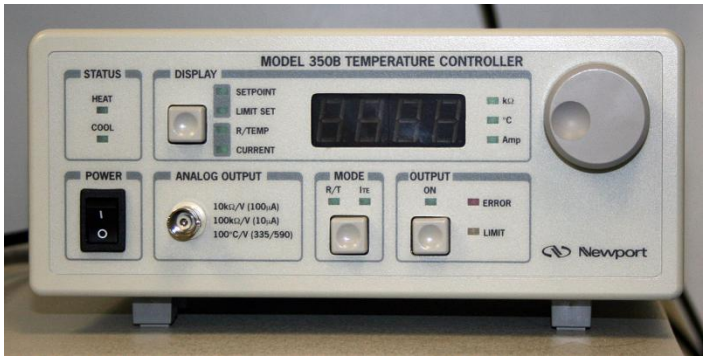
Note: *Many public carriers do not recognize claims for concealed damage reported later than 15 days after delivery. For a shipping damage claim, inspection by the carrier agent is required. Therefore, the original packing material should be kept as evidence. While HORIBA Instruments Incorporated is not responsible 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:

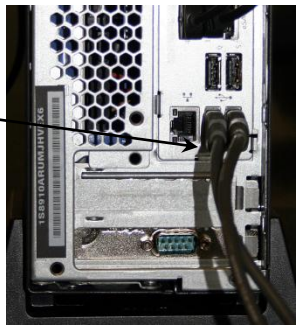
a Remove the existing sample holder from the sample compartment of the spectrofluorometer.

b Install the control unit as directed in the *Newport Temperature Controller Model 3005 Series User's Manual* (Chapter 3, "Getting Started").

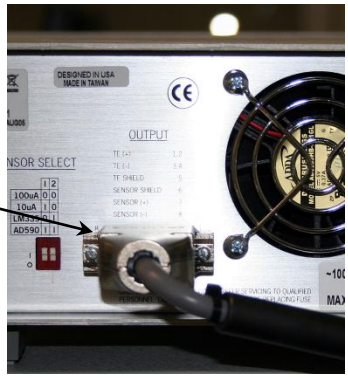


c Connect the USB cable to the back of the control unit.

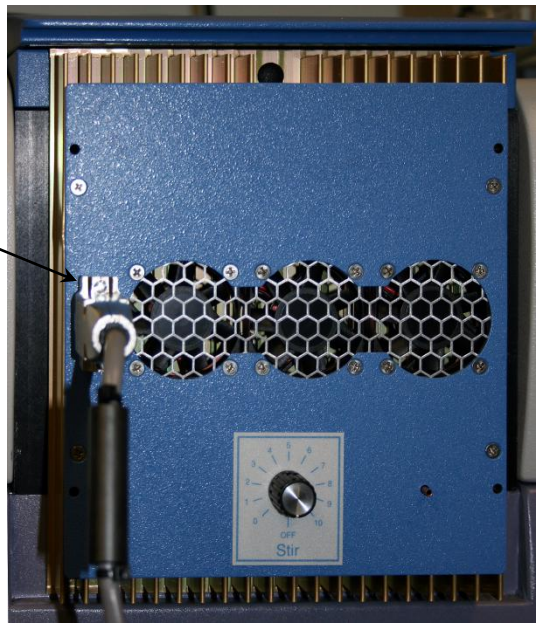
d Attach the USB cable to the USB port on the host computer.



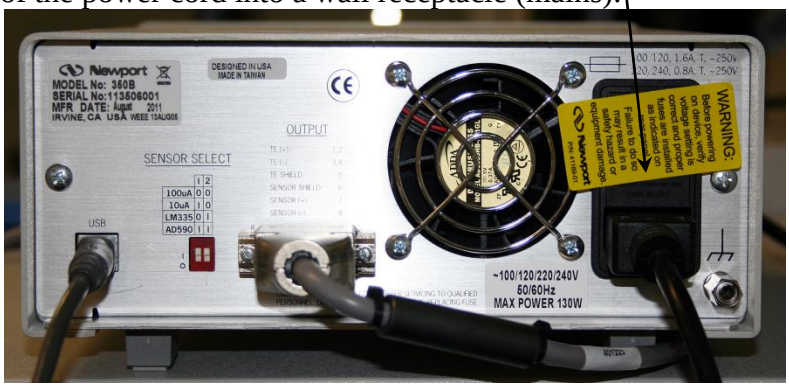
e Connect the 15-pin end of the control cable to the OUTPUT jack on the back of the control unit.



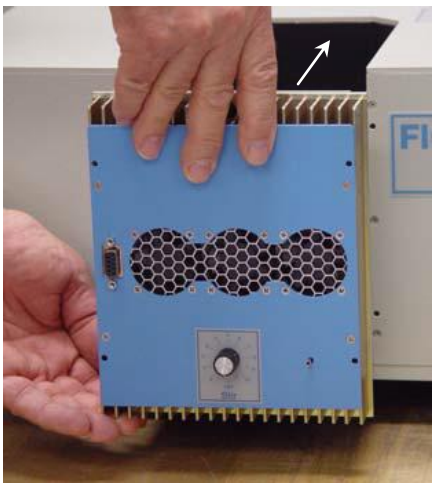
f Attach the 9-pin end of the control cable to the jack on the front of the Peltier Sample Cooler.



g Insert the power cord into the jack on the control unit. Plug the other end of the power cord into a wall receptacle (mains).

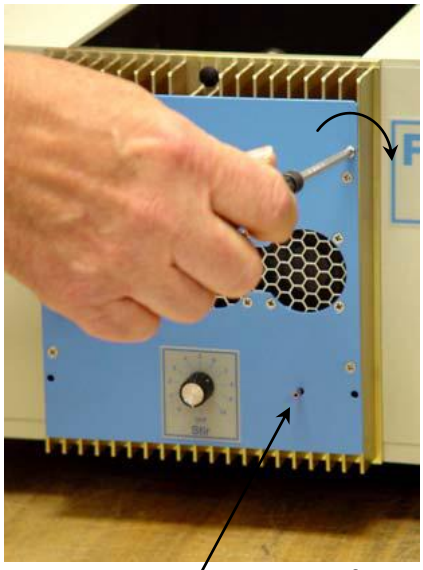


h Place the Peltier Sample Cooler into the sample compartment.



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

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



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

j (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.

Note: *Do not switch on the Peltier control unit until directed to do so.*

4 Install the software on the host computer.

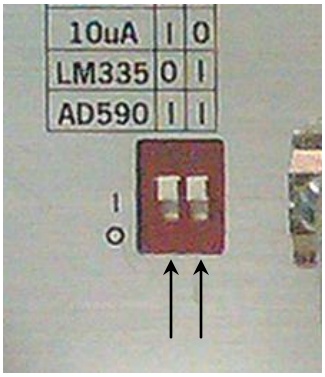
(only if the Peltier Sample Cooler was purchased separately from the spectrofluorometer)

a Install the Newport software on the host computer.
Accept all defaults. Click Install this driver anyway during the driver portion of installation.

b If your existing FluorEssence™ software is lower than 3.5.1.99, apply the software patch `HJYApplicationUpgrade 3.5.1.99`.
The software patch should be on a disk included with the accessory.

c Restart the host computer.

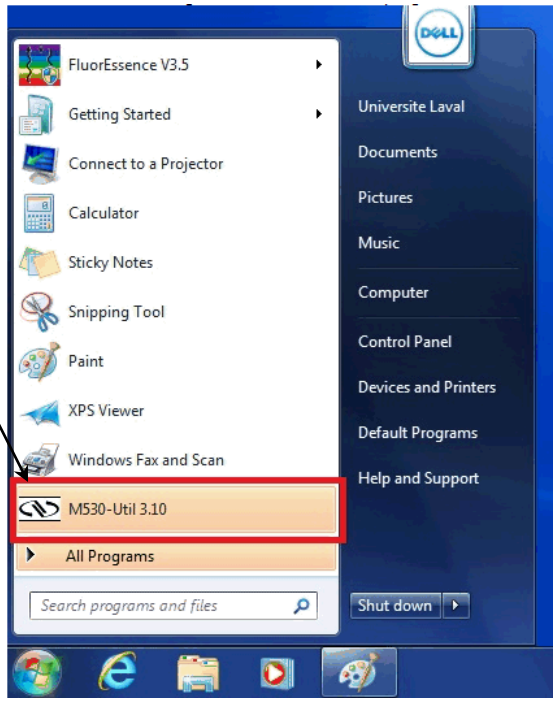
5 Set the DIP switches on the back of the Peltier controller to 1-1 (both switches upward).



6 On front panel, turn on Peltier control unit.

7 Using the Newport utility, set the *PID* settings:

a In the Windows® Start menu, select M530-Util from All Programs. Be sure to run the program with administrator privileges (right-click Run As Administrator).



The **Newport LDD/TEC** window opens:

b Select the TEC - TEC Controller tab.

c Change the USB Address field to `2`.

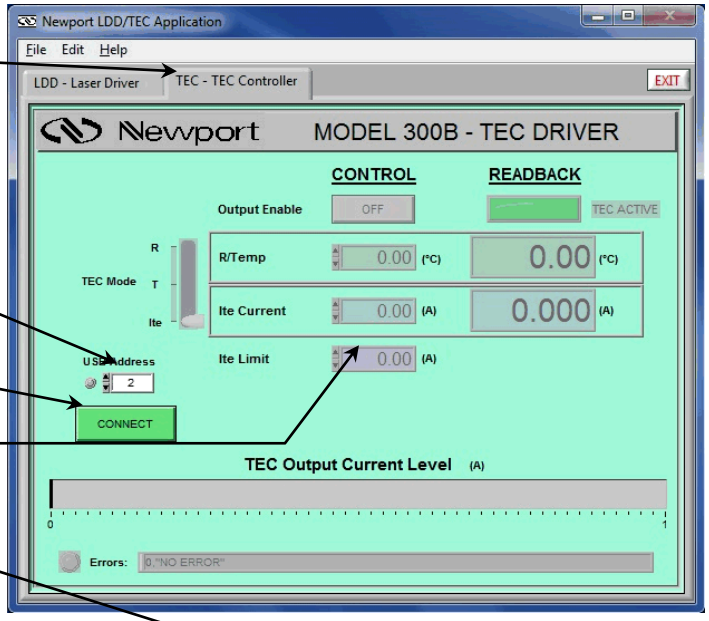
d Click the Connect button.

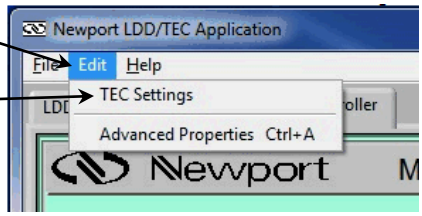
e Set the Ite Limit field to `5.00`.

f Choose Edit.

g A drop-down menu appears.

h Select TEC Settings.





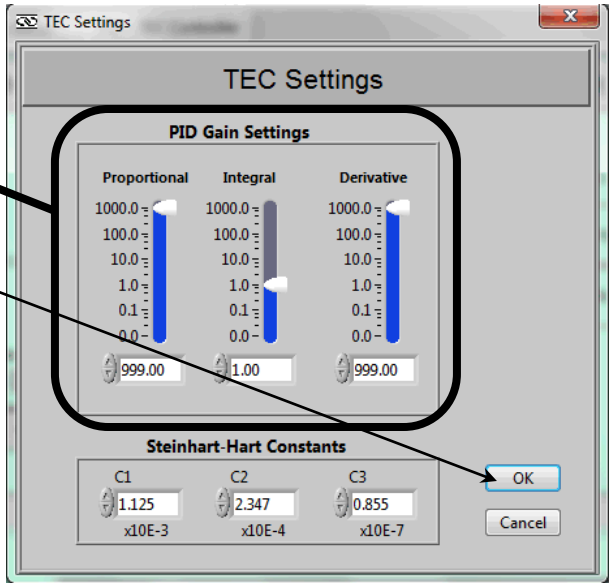
The **TEC Settings** window appears:

i Set the PID Gain Settings area as shown: Proportional = 999, Integral = 0, Derivative = 999.

j Click the OK button. The **TEC Settings** window closes.

k Click the Exit button to close the Newport Utility program.

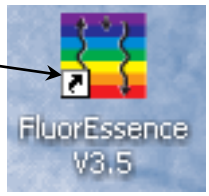
l Remove the CD from the DVD reader.
(The driver on the HORIBA Scientific DVD is newer.)



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

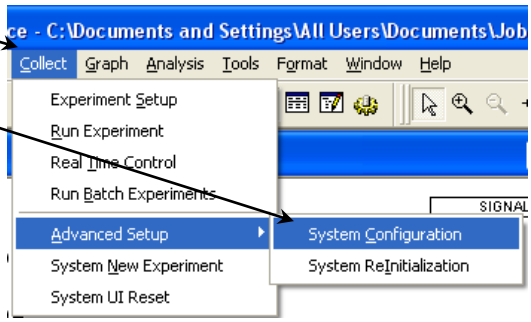
a Start the instrument and computer, according to the instrument's and computer's instruction manuals.

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.



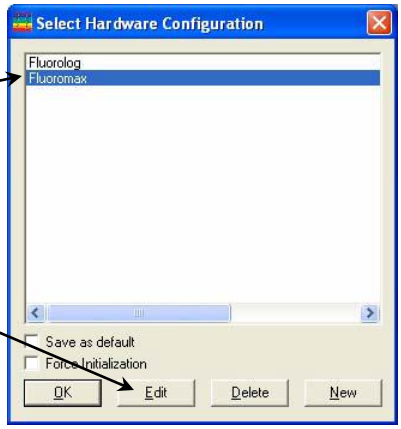
c Choose the Collect menu.

d Choose Advanced Setup, then System Configuration. If you haven't chosen a specific instrument configuration, the **Select Hardware Configuration** window appears:

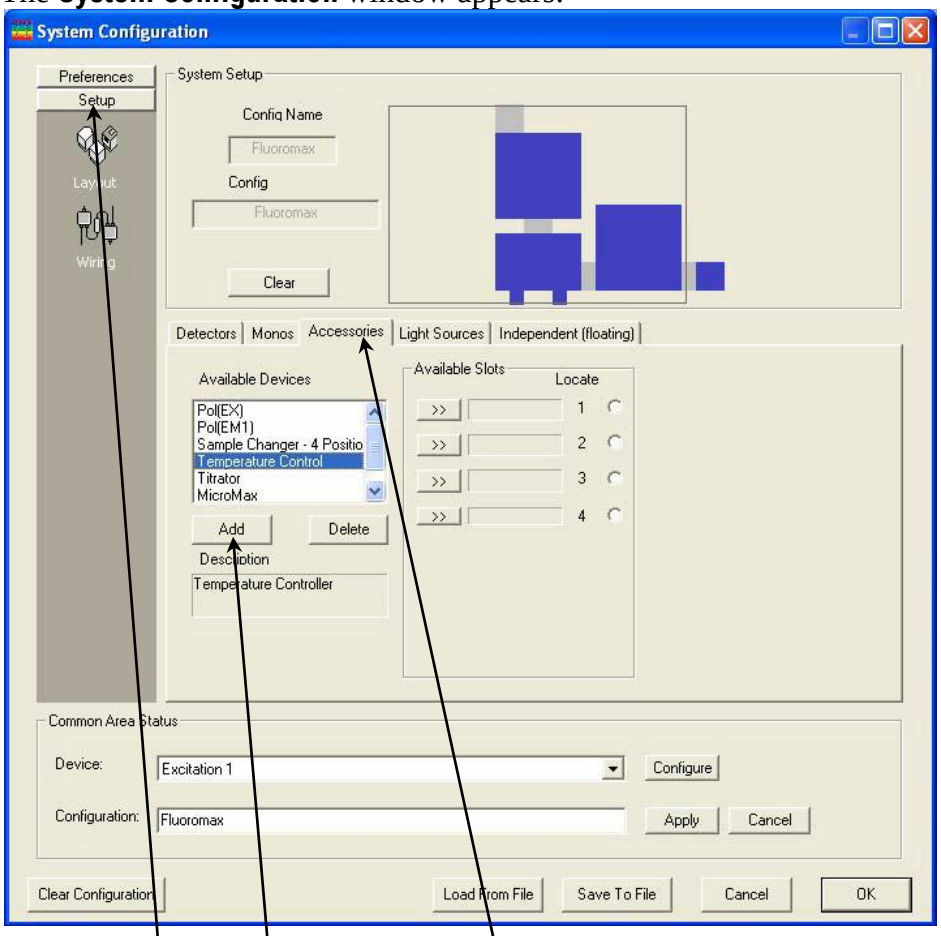


e Choose the desired instrument configuration to which you wish to add the Peltier device.

f Click the Edit button.

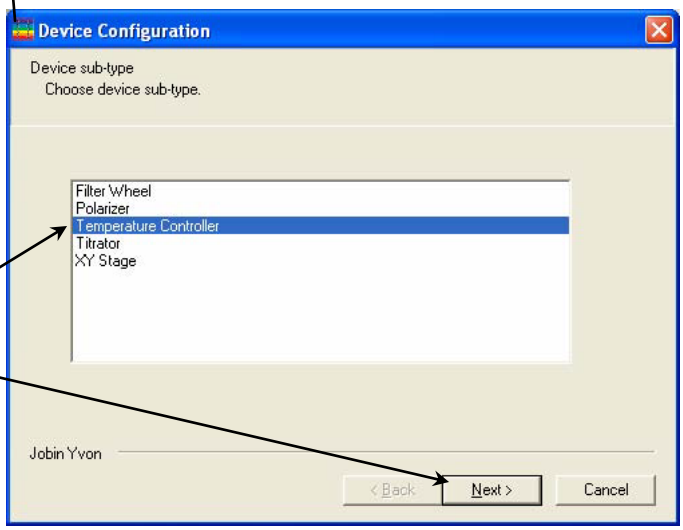


The **System Configuration** window appears:



g Choose the Setup button and the Accessories tab.

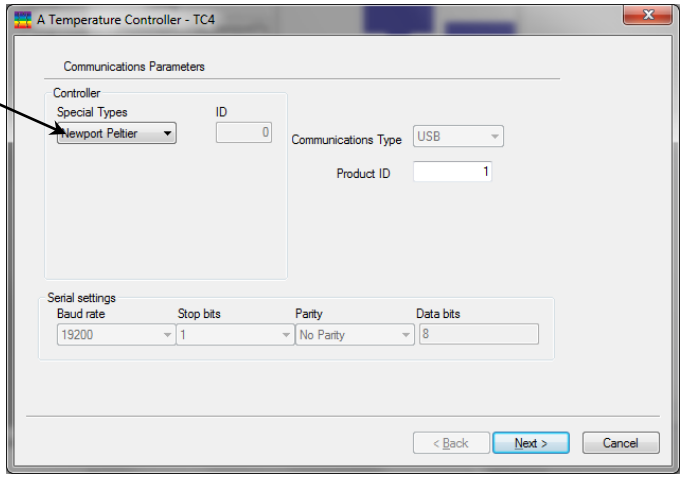
h Choose the Add button.
The **Device Configuration** window appears:



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

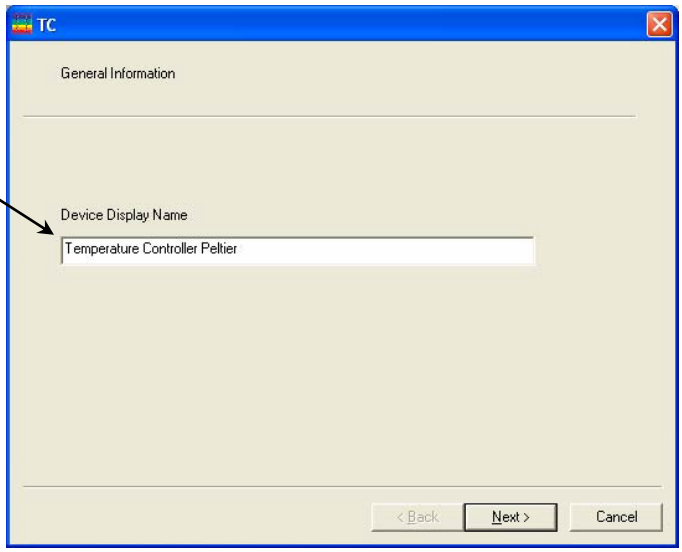
The **A Temperature Controller** window appears:

j From the Special Types drop-down menu, choose Newport Peltier.



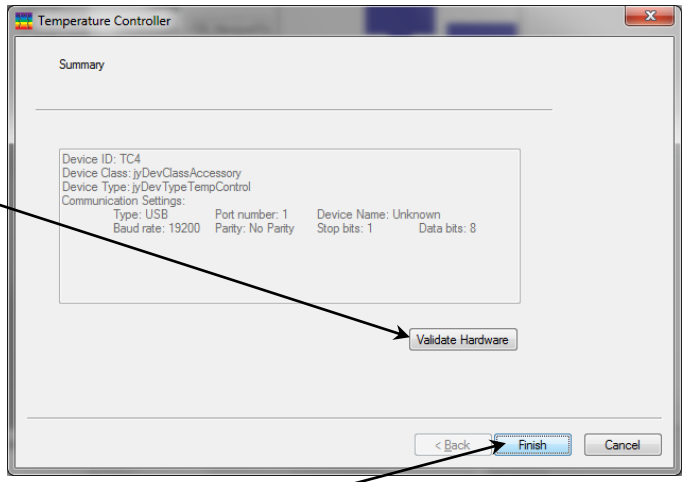
The **TC** window appears:

k In the Device Display Name field, enter a name, say `Temperature Controller Peltier`, for the Peltier device, then click the Next > button.



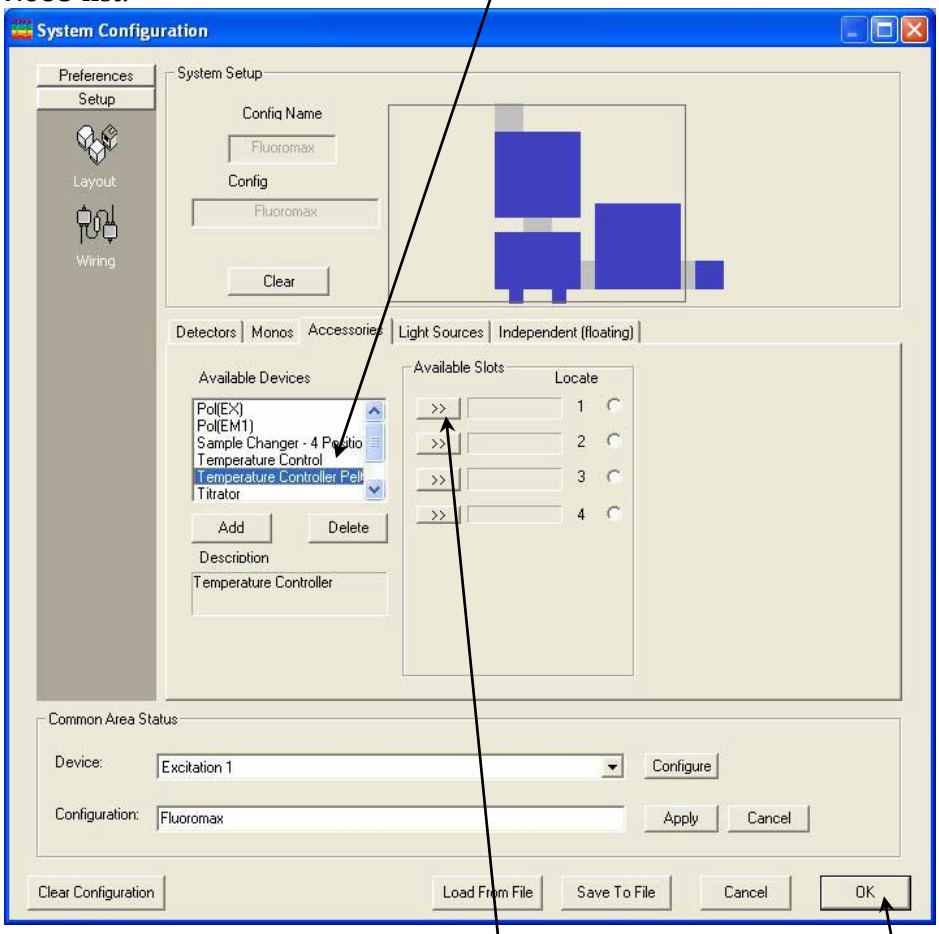
The **Temperature Controller Peltier** (or whatever name you gave the Peltier device) window appears, with a Summary of its parameters:

l If the Newport Peltier 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 all cables are firmly attached.



m Click the Finish button.

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



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

o Click the OK button to complete the session.

3: System Operation

Introduction

There are two major items not controlled from the computer:

- Magnetic stirrer
- Nitrogen purge port

To adjust some advanced Peltier-device parameters

Electric-current limit

The electric-current limit may be adjusted on the front panel of the control unit.

PID settings

PID settings for the rate of temperature change and sensitivity of the control unit to the thermal inertia of the sample holder may be adjusted using the `M530_Util` program provided by Newport (see end of this chapter). Consult the *Newport User's Manual* for details on how to adjust the *PID* parameters.

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**

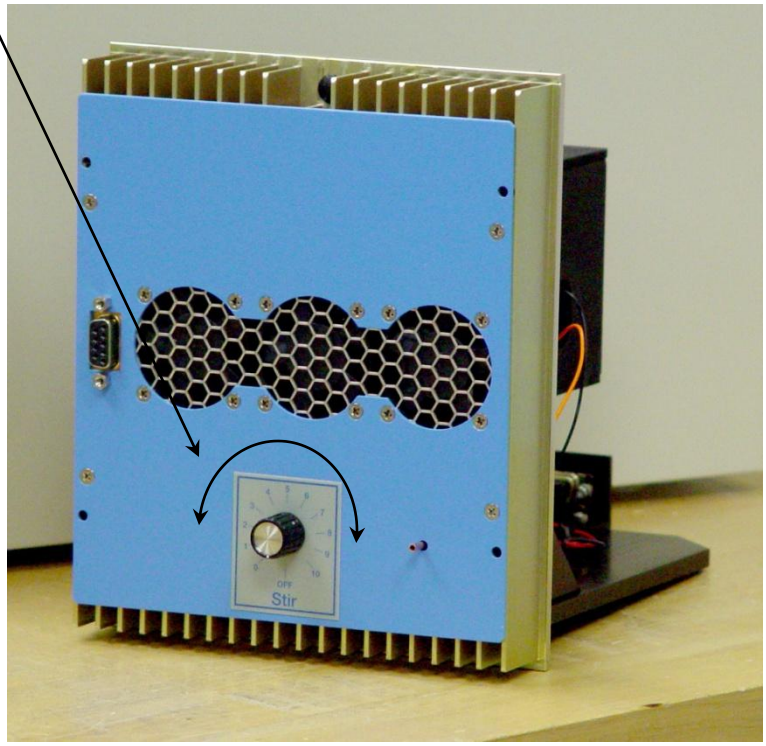
The Differentiator (or Differentiator Time Const.) uses the change in Error Voltage with time, d*V*/d*t* (the derivative). The faster the Error Voltage changes (the larger the slope of d*V*/d*t*), the larger the "opposing" current. Thus, the Differentiator opposes radical changes in the sample's temperature.

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

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.

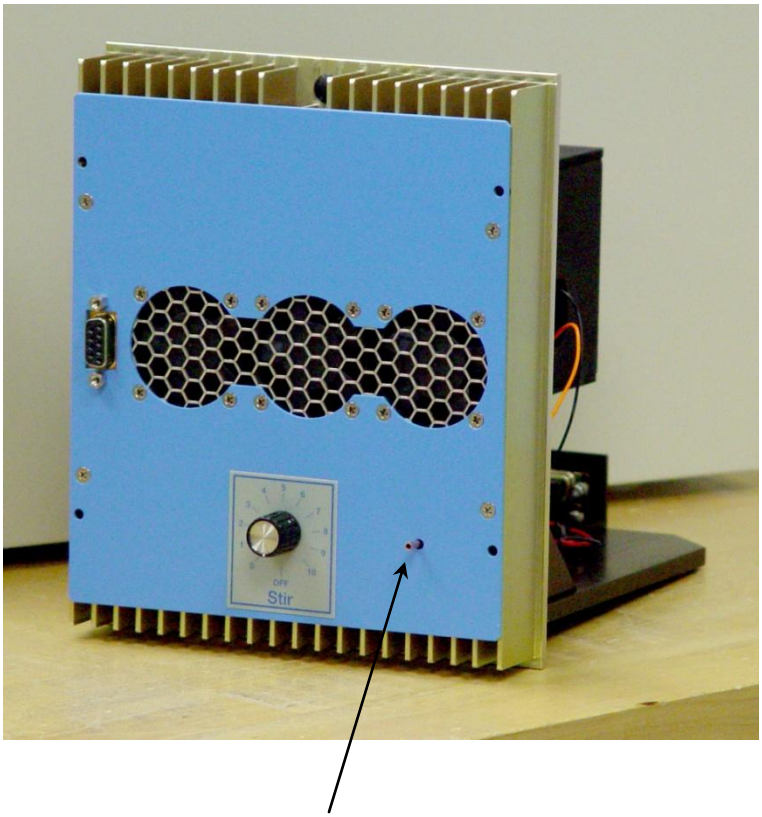
"0" is the slowest speed, and "10" is the fastest speed.



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.



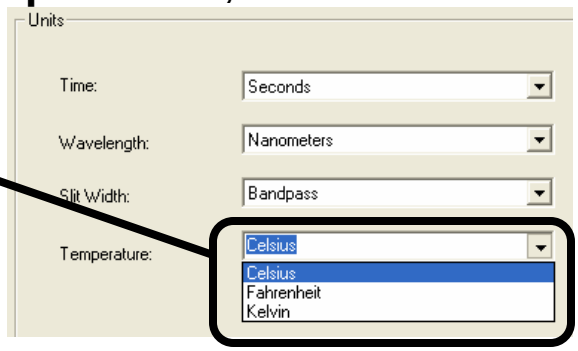
1. Attach the dry-nitrogen gas line to the purging port.
2. Start the gas.

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

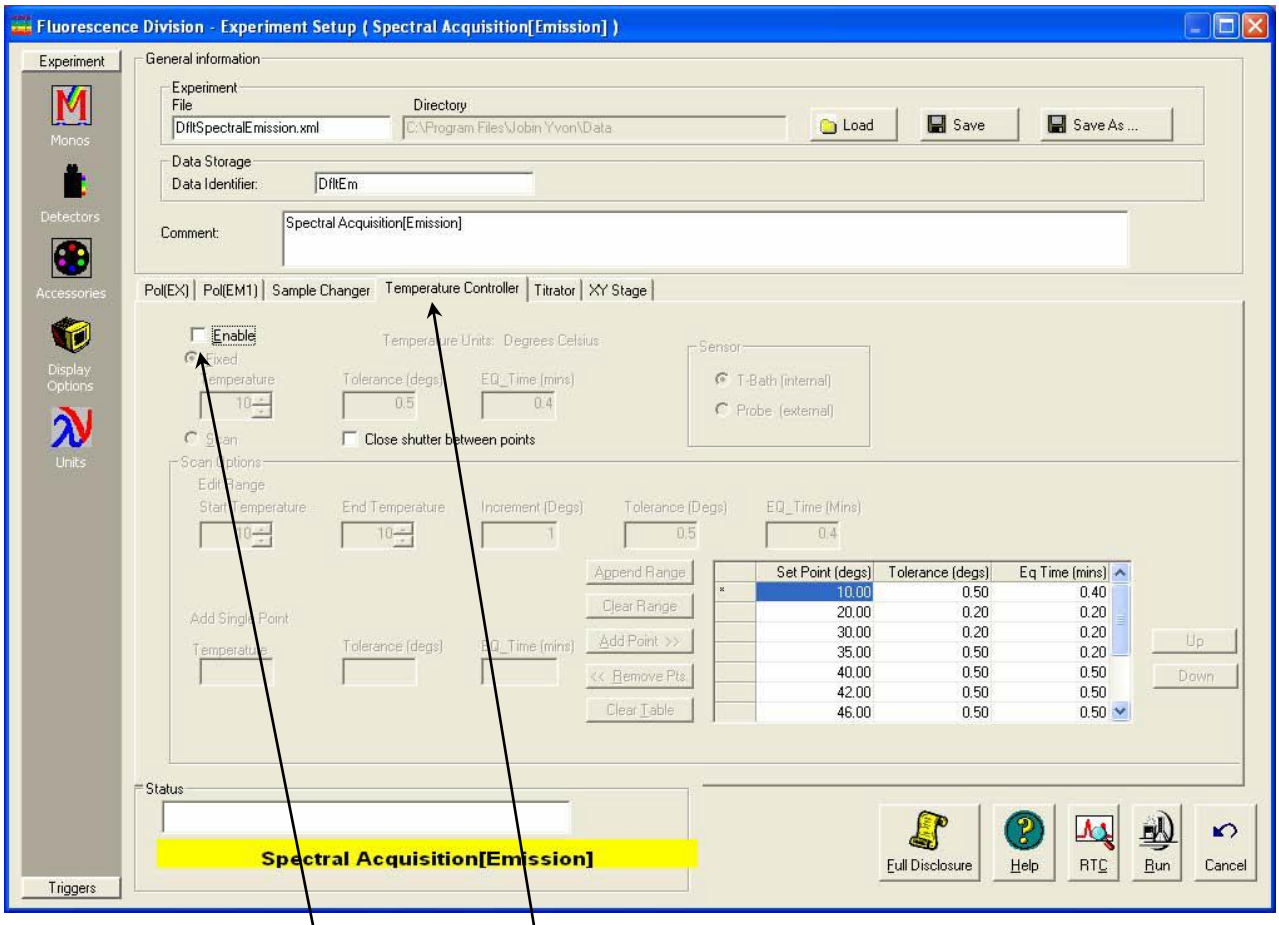
1 In the **Experiment Setup** window, choose the Units icon.

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



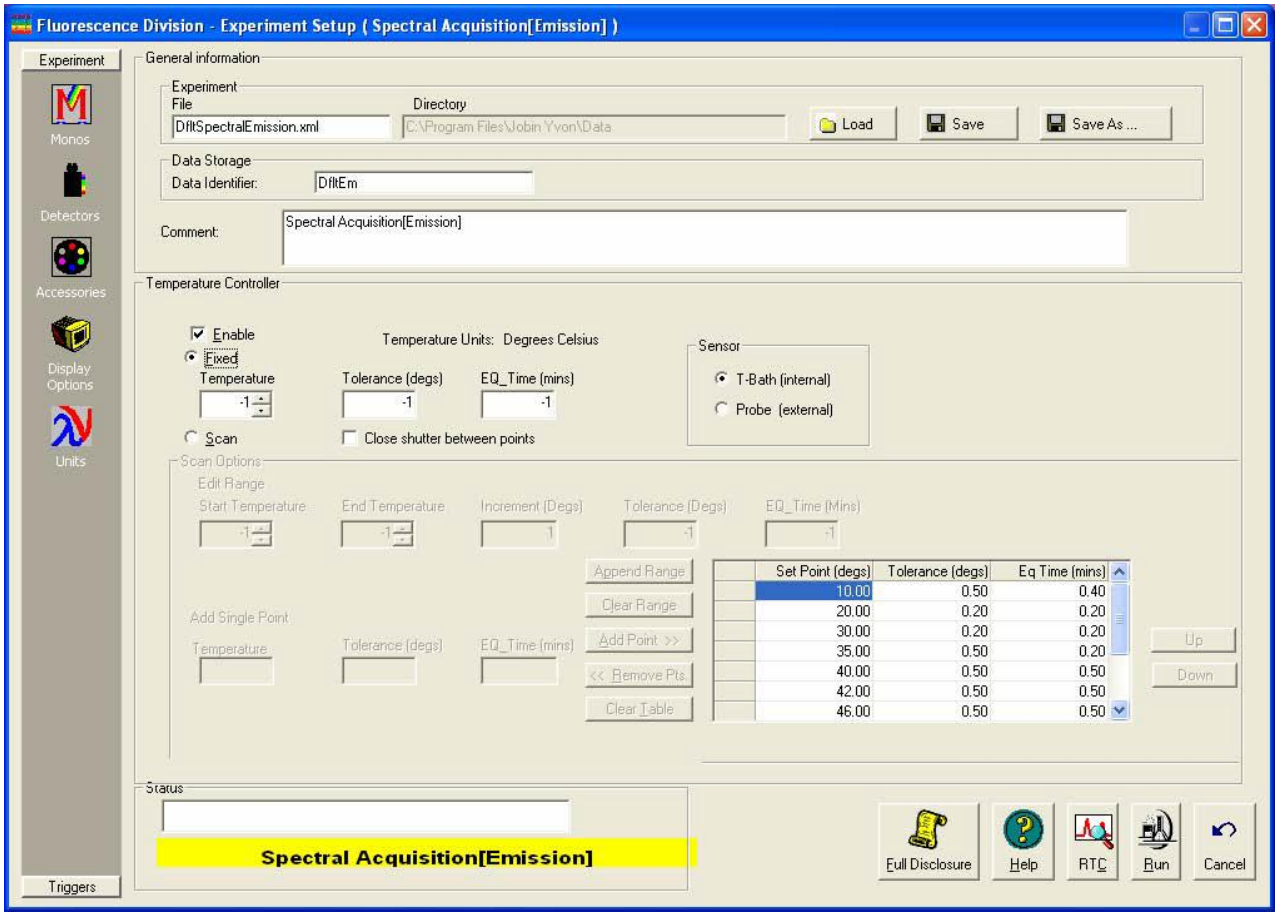
2 Choose the Accessories icon.

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



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

b Check the Enable checkbox to activate the Newport 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
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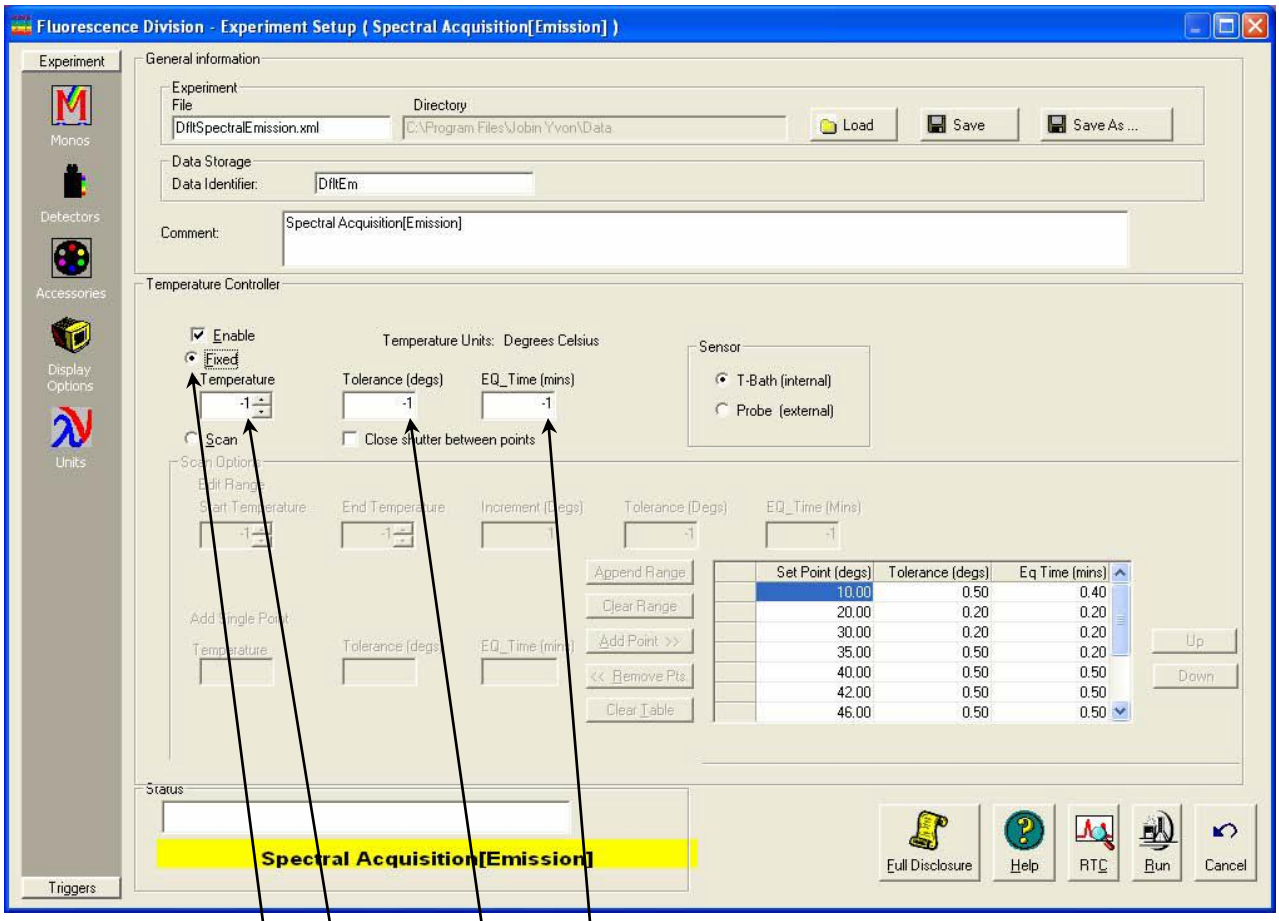
30.00 0.20 0.20
35.00 0.50 0.20
40.00 0.50 0.50
42.00 0.50 0.50
46.00 0.50 0.50
Up
Down
Status
Spectral Acquisition[Emission]
Full Disclosure
Help
RTC
Run
Cancel

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.

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

5 Be sure the T-Bath (internal) radio button is active in the Sensor area.

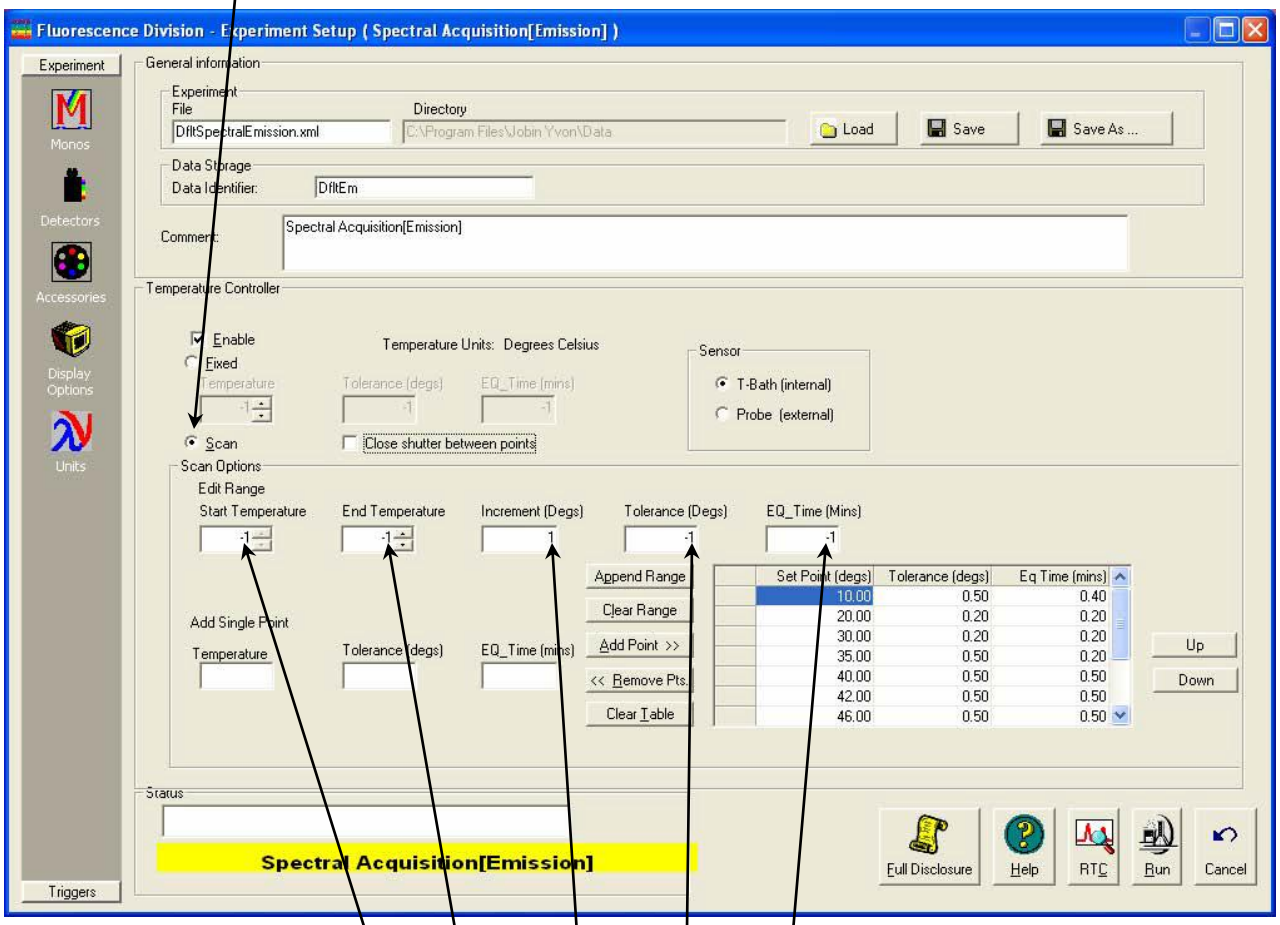
The Peltier device does not use the Probe (external) sensor.

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.

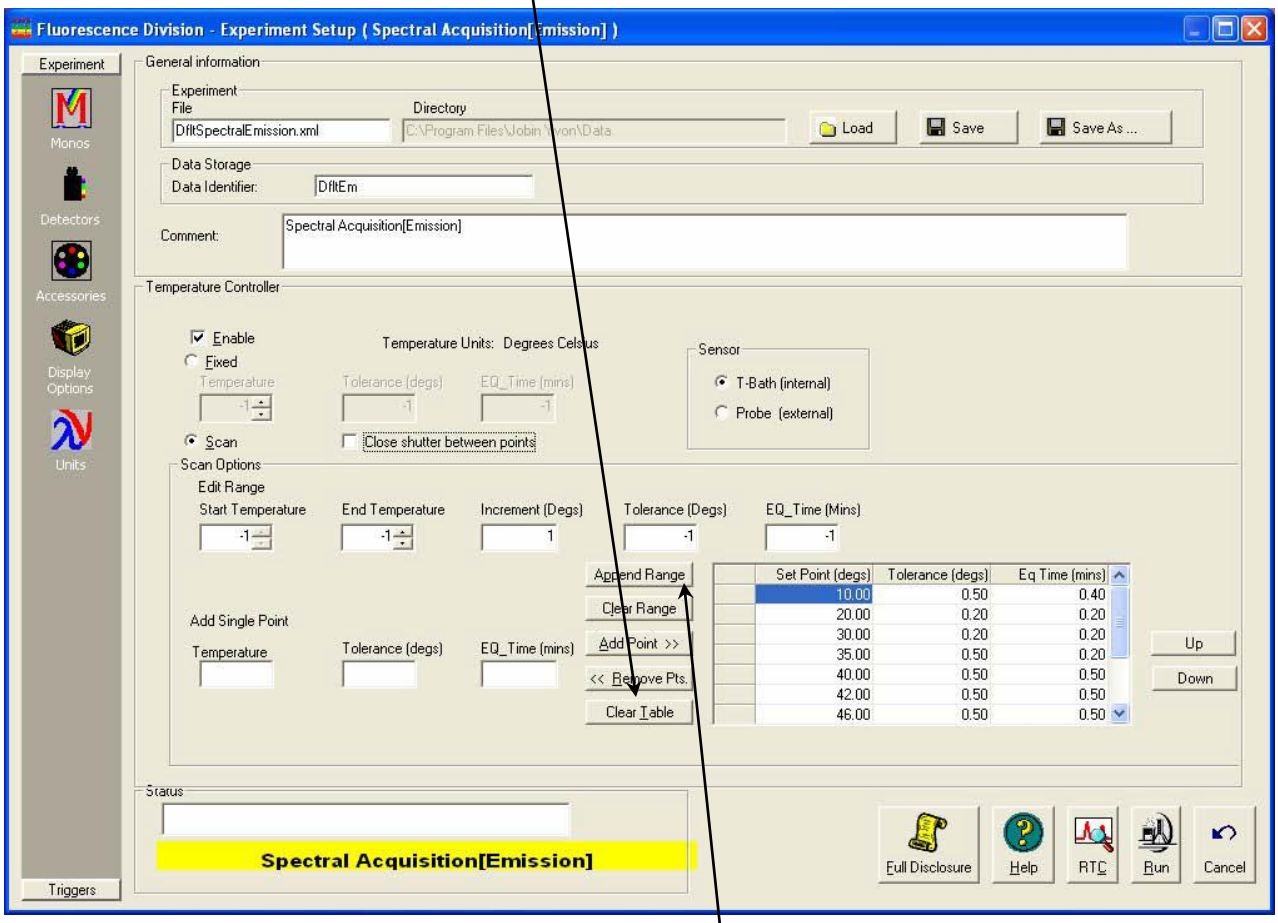
e Enter the equilibration time (in minutes) in the EQ_Time field.

Note: *Values are updated 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.

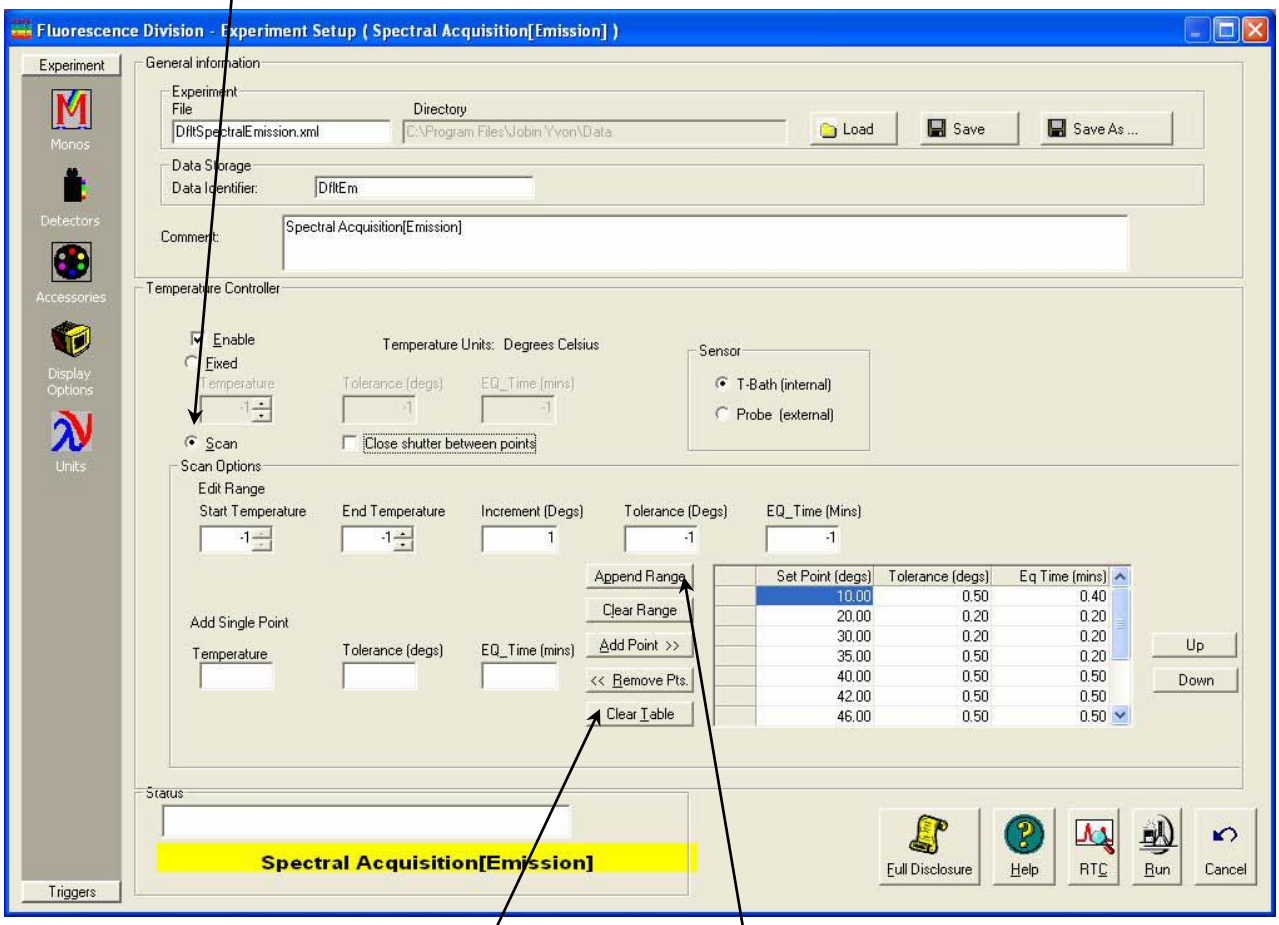
a Click the Clear Table button to clear the existing table of temperatures.



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.

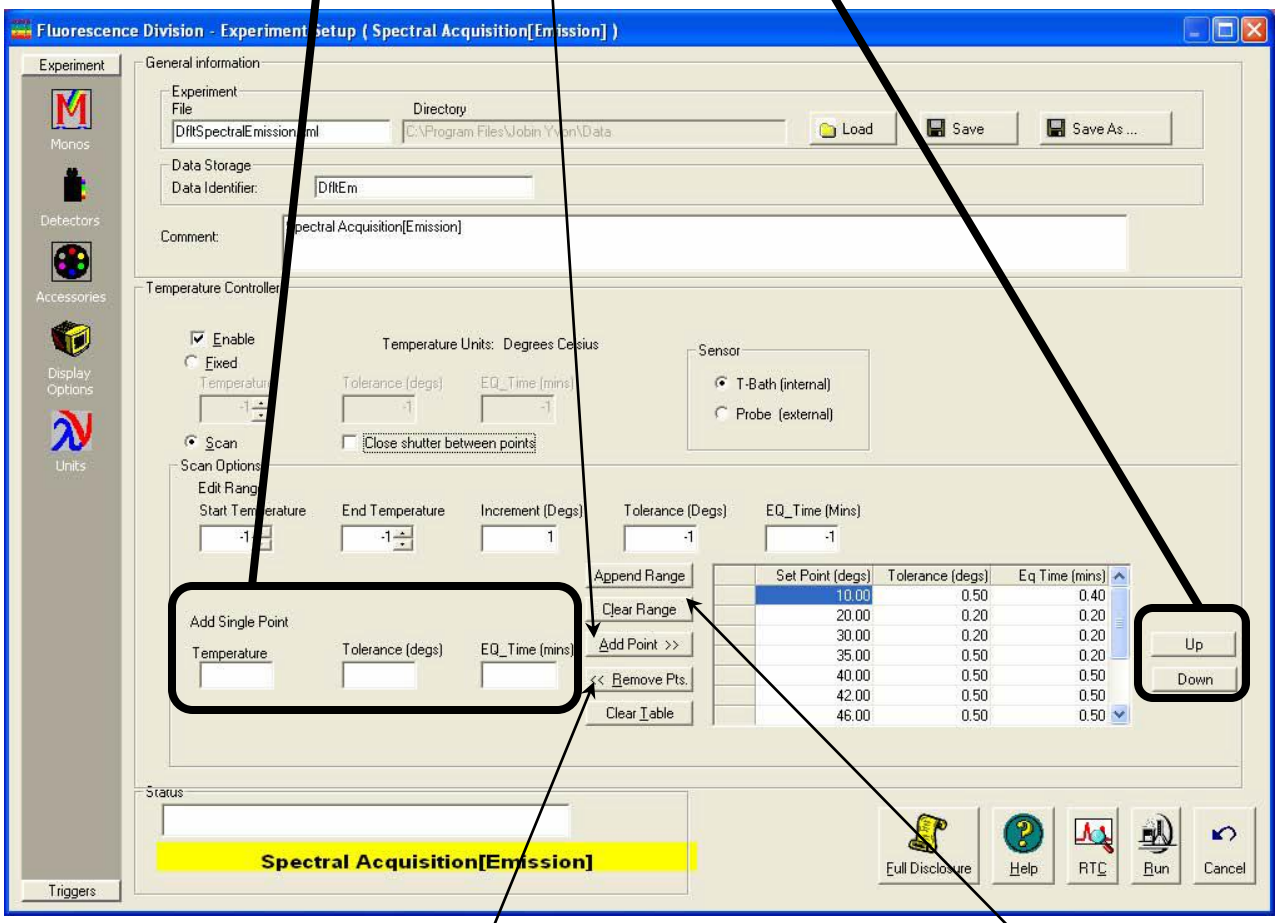
4 Click the Append Range button to place this sequence in the table.

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.

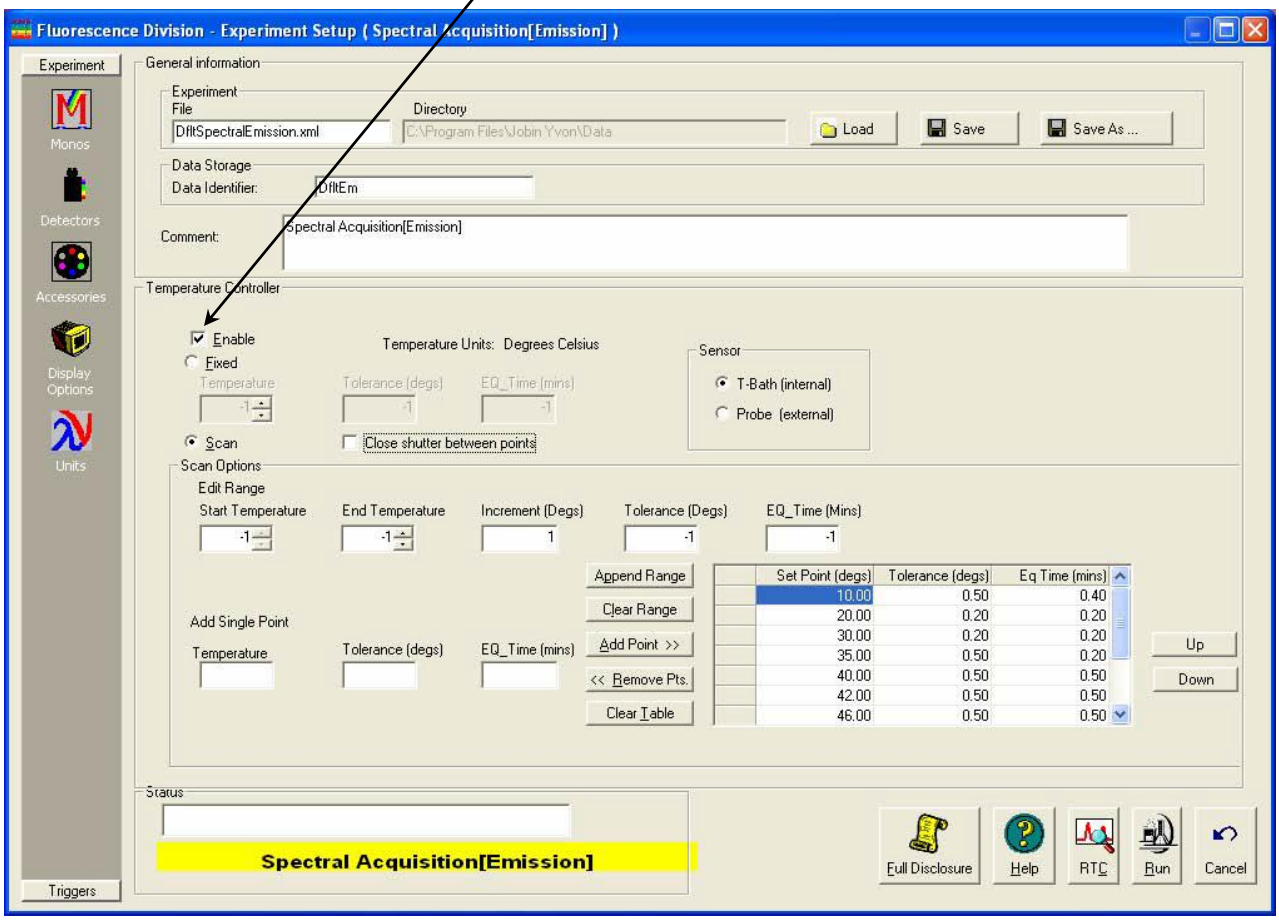


d To remove a row from the table, select the row and click the << Remove Pts. button.

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.*

To run the Peltier device

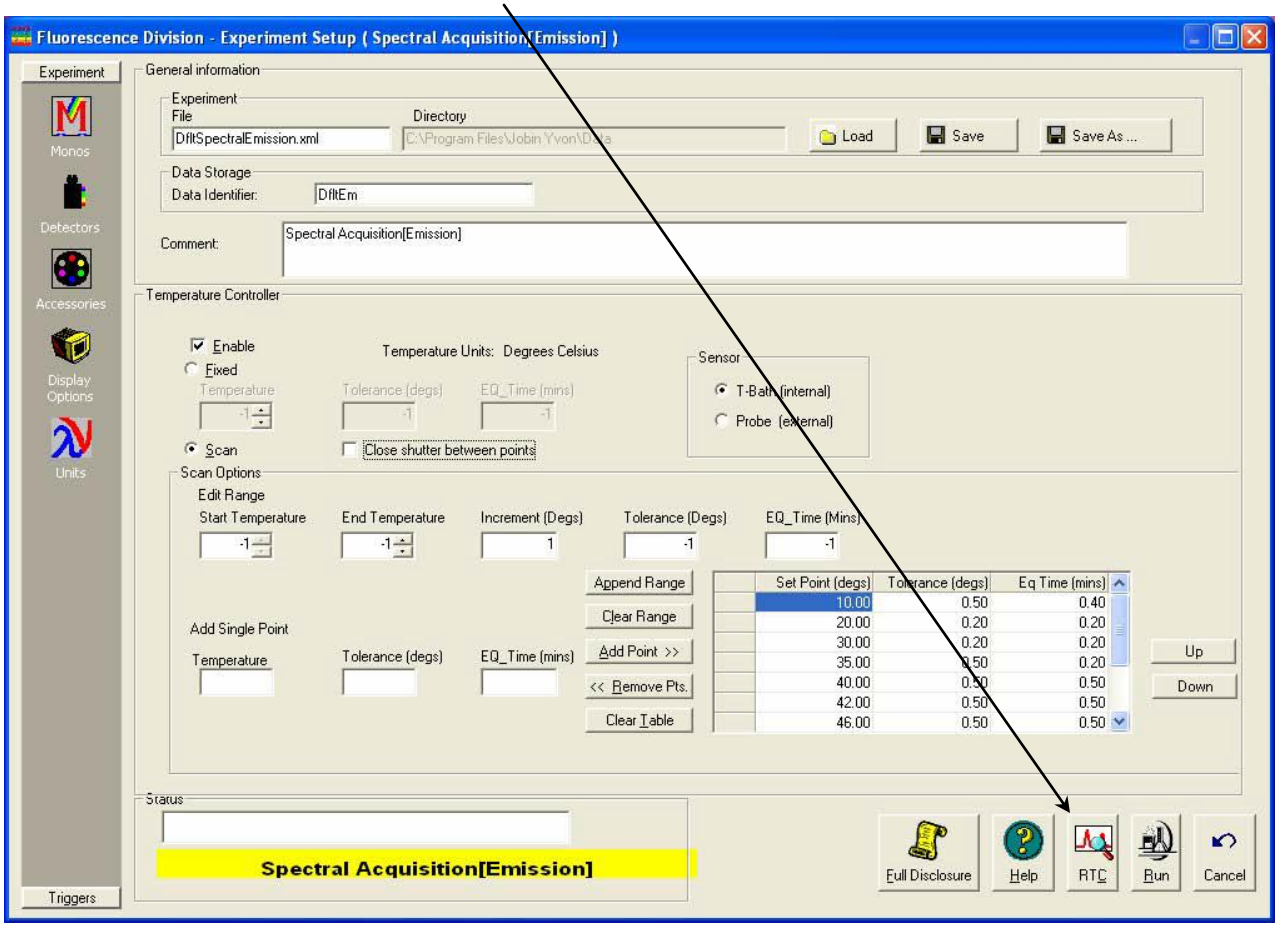
Real time control (RTC)

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.

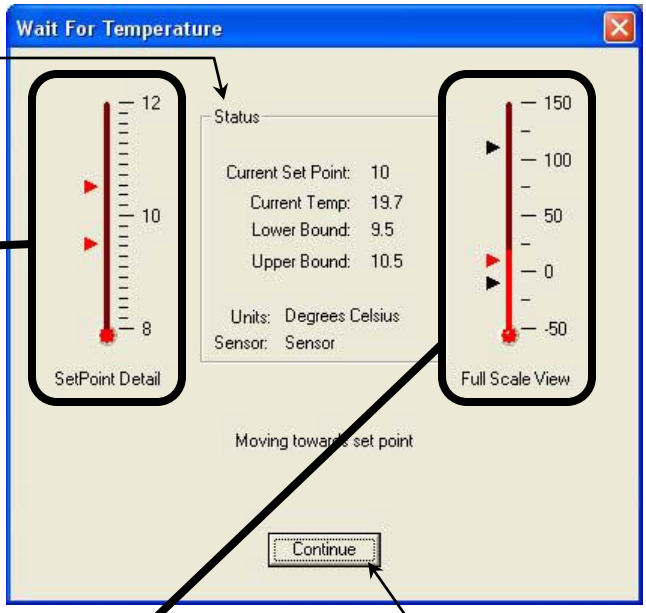
c Click the RTC button.



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.



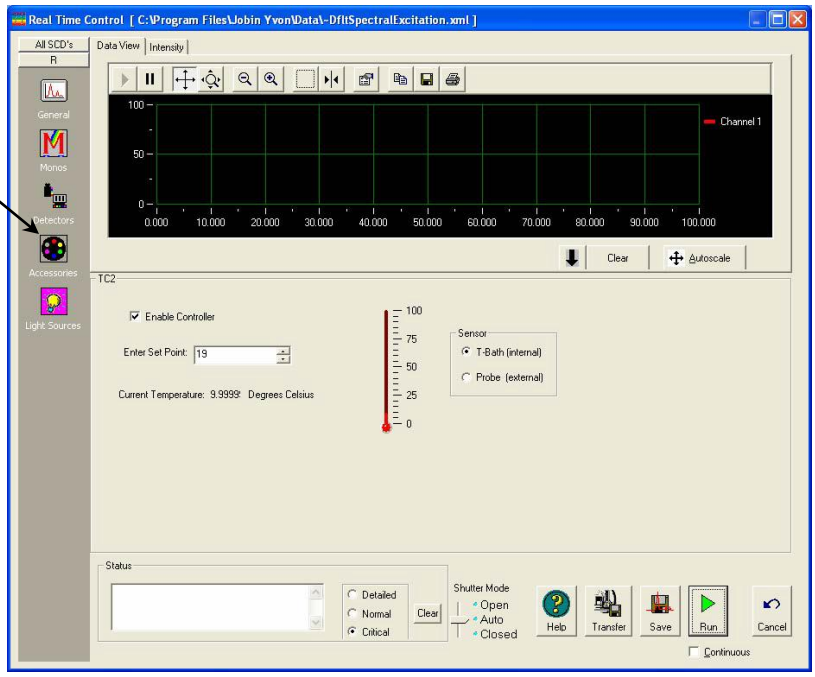
Note: *The Upper and Lower Bounds are half the Tolerance above and below the "Temperature" setpoint.*

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

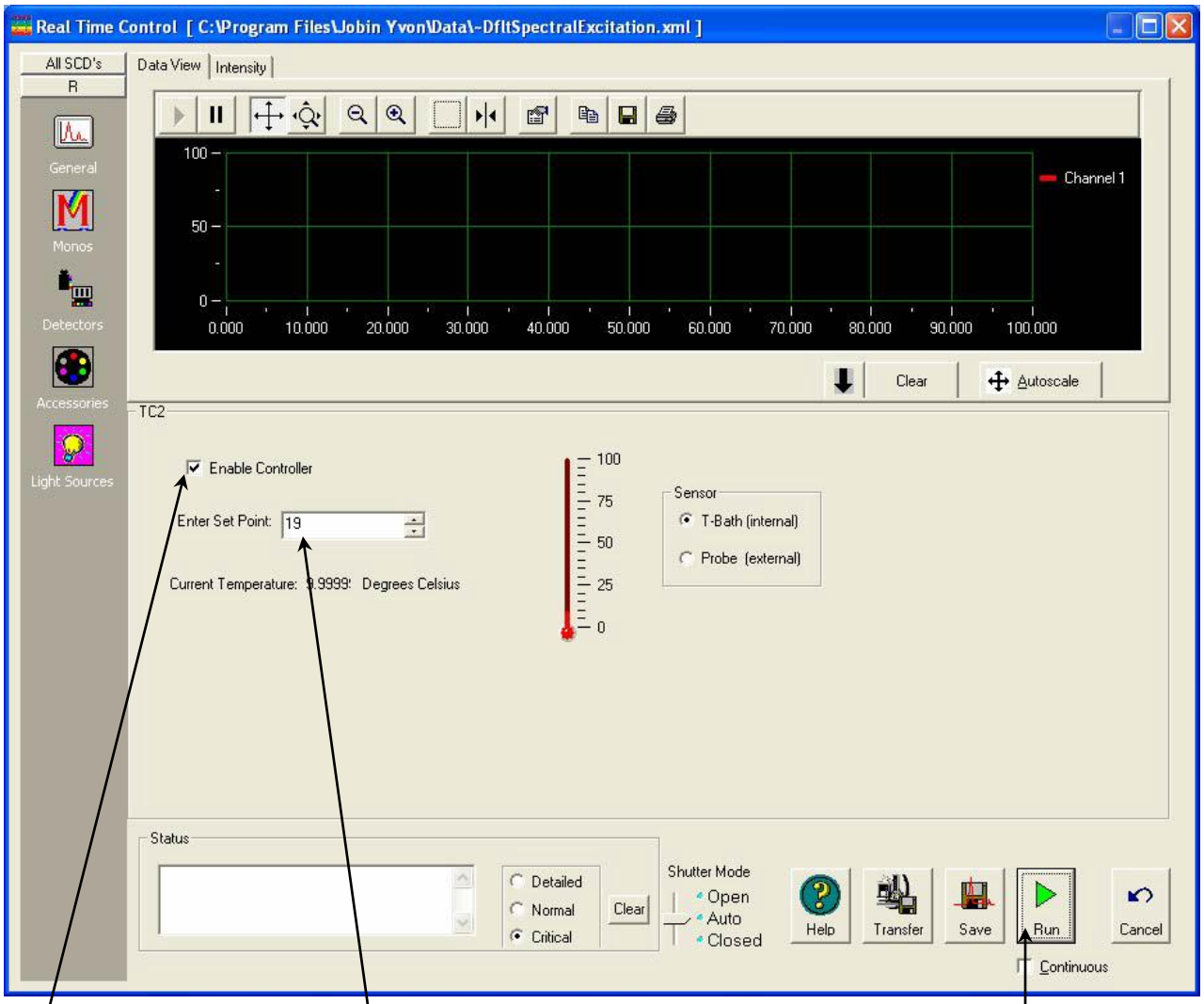
2 Click the Continue button.

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.

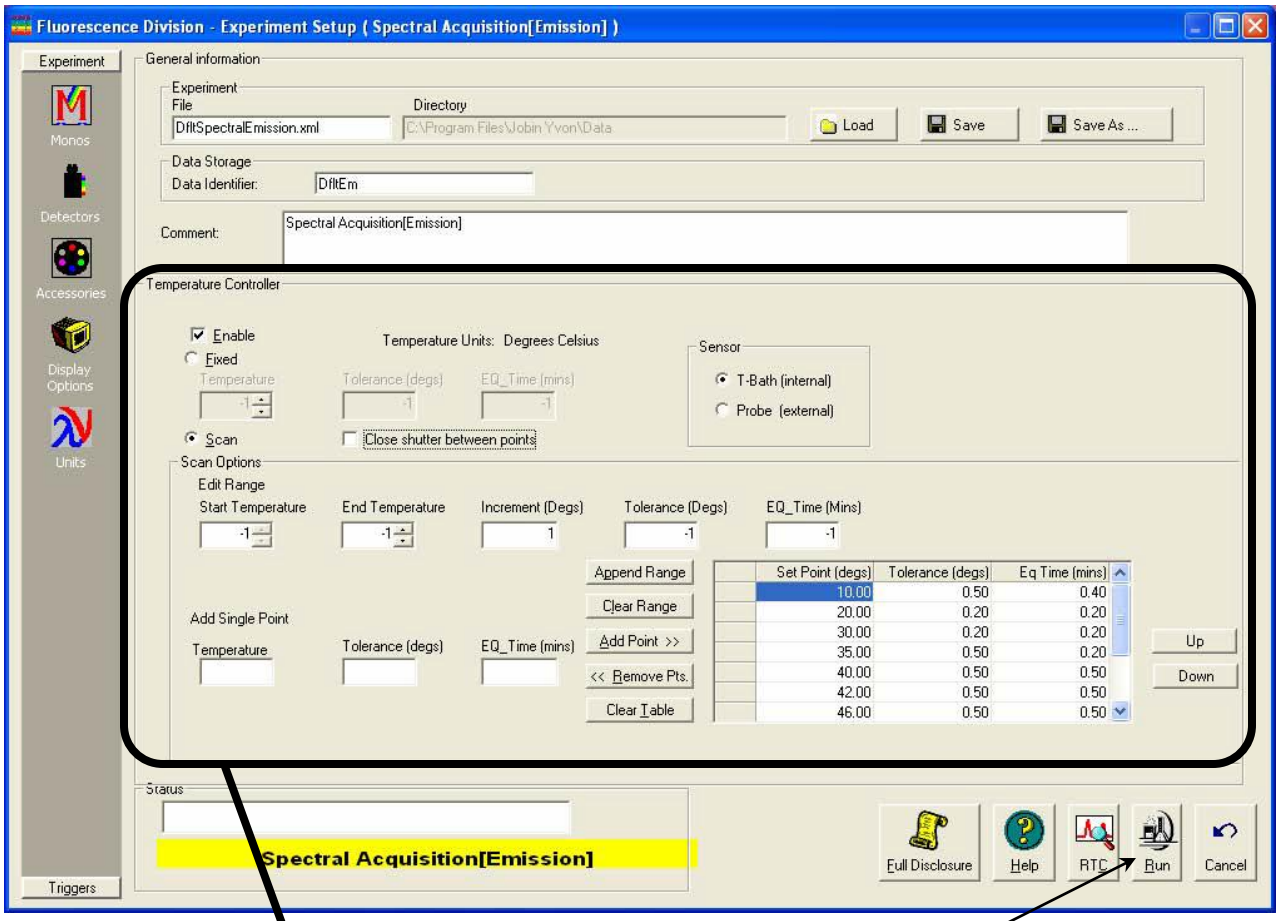


a Click the Enable Controller checkbox to activate the Peltier device.

b Change the set point in the Enter Set Point field.
The present temperature is given digitally and graphically.

5 Click the Run button to run an experiment.

Normal experiment setup



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

2 Click the Run button.

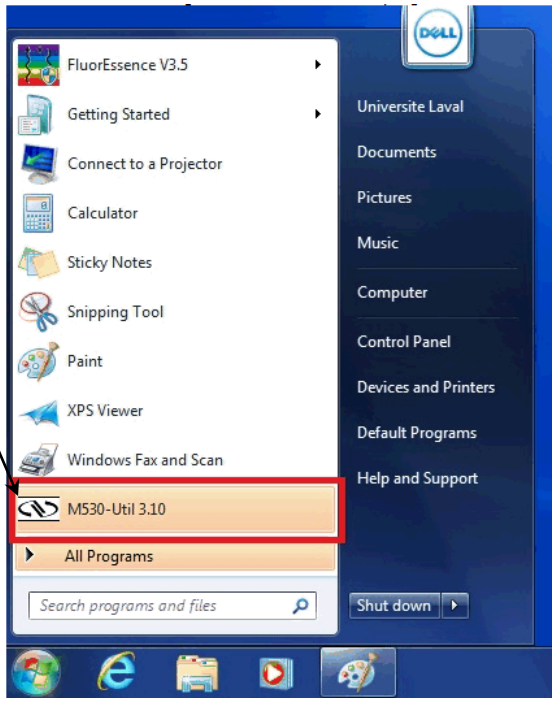
The experiment using the Peltier device starts.

To use the Newport utility program

M530_Util

1 In the Windows® Start menu, select M530-Util from All Programs.

Be sure to run the program with administrator privileges (right-click Run As Administrator).



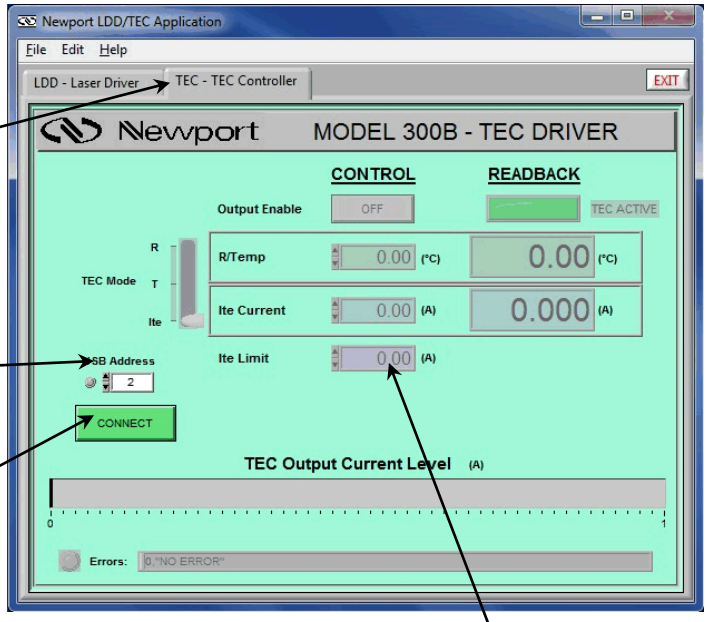
The **Newport LDD/TEC** window opens:

2 Select the TEC - TEC Controller tab.

3 Change the USB Address field to `2`.

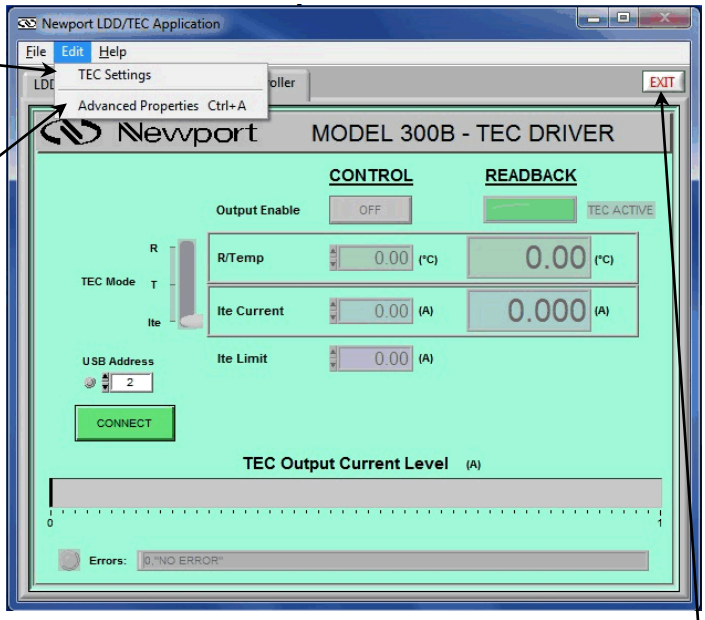
4 Click the Connect button.

5 Set the Ite Limit field to `5.00`.



6 Choose Edit.
A drop-down menu appears.

7 Select TEC Settings.

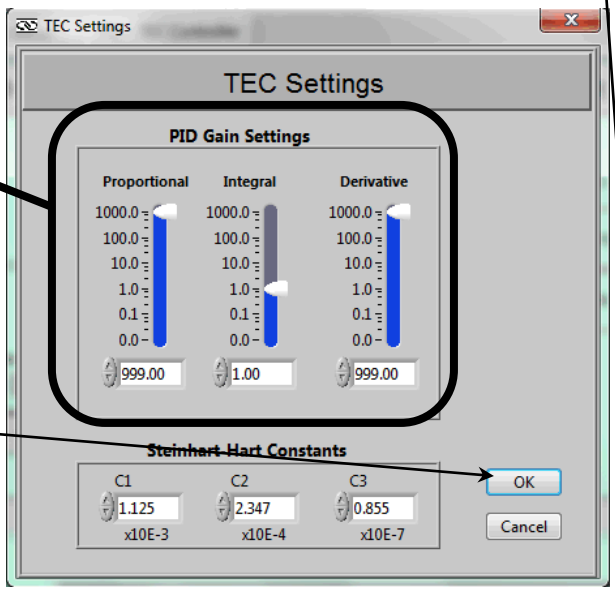


The **TEC Settings** window appears.

8 Set the PID Gain Settings area as desired:

9 Click the OK button.
The **TEC Settings** window closes.

10 Click the Exit button to close the Newport Utility.



4: Troubleshooting

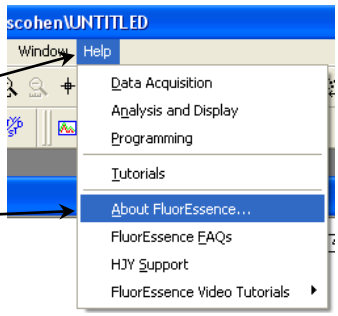
Consult the *Newport User's Guide* for any problems that may occur with the control unit. Below are listed some problems that might occur:

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.
Peltier Sample Cooler does not operate.	No Peltier accessory is available	Select configuration with Newport 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.

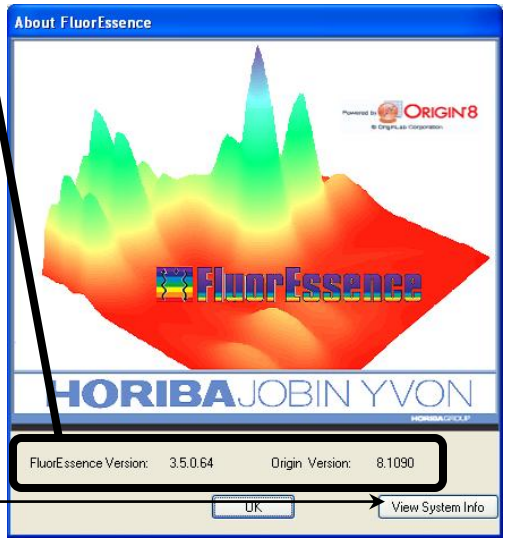
Further assistance...

Read all software and accessory manuals before contacting the 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.

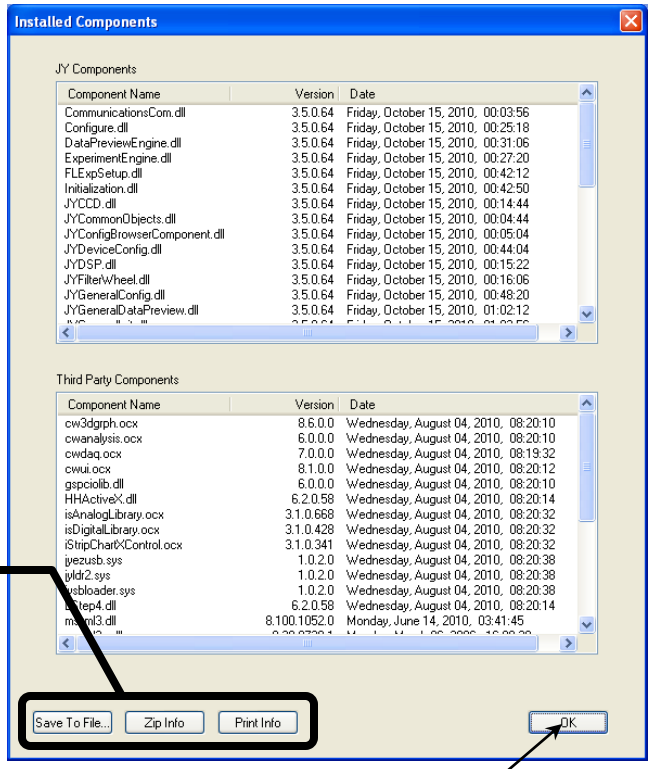
1. If this is the first time the problem has occurred, try turning off the system and accessories.
   After a cool-down period, turn everything back on.
2. Make sure all accessories are properly configured, and turned on as needed.
3. Following the instructions in System Operation, run a xenon-lamp scan 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 http://www.horiba.com/scientific/products/fluorescence-spectroscopy/ to see if the question is addressed.
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 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.
   a. Choose the Help menu.
   b. Choose About FluorEssence....
      The **About FluorEssence** window opens:



Near the bottom are the FluorEssence™ and Origin® version numbers.



c Click the View System Info button.
The **Installed Components** window appears, displaying all the software required for FluorEssence™.



d Record the information by clicking the:

- Save To File... button, which saves the information to a file;
- Zip Info button, which compresses the information while saving it;
- Print Info button, which prints out the software information.

e Click the OK button to close the **Installed Components** window.

f Click the OK button to close the **About FluorEssence** window.

g Click the OK button 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 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.).

5: Specifications

Computer requirements

- J973005 or equivalent PC computer with USB port

Software requirements

- Windows® XP or higher
- FluorEssence™ 3.5.1.99 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 3.5″ (8.8 cm) W × 8.5″ (21.5 cm) H × 12.6″ (32 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

6: Index

Key to the entries:

Times New Roman font	subject or keyword
Arial font	command, menu choice, or data-entry field
Arial Condensed Bold font	dialog box
`Courier New font`	file name or extension

F

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HORIBA
Scientific

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[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.