

AppliedPhotophysics

Ultrasensitive Spectroscopy for the Life Sciences

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User Manual Chirascan CS/PCM

**Chirascan Series Four Position Thermostatted Sample Changer
February 2012**

Document 4207Q117C03.01



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This document contains important safety information. Read this document and the Chirascan series User Manual before attempting to install or use the CS/PCM. Failure to do so could result in death or serious injury.

USE OF THIS DOCUMENT

This document is intended to inform the operator of Applied Photophysics' CS/PCM 4-Position Thermostatted Sample Changer on its design, installation and operation with either the Chirascan or Chirascan-plus spectrometer. The CS/PCM is supplied by Quantum Northwest Inc., Washington State, U.S.A, and this document should be used in conjunction with the User Manuals supplied by Quantum Northwest and that applicable to the spectrometer. It is assumed that the user of this document is familiar with the operation of the Chirascan or Chirascan-plus, and with Applied Photophysics Pro-Data software. In particular it is assumed that the user is familiar with the hazards associated with the operation of the spectrometer, and has read the safety information contained in its User Manual.

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WARNING

The CS/PCM electronics module is powered by the mains electricity supply which can produce a shock leading to serious injury or death. Do not connect or disconnect the module from the mains supply unless the supply is powered off at source. Ensure all communications and electrical connections are made before powering on the module. Exercise care during operation and do not operate units with their covers removed. Operate the accessory using only the cables provided. Never operate the accessory with damaged cables.

CAUTION

The CS/PCM turret and the sample cell may be very hot or cold, causing injury to the user when touched. Ensure that it has been allowed to reach a safe temperature before handling.

NOTICE

Corrosive chemical and organic solvents can cause damage to the CS/PCM. Do not allow corrosive fluids to come into contact with any part of the accessory. It is advisable to fill the sample cell before installing it in the CS/PCM. Do not clean the accessory with organic solvents. Use only a soft cloth and water or a mild detergent solution.

CS/PCM INSTALLATION AND OPERATIONAL REQUIREMENTS

Environmental and electrical requirements

The CS/PCM has no environmental requirements additional to those of the Chirascan spectrometer. Operation of the CS/PCM is controlled by the Chirascan software, which communicates with an electronics module (TC425) manufactured by Quantum Northwest. Cables and mains leads are supplied by Applied Photophysics.

Bench space

The footprint of the CS/PCM electronics module is about 260 mm x 260 mm. Space should be available for this to the right of the Chirascan Sample Handling Unit.

Nitrogen purge gas

The PCS3 has no purge gas requirements additional to those of the Chirascan spectrometer.

Circulating water

The CS/PCM requires a circulating water flow rate of about 200 – 300 ml per minute. To achieve this, a pressure of approximately 0.2 – 0.3 bar (3 to 4.5 bar) is needed.

Servicing

Servicing of the CS/PCM should only be undertaken by qualified personnel. If you are in any doubt at all please contact the Applied Photophysics Technical Support Department at the address give on the front of this User Manual.

GLOSSARY

The following abbreviations may be found in this User Manual

APL	Applied Photophysics Ltd.
CD	circular dichroism
DSC	differential scanning calorimetry
QNW	Quantum Northwest Inc.
SCP	spectrometer control panel
SHU	sample handling unit
TCU	temperature control unit

HYPERLINKS

This document contains hyperlinks between references (for example the Contents tables, or references to Sections or Figures in the text), and sources. To follow a link, place the cursor over the reference and use **CTRL+click**. Hyperlinks in the text are indicated by underlined [blue](#) font.

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1 INTRODUCTION

The CS/PCM 4-position cell autochanger Peltier temperature controlled system is designed to be used with either the Chirascan or Chirascan-plus circular dichroism spectrometer. The accessory consists of two main components, the turret, shown in Figure 1.1, and a separate electronics module.



Figure 1.1: CS/PCM turret

The CS/PCM is based on the Quantum Northwest (QNW) Turret 400™ cuvette holder unit, which uses the Peltier effect to raise or lower the temperature of the sample holder. When a current is made to flow across the junction of two dissimilar electrical conductors, the result is an increase in temperature on one side of the junction, and a decrease in temperature on the other. If the current is reversed, the direction of the thermal gradient will also be reversed. Unlike resistive heating, the Peltier effect can therefore be used for both heating and cooling.

To maintain the temperature gradient the current must also be maintained, meaning that electrical energy must be continuously supplied to the system, resulting in the generation of heat which has to be removed. Circulating water is used for this, but it is important to remember that the circulating water is not controlling the temperature of the system, it is only removing heat, and the temperature of the circulating water does not need to be controlled accurately.

The turret mounts into the Chirascan Sample Handling Unit (SHU), and is designed to be easily installed and removed. The temperature is read by a probe positioned in the wall of the turret at about the height of the windows, and the temperature is controlled to this probe. It may be that sample temperature differs slightly from this, particularly at very high or low temperatures, and the Chirascan software also allows the temperature to be read by a probe immersed in the sample, although it is still controlled to the turret probe.

The CS/PCM accepts rectangular cuvettes, which mount directly into the turret. Four integral stirrers are included in the unit.

The electronics module provided with the accessory is the QNW TC425, shown in Figure 1.2. The Chirascan software communicates with the TC425 to provide complete temperature control.



Figure 1.2: the CS/PCM electronics module.

2 INSTALLATION

2.1 The Chirascan Sample Handling Unit

The Chirascan Sample Handling Unit (SHU) is the lidded chamber that mounts to the right of the monochromator unit, where it is held in place by four thumbscrews. Normally, the SHU is not removed from the spectrometer, although this can easily be done for transportation or to install the SF3 stopped-flow accessory.

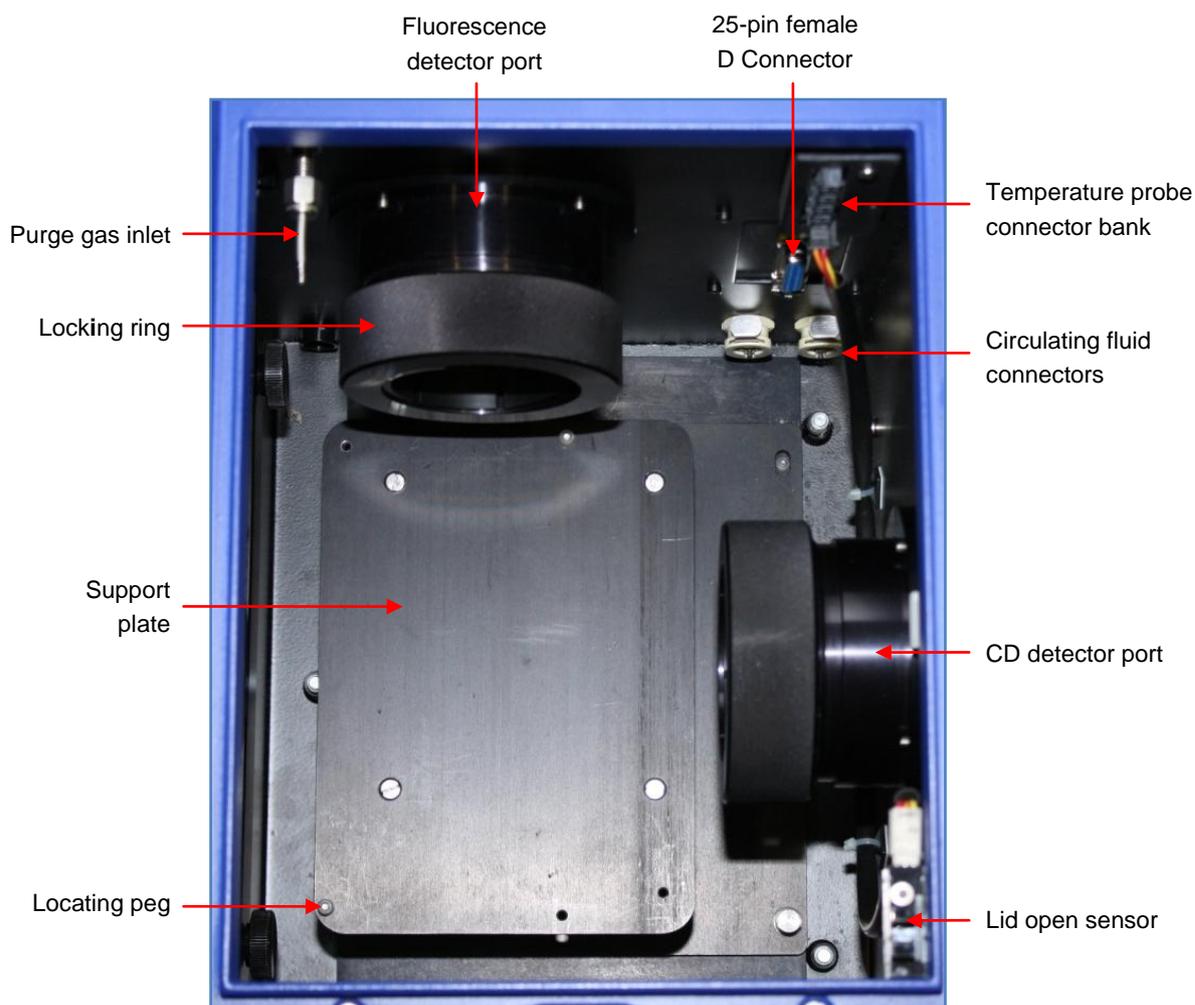


Figure 2.1: the interior of the Chirascan Sample Handling Unit

The interior of the SHU is shown viewed from the front with the lid raised in Figure 2.1 (the aperture for light inlet from the monochromator is to the left, opposite the CD detector port).

There are two detector ports on the SHU, to the right and rear. Normally the right port is used for CD and the rear port for fluorescence. The purge gas inlet is to the left rear upper, the circulating fluid connectors to the right rear lower, and the electrical and additional temperature probe connectors are above the circulating fluid connectors.

On the right within the SHU is a lid open sensor; when the lid is raised, no experiment can be started and any that are in progress will be paused.

2.2 Hardware installation

2.2.1 Installing the temperature control unit

The temperature control unit stands on a mounting plate which locates on a support plate within the SHU. The unit on its mounting plate is shown in Figure 2.2.

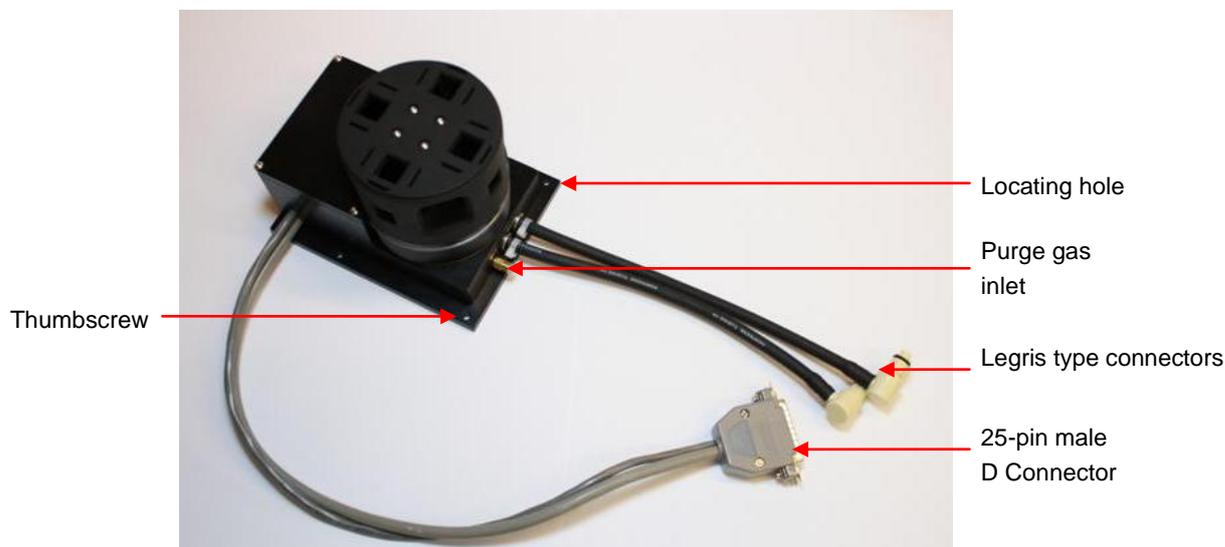


Figure 2.2: CS/PCM turret on its mounting plate

Before installing the turret, remove any detectors from the SHU. The detectors are held in place by knurled locking rings, and are released by turning the ring anticlockwise (counterclockwise); the detectors can then be slid gently from their ports.

Remove the purge gas line connecting the inlet on the SHU to the CD detector cowling. The purge gas lines are secured by push fit connectors, which are released by pressing the outer (orange) flange towards the connector body.

Place the mounting plate of the turret over the support plate in the SHU, with the circulating fluid hoses to the rear. The plate seats down with the holes at the front left and rear right locating over the corresponding pegs on the support plate. When the plate is seated down, secure it in place by tightening the two captive thumbscrews, positioned at the right front and left rear of the mounting plate.

Connect the Legris type fluid circulator connectors by pushing home the male on the turret to the female on the SHU. Connect the electrical cable and secure with the wire clips. Note that the route for the cable is over the CD detector port, where it is held in place by a cable clip.

Fit the purge gas line supplied with the CS/PCM from the SHU inlet to the turret inlet. The interior of the SHU should now appear as in Figure 2.3.



Figure 2.3: interior of the SHU with the turret in place

The detectors can now be re-installed. The snout of the CD detector should be set about 4 mm from the temperature control unit, i.e. close but not touching (if a cowling is present on the detector snout, this should be removed). The snout of the fluorescence detector fits into a cowling with a rubber tip that should also be set about 4 mm from the temperature control unit. The fluorescence detector cowling is shown in Figure 2.4.

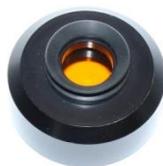


Figure 2.4: fluorescence detector cowling

The interior of the SHU should now appear from the front as in Figure 2.5, and from the right as in Figure 2.6. The cuvette is in the measuring position.



Figure 2.5: interior of the SHU with all connections made (front view)

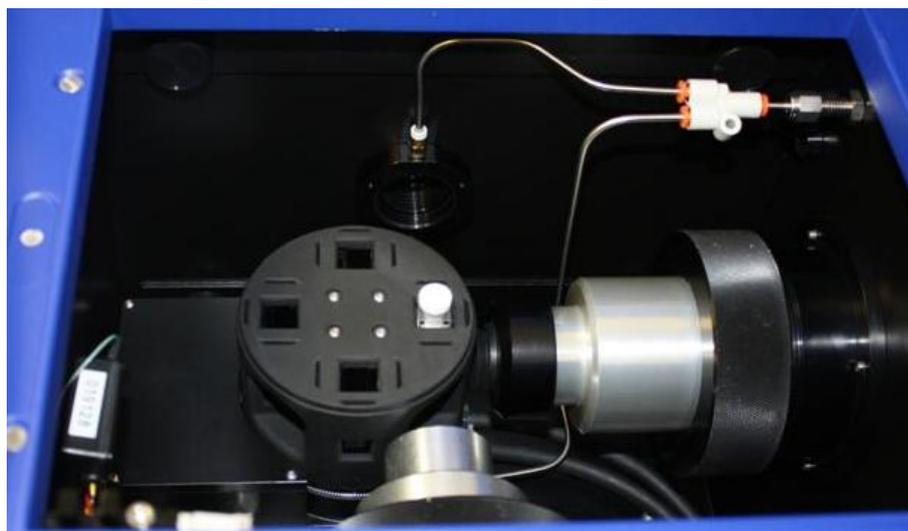


Figure 2.6: interior of the SHU with all connections made (right view)

2.2.2 Circulating water connections

The CS/PCM requires a circulating water flow rate of about 200 – 300 ml per minute. To achieve this, a pressure of approximately 0.2 – 0.3 bar (3 to 4.5 p.s.i.) is needed.

NOTICE

If the water pressure is too high, the tubing can be forced from the connectors within the spectrometer sample housing unit. Do not exceed an input water pressure of 1.7 bar (24 p.s.i.).

The circulating water flows through a heat exchanger which removes heat from the Peltier device. Insufficient flow will allow the temperature of the heat exchanger to rise, and if a certain cut-off value is exceeded, the temperature controller will automatically shut down.

Cooler circulating water can improve the performance of the temperature controller at low temperatures, whereas warmer water can improve performance at very high temperatures.

NOTICE The Peltier control loop operates most efficiently if the circulating water is a few degrees below the set temperature. Do not use warm or room temperature water when setting temperatures close to ambient, as this can result in instabilities

The circulating water connections are to the male Legris type fittings on the tubing at the rear of the SHU (Figure 2.7).

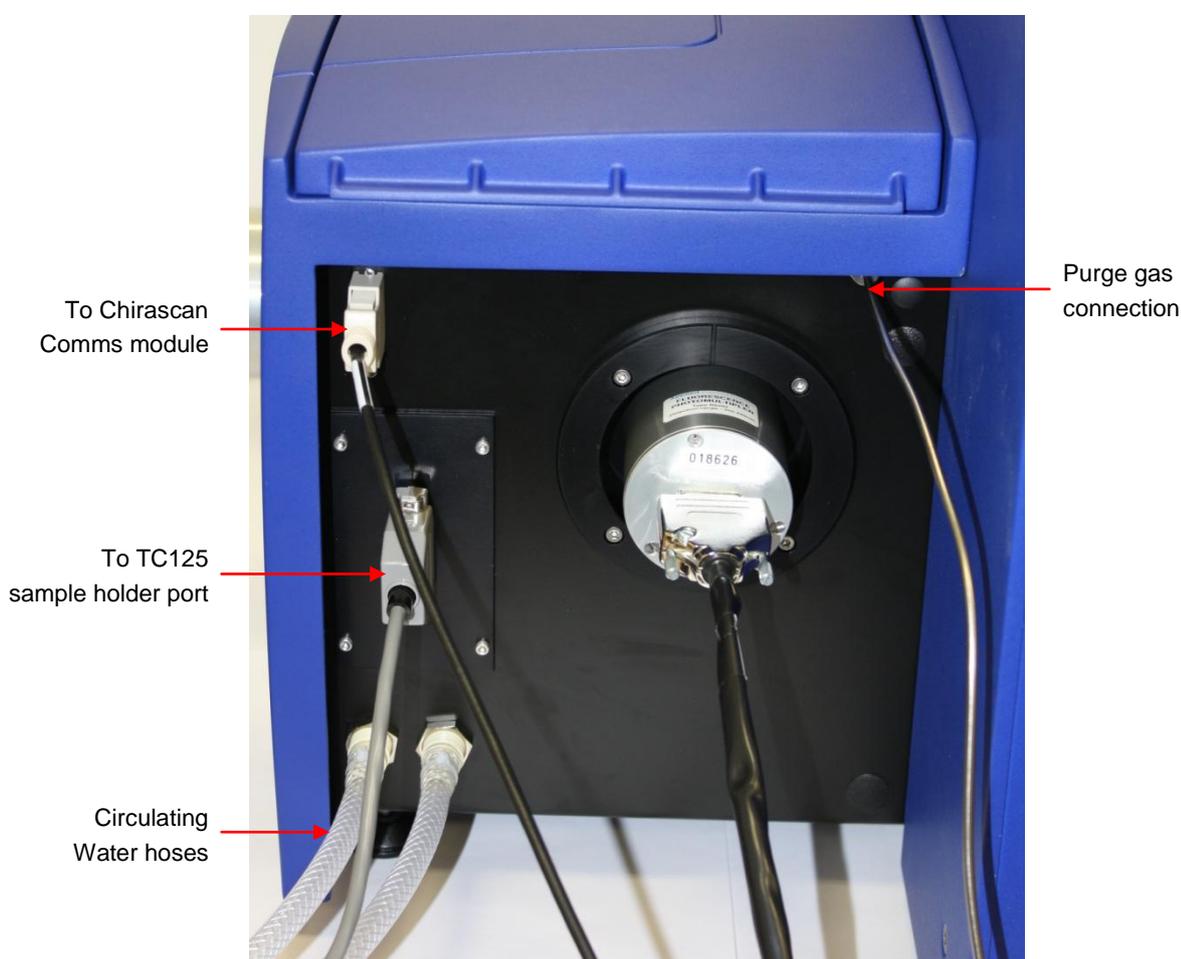


Figure 2.7: rear of the Chirascan Sample Handling Unit

2.2.3 Purge gas connection

The turret should be purged with oxygen free nitrogen at a flow rate of 2 litres per minute for at least 10 minutes before use (but note that the lamp housing and monochromator may require purging at higher flow rates for

longer periods: see the main Chirascan or Chirascan-plus manual for details). During operation the purge gas flow rate to the temperature control unit can be reduced to 1 litre per minute.

The purge gas connection is at the rear of the Chirascan Sample Handling Unit (Figure 2.7)

2.3 Software Installation

Do not connect the USB cable from the computer to the Temperature Controller until the drivers and software have been installed. If the CS/PCM has been supplied by APL with a Chirascan or Chirascan-plus spectrometer, then the QNW drivers will already have been installed. If it has been supplied separately, or if the user wishes to use their own computer, then the QNW drivers will need to be installed. Please contact a member of the APL Technical Support Department for information on driver installation.

2.4 Electrical connections



The TC425 is powered by the mains electricity supply which can produce a shock leading to serious injury or death. Do not connect or disconnect the instrument from the mains supply unless the supply is powered off at source. Ensure all communications and electrical connections are made before powering on the spectrometer. Exercise care during operation and do not operate units with their covers removed. Operate the accessory using only the cables provided. Never operate the accessory with damaged cables.

The rear of the TC425 electronics module is shown in Figure 2.8. The mains connection port connects to the mains electricity supply using the mains cable provided. The USB-B port connects to a USB port on the computer. The sample holder port connects to the 25-pin D port on the rear of the Chirascan Sample Handling Unit (Figure 2.7).



Figure 2.8: rear of the TC425



The turret and electronics module are calibrated as a pair; the electronics module should only be used with the turret indicated by the serial number on the rear.

2.5 Installing the temperature probes

The Chirascan SHU can accept up to four temperature probes, including the one supplied as standard with the CS/PCM. The probes connect to the block to the right rear of the sample handling unit (Figure 2.5). Note that the lowest connector on the block, marked **Stir** is no longer used, the next, marked **Lid**, is used for the lid open sensor, and the upper four, marked **T1** to **T4** from the lowest up, are used for temperature probes. Normally, if a single sensor is used, it connects to **T1**.

The standard probe, shown in Figure 2.9, is normally connected to T1. The probe diameter is 0.6 mm, and the probe is not suitable for use with cells with a pathlength of less than 1 mm. Please contact Applied Photophysics if a narrower diameter probe is needed. Normally all the samples on the CS/PCM are at close to the same temperature, and it is not necessary to use more than one temperature probe, but if the temperature of each sample needs to be known separately, please contact Applied Photophysics for additional probes.

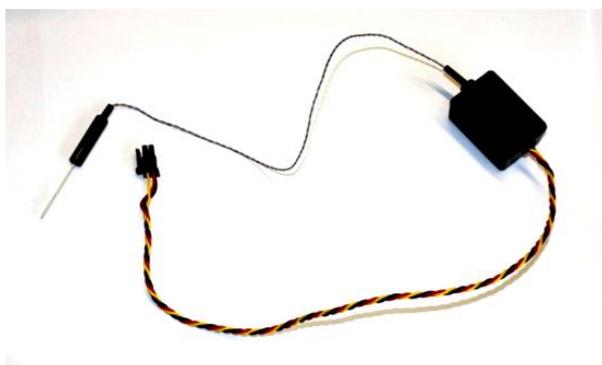


Figure 2.9: temperature probe supplied with the CS/PCM

2.6 Configuring the Chirascan

If the QNW drivers are installed, the connections made and the electronics module powered on, the Chirascan should automatically configure for the CS/PCM when the spectrometer is powered on. If this does not happen, on the **View** menu click **Devices** to display the **Device Window**, then double click on the circulator icon  to display the **Circulator or Peltier Temperature Control Unit** dialog box (Figure 2.10)

Select Quantum Northwest Peltier from the drop-down list, and click **Probe** to search for the computer USB port. When the port has been located, click the **Assign** button, and **Set as default** if required.

The temperature range of the Turret is also set on this dialog box: for more information see the Turret 400 Instruction Manual.

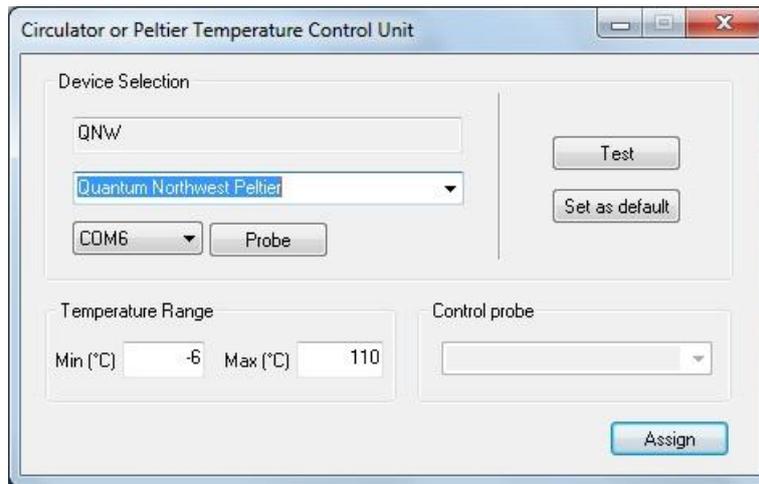


Figure 2.10: Circulator or Peltier Temperature Control Unit dialog box

3 OPERATION

A general description of the Pro-Data Chirascan software is given in the main Chirascan and Chirascan-plus User Manuals, and only those features specific to the CS/PCM are described here.

3.1 Using the electronics module front panel

Several functions of the CS/PCM can be controlled using the electronics module front panel, which also displays the current status of the TURRET. The electronics unit front panel is shown in Figure 3.1.



Figure 3.1: the CS/PCM electronics unit front panel

To set the target temperature for isothermal operation, use the **up/down** buttons to the right of the display window. To begin controlling the temperature, depress the **run/stop** button; depressing the button a second time will turn the temperature control off. When the TURRET temperature approaches the target temperature, the red indicator light will flash slowly. When the TURRET reaches and remains within $\pm 0.2^\circ\text{C}$ of the target temperature, the red light will remain constant.

The stirrer speed is set using the knob to the left of the display window, labelled **Off...Stir**. The speed is increased by turning the knob clockwise, but note that the stirrer is activated on the **Stirrer setup** dialog box (Section 3.3.1).

The cell positioning buttons can be used to set the turret position, so that the selected cell is positioned in the spectrometer light beam.

3.2 Using the QNW Auto-cell Changer... dialog box

To display the **QNW Auto-cell Changer...** dialog box (), on the **View** menu click **Devices** to display the **Device Window**, then double click on the QNW icon  to display the **QNW Auto-cell Changer....** dialog box (Figure 3.2).

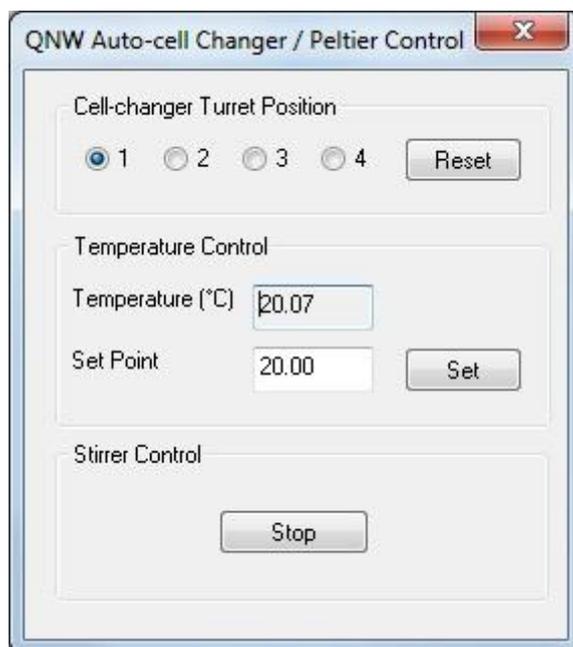


Figure 3.2: Circulator or Peltier Temperature Control Unit dialog box

The turret position can be set using the radio buttons **1** to **4**. If the turret position is lost it can be repositioned using the **Reset** button, although to avoid driving it against the end stop, this is best done manually.

To set the turret temperature, enter the target temperature in the **Set Point** box and click **Set**. To start or stop the stirrer, click the button in the **Stirrer Control** panel: the stirrer speed is set on the electronics module front panel (Section 3.1).

3.3 Sample Handling Unit (SHU) panel

The Sample Handling Unit panel (Figure 3.3) on the main Spectrometer Control Panel (SCP) displays the temperature read by the SHU probe selected from the drop-down list.



Figure 3.3: the Sample Handling Unit panel

3.3.1 Stirrer setup dialog box

Click the **Stirrer** button to display the **Stirrer setup** dialog box (Figure 3.4). The slider has only two positions, off (left) and on (right). The stirrer speed can only be set on the front panel of CS/PCM electronics module.



Figure 3.4: the stirrer setup dialog box

3.3.2 Multi-cell Sample Setup dialog box

Figure 3.5), where the number of cells to be used is entered. Tick the boxes for the required cell positions. Details of the samples and sample cells can also be entered; these are for reference only, they are stored with the data file, but are not otherwise used by the instrument software.

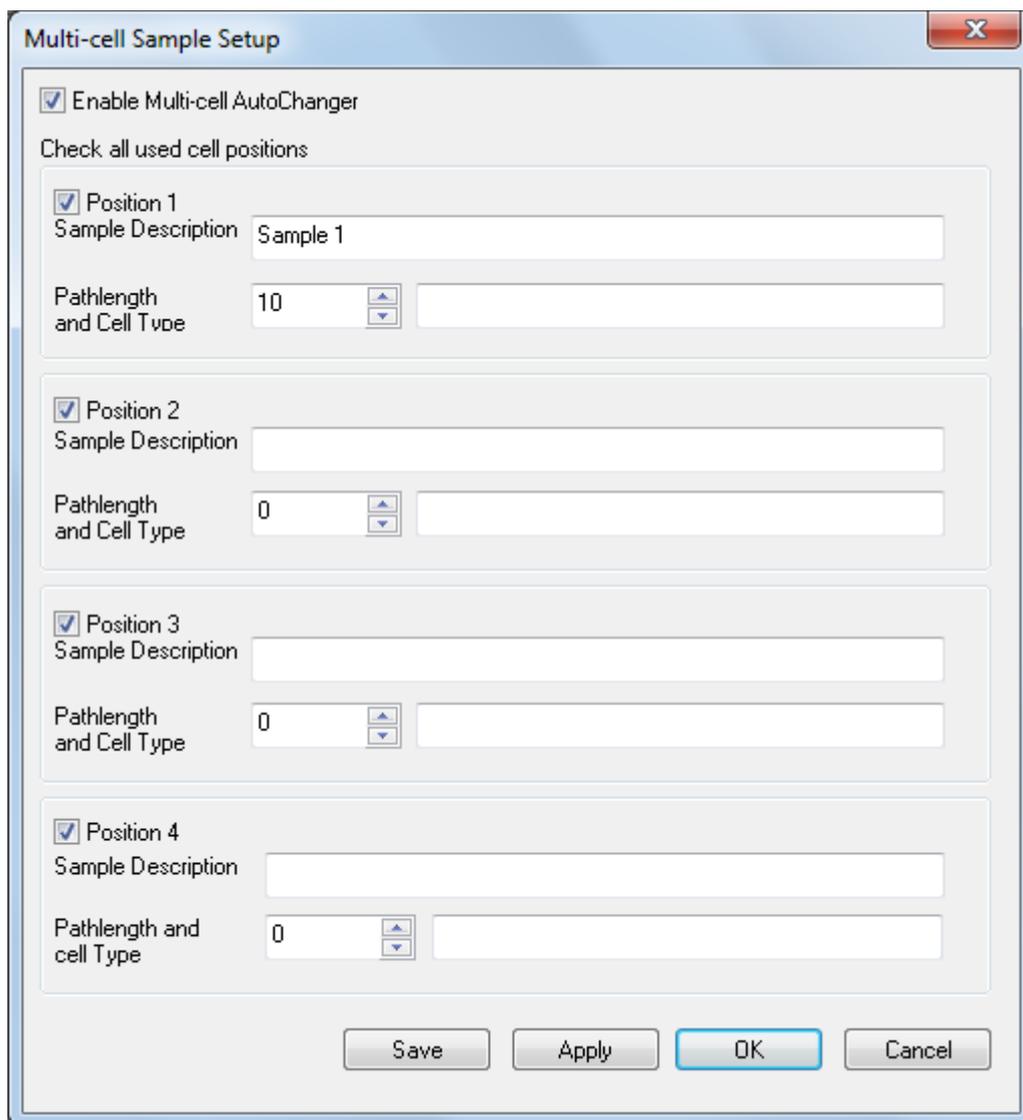


Figure 3.5: the Sample and Cell Description dialog box

3.4 Temperature Control Unit panel

The **Temperature Control Unit** panel (Figure 3.6) shows the status of the temperature control unit (i.e. the turret).



Figure 3.6: the Temperature Control Unit panel

The **Temperature** box displays the temperature read by the probe positioned in the turret. Click the **Settings** button to display the **Temperature Control** dialog box (Section 3.5).

3.5 Temperature Control dialog box

The **Temperature Control** dialog box (Figure 3.7) is used to set the temperature conditions for an experiment.

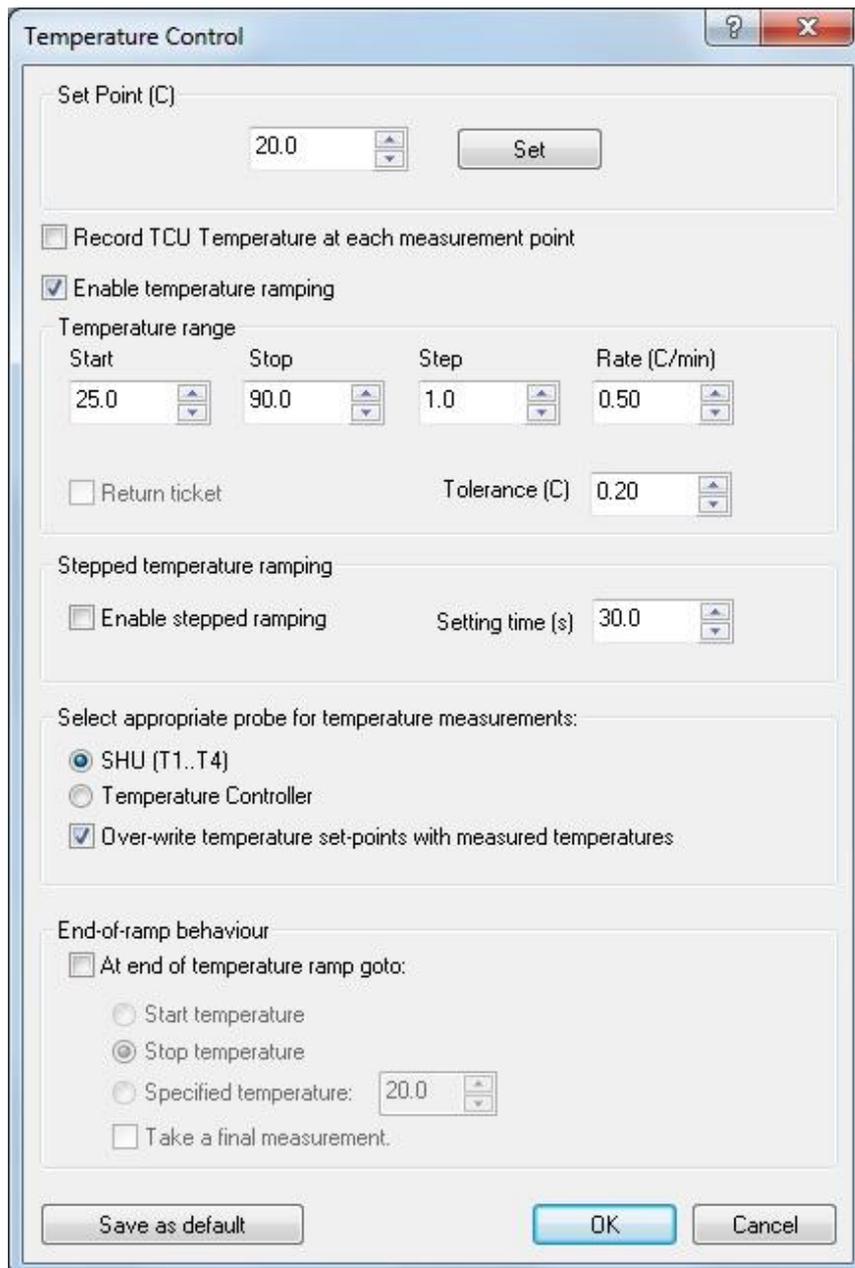


Figure 3.7: the Temperature control dialog box

3.5.1 Set Point panel

To set the temperature of the turret, enter the required temperature into the field on the **Set point** panel and click **Set**. The turret will immediately go to the set temperature.

3.5.2 Record temperature controller... tick box

During an experiment, the target temperature is recorded by default as an x-axis variable. If the **Record Temperature Controller temperature at each temperature** box is ticked, the temperature controller probe reading will also be recorded as a y-axis variable.

3.5.3 Enable temperature ramping tick box

To perform a constant temperature (isothermal) experiment, clear the **Enable temperature ramping** box.

To perform a non-isothermal experiment, tick the **Enable temperature ramping box**. Two types of temperature ramp can be performed, a *smooth* ramp, in which the temperature changes at a constant rate, and a *stepped* ramp, in which the temperature changes in a stepwise fashion.

A smooth temperature ramp is used, for example, to determine the transition temperatures of a protein in solution, usually at a fixed wavelength, or to perform a Dynamic Multimode Spectroscopy experiment on the Chirscan-plus (see the main Chirscan-plus User Manual). A stepped ramp is usually performed to record a series of spectra at set temperature intervals, for example to establish the nature of a transition.

Note that the smooth ramp is not normally used with the CS/PCM, because the operating sequence is for a ramp to be performed on each cell in turn, so for example if cells 1 and 2 are selected, the instrument will perform a full temperature ramp while making measurements on cell 1, before moving to cell 2 and repeating the temperature ramp. The sample in cell 2 will therefore have experienced a complete thermal cycle before measurements are made on it.

A stepped ramp is usually more suitable for the CS/PCM, because the operating sequence is for measurements to be made on each cell in turn at each set temperature.

3.5.4 Temperature range panel

The start and stop temperatures for the ramp are entered on this panel.

The **Step** box entry is the temperature increment between data acquisitions. For a stepped ramp, the temperature will be constant during the acquisition. For a smooth ramp, the temperature will continue to change in the background while the acquisition is made, in which case the step should be set to be *longer* than the acquisition time. For example if the ramp is conducted at a single wavelength, the time per point should be less than the time between steps.

The entry in the **Tolerance** box sets the range for data acquisition to begin for each step, for example if target temperature for a step is 30°C, and the tolerance is set to 0.2°C, data acquisition for that step will begin when the temperature is between 29.8 and 30.2°C.

The **Rate** box entry is the temperature ramp rate for a smooth ramp; for a stepped ramp the box is greyed out. The lower the ramp rate, the longer the experiment, but if the rate is set too high, a thermal lag between the temperature holder and the sample may result. A ramp rate of 1°C is often a good compromise; this also enables comparison with DSC experiments which are usually conducted at that rate.

If the **Return ticket** box is ticked, a second ramp will be performed in which the temperature returns to its start value. This is only available for a stepped ramp, for a smooth ramp the box will be greyed out.

3.5.5 Stepped temperature ramping panel

To perform a stepped ramp, tick the **Enable stepped ramping** box, for a smooth ramp the box should be cleared.

The **Setting time** is the time at each temperature before data acquisition begins, to allow the sample to equilibrate. For a smooth ramp this refers only to the start temperature.

3.5.6 Select appropriate probe... panel

During an experiment, the target temperature is recorded by default as an x-axis variable. The temperature of either the temperature controller or the SHU probes can also be recorded as y-axis variables using the radio buttons.

If the over-write temperature set-points is ticked, the target temperature will be over-written as an x-axis variable by the temperature selected by the radio buttons.

3.5.7 End-of-ramp behaviour panel

This box can be used to send the temperature to the start or stop or other specified temperature. For a stepped ramp, a final measurement will be taken at that temperature if the **Take a final measurement** box is ticked. For a smooth ramp, this box will be greyed out.

3.6 Sample loading

The CS/PCM accepts standard 10 x 10 mm internal dimension cells. APL recommend CD or CD/fluorescence cells from Hellma GmbH, Müllheim, Germany or Starna Group, Hainault, United Kingdom. Other cells sizes can be used, using spacers available from Hellma or Starna. For more information please contact these companies.



The CS/PCM may be very hot or cold, causing injury to the user when touched. Ensure that it has been allowed to reach a safe temperature before handling.

To avoid spillages, the cell is best removed from the instrument before loading. Most samples can be pipetted into the cell. Make sure that there is enough sample in the cell to cover the window in the turret, and that there are no bubbles in the sample. If the sample is to be run at high temperatures, it may be necessary to de-gas before running to prevent bubble formation.

The cell should then insert easily into the turret, and is sprung against the face of the turret to ensure reproducible positioning. Always record the orientation of the cell so that it can be replaced in the same orientation; most cells are inscribed with identification marks.

If using a temperature probe, ensure that the probe is immersed in the sample to a depth of at least 2 mm, but is not intruding on the light beam. If not using a temperature probe, place the turret cap on the turret (Figure [3.8](#))

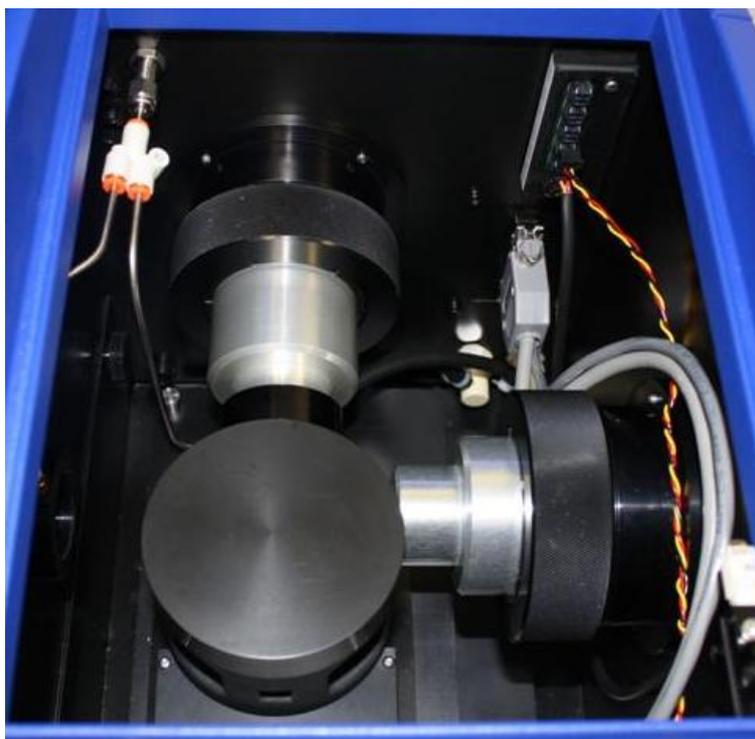


Figure 3.8: the turret cap in place on the turret

Finally, close the lid on the sample handling unit and run the experiment as described in the main Chirascan or Chirascan-plus User Manual.

3.6.1 Removing the cell from the turret

On completion of the experiment, the cell can be removed from the holder for cleaning and re-use.



The sample cell and turret may be very hot or cold, causing injury to the user when touched. Ensure that they have been allowed to reach a safe temperature before handling.