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Web Page: <b><a href="http://www.qnw.com/NeutroniQ-product-family/">http://www.qnw.com/NeutroniQ-product-family/</a></b>			
Description: <b>Peltier-based temperature-controlled, 9-position holder for use for neutron scattering, with extended temperature control</b>			

# QNW NeutroniQ 3x3 User's Guide

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A CAUTION notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly performed or adhered to, could result in damage to the product or loss of important data. Do not proceed beyond a CAUTION notice until the indicated conditions are fully understood and met.

#### WARNING

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or the like that, if not correctly 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.

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# 1. General Information

Thank you for purchasing a Quantum Northwest **NeutronIQ 3x3**. We want you to enjoy many years of faithful service from your instrument. If you have any questions, feel free to contact us directly through our website, [www.qnw.com/contact-us/](http://www.qnw.com/contact-us/), or by email at [service@qnw.com](mailto:service@qnw.com).

Your Quantum Northwest cuvette holder and accessories have been carefully designed so that when used properly, you have an accurate, fast, flexible and safe temperature control system.

Information about safety practices appears throughout the documentation provided with your instrument and accessories to help you safely operate the instrument and accessories. Before using the instrument or accessories, you must thoroughly read these safety practices. ALWAYS operate the instrument and accessories in accordance with these safety practices.

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## 1.1. User Documentation

You have been provided with the following documentation to help you set up and operate your Quantum Northwest **NeutronIQ 3x3** instrument:

- This manual, with safety practices and hazards information, instructions for installing and maintaining the components of the **NeutronIQ 3x3**, and troubleshooting information.
- If **T-App** is included, the T-App Help file, located on the T-App storage media ("T-App Help.chm" file in the T-App directory).

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## 1.2. Modules, Covers and Panels

Operators and other personnel will routinely access the QNW **NeutronIQ 3x3** sample compartment by removing the pairs of knurled screws that attach the lids. Customers may insert samples and sample accessories into the QNW **NeutronIQ 3x3** while the sample compartment is open.

Any other panels or covers that are retained by screws on the sample compartment may be opened **ONLY** by Quantum Northwest service technicians.

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## 1.3. Other Precautions

Do not block any ventilation grills present on the QNW **TC 1** Temperature Controller.




Use of the QNW **NeutronIQ 3x3** system and accessories may involve materials, solvents and solutions that are flammable, corrosive, toxic or otherwise hazardous. Careless, improper, or unskilled use of such materials, solvents and solutions can create explosion hazards, fire hazards, toxicity and other hazards which can result in death, serious personal injury, and damage to equipment and property.

ALWAYS ensure that laboratory safety practices governing the use, handling and disposal of such materials are strictly observed. These safety practices should include the wearing of appropriate safety clothing and safety glasses.

## 1.4. Warning and Other Symbols

The following is a list of symbols that appear in conjunction with warnings on the QNW cuvette holder and accessories. The hazard they describe is also shown.

A triangular symbol indicates a warning. The meanings of the symbols that may appear alongside warnings in the documentation or on the instrument itself are as follows.

	<p>When used on a warning label, the user is in danger of an electrical shock.</p>
	<p>When used on warning labels attached to the instrument, refer to the relevant operation or service manual for the correct procedure referred to by that warning label.</p>
	<p>When attached to the rear of the instrument, indicates that the product complies with the requirements of one or more EU directives.</p>

## 1.5. CE Compliance

Your QNW **NeutronIQ 3x3** cuvette holder has been designed to comply with the requirements of the Electromagnetic Compatibility (EMC) Directive and the Low Voltage (electrical safety) Directive (commonly referred to as the LVD) of the European Union. Quantum Northwest has confirmed that each product complies with the relevant Directives by testing a prototype against the prescribed EN (European Norm) standards.

Proof that a product complies with these directives is indicated by:

- the CE Marking appearing on the rear of the product, and
- the following Declaration of Conformity. The Declaration of Conformity is the legal declaration by Quantum Northwest that the product complies with the directives listed above, and shows the EN standards to which the product was tested to demonstrate compliance.

## EC Declaration of Conformity

We: Quantum Northwest, Inc.  
Of: 22910 E Appleway Ave, Suite 4  
Liberty Lake, WA 99019-8606  
USA

Declare under our sole responsibility that the products:

Models: **NeutroniQ 3x3, TC 1,**

Types: Temperature Controller, Temperature-Controlled Sample Compartment

Are in conformity with the following standards and relevant EC directives:

Emissions: EN 61326-1 (2013)

Immunity: EN 61326-1 (2013)

EU Directive: 2004/108/EC, EMC

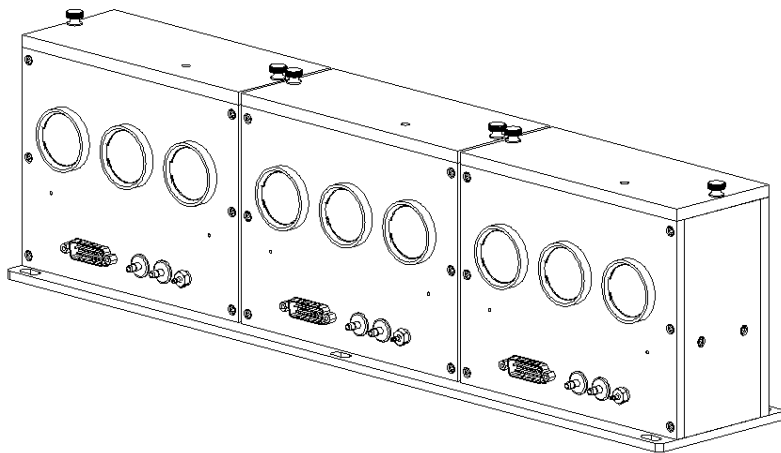
I hereby declare that the equipment models named above have been assessed and found to comply with the relevant sections of the above-referenced specifications. The units comply with the relevant requirements of the applicable Legislation, and I am the person authorized to compile the technical documentation.

Signed by:



Name: Enoch W. Small, Ph.D.  
Position: President, Quantum Northwest, Inc.  
Date: August 22, 2014  
Location: Liberty Lake, Washington, USA

## 2. Specifications



**Figure 1.** NeutronIQ 3x3 temperature controlled sample compartment.



**Figure 2.** TC 1 Temperature Controller

### 2.1. Introduction: QNW NeutronIQ 3x3 Cuvette Holder with TC 1 Temperature Controller

#### Intended Use

The QNW **NeutronIQ 3x3** Sample Compartments are designed for use in neutron scattering measurements. Each of the three compartments of the **NeutronIQ 3x3** has positions for three cuvette carriers aligned with fused silica optical ports (for neutron beam entrance and exit). The temperatures of the compartments are independently controlled by three **TC 1** Temperature Controllers during measurements. Each **TC 1** can be controlled using front panel controls or by using the QNW **T-App** software (or other program) on a computer interfaced to the temperature controllers. Each **TC 1** will require its own **T-App** application window.

#### Specifications

- |                               |  |
|-------------------------------|--|
| • Temperature range           | -50 to +150 °C*  |
| • Temperature precision       | ±0.05 °C   |
| • Temperature accuracy        | better than ± 0.3 °C (-25 to 100 °C)<br>better than ± 0.5 °C (>100 °C) |
| • Temperature reproducibility | better than ± 0.2 °C   |
| • Optical port dimensions     | input 25.4 mm, exit 30.7 mm diameter                                   |
| • Probes accepted             | one Series 400 or Series 500 thermistor                                |
| • Sample Holder z-height      | 21.6 mm  |
| • Optical Centerline height   | 82.6 mm  |

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**Note:**

A cell holder (compartment) temperature of -25 °C can be attained using ice water.

Temperature precision, accuracy and reproducibility values are measured with the RTD probe in the probe hole of the compartment.

Work below -25 °C requires pre-cooled circulating fluid with a temperature (entering the sample holder) less than 20 °C above the required sample holder temperature.

The “z-height” is the distance between the floor of the sample compartment and the centres of the input and exit windows.

The Optical Centerline height is the distance between the bottom (of the metal mounting plate) and the centers of the windows.

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## 2.2. Measurement Category

The measurement category is IEC 61010:I. Do not to use this equipment for measurements within measurement categories II, III and IV.

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## 2.3. Pollution Degree

The pollution degree is IEC61010:2. Pollution degree “2” applies to a normal indoor atmosphere.

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## 2.4. Overvoltage Category

The overvoltage category (installation category) is CAT II. See IEC 664 & IEC 61010.

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## 2.5. Environmental Conditions

The area should have a dust-free atmosphere with minimal drafts, vibrations, and corrosive fumes. For optimum performance, the ambient air is recommended to be between 20 and 25 °C, but can be from 5 to 40 °C. Relative humidity should be less than 80%. The instrument is designed for operation at 2,000 m elevation or less.

To avoid damage through spillage of solutions and samples being analyzed, the worktops should be covered with a material that is corrosion resistant and impervious to liquids.

For the **TC 1** temperature controllers, allow at least two inches of space on both sides, and six inches at the rear to permit free air circulation. The power switch and all connectors are located on the rear panel.



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## 2.6. Electrical Specifications

### Mains Supply

A standard power cable is provided based on the user's country requirements (18 AWG/115 V AC US/Canada; 1.0 mm/220 V AC international). The required supply voltage is 100-240 V AC (frequency 50 or 60 Hz). The mains voltage fluctuations must not exceed 10% of the nominal voltage.

The installation of electrical power supplies must comply with the rules and/or regulations imposed by local authorities responsible for the supply of electrical energy to the workplace.

All power supplies for the **TC 1** Temperature Controller must be single-phase, AC voltage, three-wire system (active, neutral, earth) and should be terminated at an appropriate power outlet receptacle that is within reach of the power cord. For safety reasons, a separate power outlet receptacle should be provided for each unit in the system. The use of extension cords or outlet adaptors is not recommended.

### WARNING



**Shock Hazard - Danger of electrocution.** Good electrical grounding is essential to avoid potentially serious shock hazards. A 3-wire outlet with ground connection must be provided for the **NeutronIQ 3x3**. Make certain that power outlets are earth-grounded at the grounding pin.

### CAUTION



**Caution** - Never connect or disconnect any cables while the **NeutronIQ 3x3/TC 1** Temperature Controller is switched on. Damage to the printed circuit boards may occur.

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## 2.7. Computer Requirements

A computer is not required because all essential functions can be controlled from the front panel of the **TC 1** temperature controller.

QNW's **T-App** software can be used for computer control of the **TC 1**; **T-App** requires a computer running Microsoft Windows™ (XP, Vista, 7, 8 or 10). **T-App** software is provided on a USB flash drive.

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## 3. Installing, Starting and Stopping the QNW NeutronIQ 3x3

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### 3.1. Installing the QNW NeutronIQ 3x3

#### **Unpacking**

After accepting delivery, take the equipment to the installation site. Quantum Northwest instruments are inherently robust, and the packaging is designed to prevent internal damage. However, the contents form part of a precision measuring system and all packages should be handled with care. In transit, sharp jolts must be avoided and the packages should not be inverted or tilted unnecessarily. Markings on the shipping cartons generally indicate which side of the package should be kept on top.

Unpacking the equipment is your responsibility. As the packages are opened, ensure that you have received everything you ordered. If there are any discrepancies, notify the supplier. If any items are found to be damaged, immediately notify the carrier and supplier.

You should have received:

- 1x **NeutronIQ 3x3** Sample Holder
- 3x **TC 1** Temperature Controller
- 3x DB15 cable to connect **TC 1** to sample holder unit
- 3x submersible pump
- 3x bucket for submersible pump
- 3x AC power cable
- 3x USB cable
- 3x 1/8-inch inside diameter vinyl tubing to connect water
- 3x 1/16-inch inside diameter vinyl tubing to connect dry gas
- This manual

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**Note:** Any additional accessories ordered are not listed here.

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Any differences from the original order should be referred immediately to your Quantum Northwest sales office. Do not discard any packaging components or filler materials.

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**Installing the QNW T-App Software (if included)**

1. Follow the instructions provided with the software to install drivers required for serial communication between **T-App** the **TC 1** temperature controller.

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**Note:** It is important to install drivers before connecting the **TC 1** to your computer. If the **TC 1** is connected to the computer and turned on before driver installation, the computer may automatically load an incorrect driver that may be difficult to remove.

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2. Connect the **TC 1** to your computer using the USB cable provided. The New Hardware installation process will begin automatically and take a few moments to finish.



**Figure 3.** Submersible pump with electrical cable, water bucket and vinyl tubing for the **NeutroniQ 3x3** (one of three each).

**Installing the QNW NeutroniQ 3x3 Hardware**

1. Position the **NeutroniQ 3x3** sample holder in the location where it will be used and secure it in place..
2. Stack the three **TC 1** temperature controllers in a convenient location relative to the sample holder.
3. Use the three gray DB15 cables to connect the three **TC 1** temperature controllers to the three connectors on the **NeutroniQ 3x3**. Connect A to A, B to B and C to C. The three (**TC 1**) / (sample holder section) pairs **are not interchangeable**; if they are misconnected the temperature calibrations will not be correct. If the labels on the sample holder are "lost", A is on the left side of the sample holder (as viewed from the connector side – see Figure 4).
4. Position the water buckets within 75 cm (30 in) above or below the sample holder (or use a larger container as a source of cooling water to all three sample holder sections).

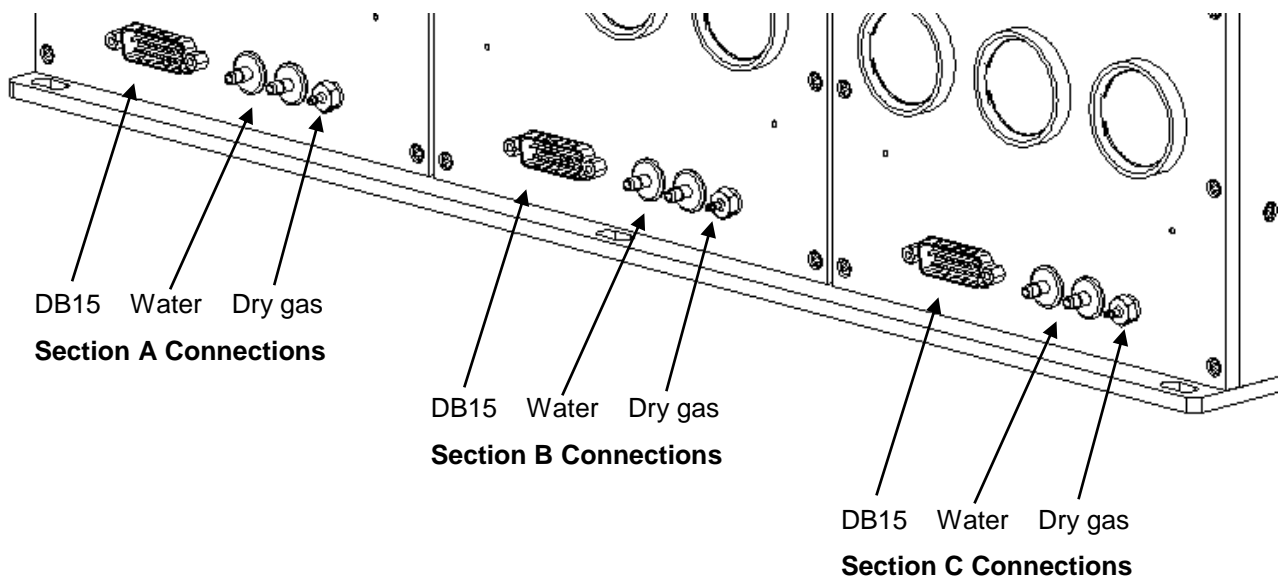
**CAUTION**


**Caution** – Position the bucket in a location that minimizes the risk of spilling the liquid contents on sensitive components.

5. OPTIONAL - If a dry gas such as nitrogen is used to reduce condensation or frost formation on the windows and the cuvette surfaces, secure the tank to a solid object in the vicinity of the **NeutroniQ 3x3**. Connect the gas source to the three 1/16 inch connectors on the sample holder sections using the 1/16 inch (ID) tubing supplied.

**CAUTION**


**Caution** – When using dry gas, ensure that the working area is adequately ventilated.



**Figure 4. NeutroniQ 3x3 utility connections.**

6. Cut the  $\frac{1}{4}$ -inch vinyl tubing provided into six lengths. Attach lengths of tubing to the hose barbs of the three submersible pumps, shown in Fig. 3, and the other end to either one of the water hose barbs on each section of the **NeutroniQ 3x3**, labeled "Water" in Fig. 4. Attach another piece of tubing from the other hose barbs with the free ends returning to the water bucket(s). Add water to the bucket(s); stir in a small quantity of liquid dish detergent (one drop per liter, to reduce surface tension) and household bleach (0.5 ml/ liter, to prevent bacterial and algae growth). Lower the pumps into the water. Using the electrical cable from each pump, connect it to the socket on the back of the corresponding (A, B, or C) **TC 1** Temperature Controller. The pump will start automatically when the **TC 1** is operated later.
7. Plug the AC power cables into the back of the **TC 1** Temperature Controllers and into a source of AC voltage.

**CAUTION**


**Caution** - Never connect or disconnect any cables between the s **TC 1** Temperature Controllers while it is powered on. Damage to the printed circuit boards may occur.

## 3.2. Starting the QNW NeutroniQ 3x3

1. Remove the knurled screws and the sample holder lids.
2. Place the cuvette holder assemblies (may not have been provided by QNW) and cuvettes in the **NeutroniQ 3x3**.
3. Replace the sample holder lids.
4. OPTIONAL – If you wish to monitor the temperature in a sample (with the lid off), plug a standard Series 400 or Series 500 thermistor probe (not provided unless ordered) into the ¼ - inch phone jack labeled “probe” in the back panel of the **TC 1**. Place the end of the probe in a region of the sample where it will not interfere with the neutron beam.
5. Turn each **TC 1** controller on using the switch on the back panel.
6. Check that water is flowing from each of the free tubing ends. If it does not, lift the corresponding pump off the bottom of the bucket, but not out of the water, and drop it. This usually will get the flow started. If it does not after several tries, lift the pump up close to the water surface and let a small drop of liquid detergent land in the water close to the pump intake.
7. Check for water leaks.



**Figure 5. TC 1 Menu Button**

8. Use the left or right arrows on the Menu Button, shown in Fig. 5, to cycle through the four pages of options. These options are described in detail in Section 4.3 below. Go to the Set Target Temp page and use the up and down arrows to set a target temperature, for example, 37.0 °C. Press SET. These steps turn temperature control on (and turn the corresponding pump on). The temperature will then change and stabilize. The **NeutroniQ 3x3** does not have magnetic stirring, so changes made on the Stirring page will have no effect.

## 3.3. Stopping the QNW NeutroniQ 3x3

Turn off power on the back of the **TC 1** controller (it is not necessary to turn temperature control off before this step). This action will also turn off the **BATH 10** pump if it was on. If you are using a circulator pump other than the **BATH 10**, you will need to unplug it or switch off the power source.

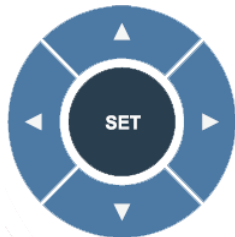
## 4. Step-by-Step Instructions for Using the QNW NeutronIQ 3x3

### 4.1. Detailed Instructions

#### Computer Control using the QNW T-App program

**T-App** is a program that permits external computer control of the **TC 1** Temperature Controller. The temperature of the sample holder and a temperature sensed by an external probe may be plotted vs time. Simple text scripts may be used to automate multiple operations. Full instructions for using **T-App** may be found in the associated Help file.

#### Using the front panel Menu Control Buttons



**Figure 6.** The Menu Control Buttons (**SET**, **left arrow**, **right arrow**, **up arrow**, and **down arrow**) on the front panel of the **TC 1** controller allow full functionality without an external control program.

The Menu Control Buttons allow control of temperature and ramping for the corresponding **NeutronIQ 3x3** section (A, B, or C)

The relationships between the different front panel display screens are summarized in **Figure 7**. The text in the display screens in **Figure 7** is that which will be seen immediately after the **TC 1** power is turned on.

Right after the power is turned on, temperature control will be off with the target temperature (TT) set to 20 °C (power on default). Ramping will be off with the ramp rate set to 0.50 °C/min.

#### Using the TC 1 Temperature Controller

When first powered on, the **QNW COPYRIGHT** display will appear for a short time before it is replaced by the **MAIN** display.

```
Holder = 22.9°C SAM
Target = 20.0°C
Probe = 23.0°C
Ramp Off Stir Off
```

```
Holder = 22.9°C SAM
Target = 20.0°C LOK
Probe = 23.0°C
Ramp Off Stir Off
```

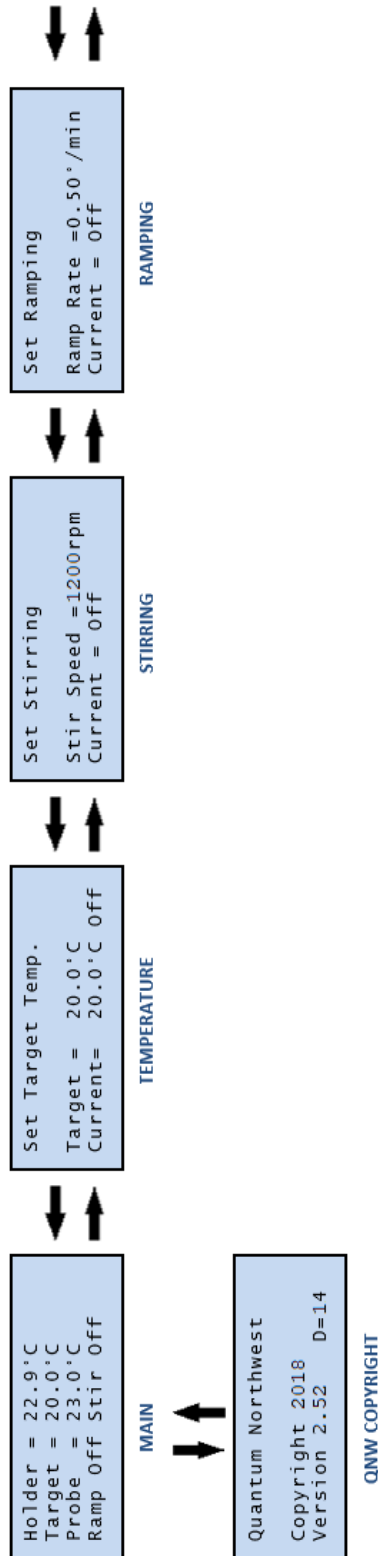
```
Holder = 22.9°C SAM
Target = 20.0°C LKK
Probe = 23.0°C
Ramp Off Stir Off
```

An external control program can set the controller to locked mode, which will cause the text “LOK” to appear in the **SAMPLE** and **REFERENCE** displays (middle above). In locked mode, the user is locked out of the **UNLINK/RELINK** display. Also, the controls for changing settings in both the sample and the reference displays (**up arrow**, **down arrow**, **SET**) will have no effect.

The external control program can also determine whether the controller is in linked mode and can set it to be in both linked mode and locked mode. In this case, the text in the sample and reference displays will be “LKK” (illustrated by the display on the right above).

**Figure 7. Access relationships between the front panel display screens for a Quantum Northwest NeutronIQ 3x3/TC 1 Temperature Controller**

The black arrows represent the arrow buttons on the Menu Button. The pairs of arrows at the far right indicate that the arrow buttons may be used to move directly between the far-right display and the far-left display.



The **MAIN** display persists until one of the arrow buttons is pressed.

The **QNW COPYRIGHT** display persists for a short time to allow viewing.

For the remaining displays shown horizontally, the up/down arrow buttons are used to change the adjustable setting ("Target," "Stir Speed" or "Ramp Rate") while the **SET** button is used to lock in the new value and turn on temperature control, stirring or ramping. When **SET** is pressed, the display immediately reverts to the **MAIN** display. If the "Current" setting is identical to the adjustable setting, then pressing the **SET** button turns temperature control, stirring or ramping off and the display immediately changes back to **MAIN**. If a change is made in the "Target," "Stir Speed" or "Ramp Rate" setting, and the **SET** button is not pressed before the display automatically reverts to **MAIN**, the change made will still be there when you return to that display.

After the **TC 1** has been turned on, the **MAIN** display will change depending on the current settings. Several combinations are illustrated below.

```
Holder = 22.9°C
Target = 20.0°C

Ramp Off Stir Off
```

No probe, ramping off,  
stirring off.

```
Holder = 36.9°C
Target = 37.0°C
Probe = 36.2°C
Ramp wTT Stir Off
```

With probe, ramping in "wait  
for new TT" mode, stirring off.

```
Holder = 73.4°C
Target = 100.0°C
Probe = 98.7°C
Ramp On Stir On
```

With probe, actively ramping,  
stirring on.

The "Holder = " line gives the current temperature of the cuvette holder.

The "Target = " line gives the current target temperature (TT) setting. When temperature control is on, the holder temperature will be converging on the TT, and the green LED near the upper right corner of the display will be blinking slowly. When the holder temperature reaches the TT and has stayed within a range of  $TT \pm 0.05\text{ }^{\circ}\text{C}$  for 1 minute, the green LED will stop blinking and stay on.

The "Probe = " line is the temperature registered by an optional series 400 or 500 thermistor probe plugged into the external probe socket in the back of the **TC 1**.

### Examples of using the Menu Buttons for control of the TC 1

In the default mode (after power is first turned on), the **TC 1** is in linked mode and both sample and reference parameters are identical:

- temperature control is off with the "Target" parameter set to 20 °C
- stirring is off with the "Stir Speed" parameter set to 1200 rpm
- ramping is off with the "Ramp Rate" parameter set to 0.50 °C

When you turn the **TC 1** on, the **MAIN** page is displayed (shown to the right).

In addition to the current sample holder temperature, the current target temperature and the probe temperature (if an external thermistor probe is being used), the display also shows the state of the ramping function and whether stirring is on or off (the **SNB 3x2** does not include magnetic stirring capability).

```
Holder = 22.9°C
Target = 20.0°C
Probe = 23.0°C
Ramp Off Stir Off
```

### Example: Temperature Control

**Figure 8.** Setting the target temperature and turning on temperature control.

```
Set Target Temp.
Target = 20.0°C
Current = 20.0°C Off
```

initial display

```
Set Target Temp.
Target = 37.0°C
Current = 20.0°C Off
```

after adjustment

```
Set Target Temp.
Target = 37.0°C
Current = 37.0°C
```

return display

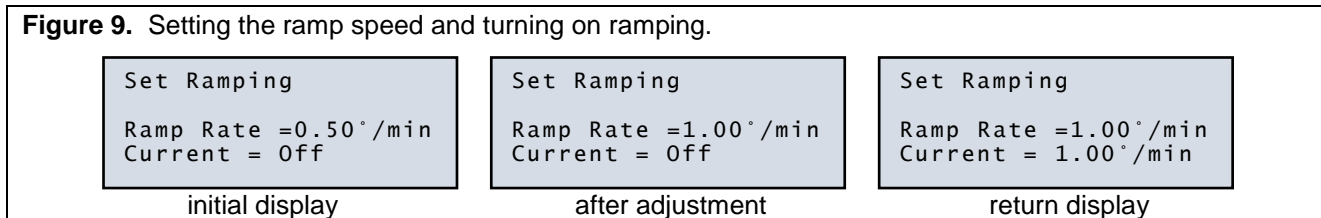
Press the **right arrow** to go to the **TEMPERATURE** display, which will look like the "initial display" in **Figure 8**. Then use the **up arrow** and **down arrow** buttons to adjust the "Target" parameter to 37 °C and the display will be equivalent to the "after adjustment" example in the **Figure 8**. Press **SET** to begin temperature control (and immediately return to the **MAIN** page). The green light on the **TC 1** will begin flashing slowly and the temperature of the sample cuvette holder will begin to increasing toward the new target temperature. The



“return display” in **Figure 8** shows what the display would look like if you now press the **right arrow** to go back to the **TEMPERATURE** display.

**Example: Setting up for ramping**

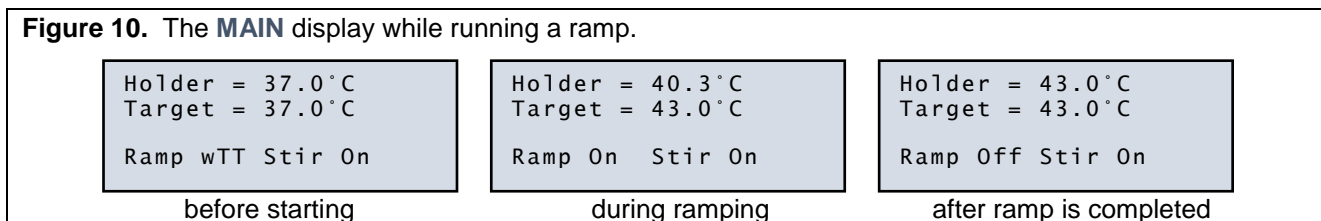
**Figure 9.** Setting the ramp speed and turning on ramping.



Press the **left arrow** (or press the **right arrow** three times) to go to the **RAMPING** display, which will look like the “initial display” in **Figure 9**. Then use the **up arrow** and **down arrow** buttons to adjust the “Ramp Rate” parameter to 1.00 °C/min (1.00 °/min in the display). The display will be equivalent to the “after adjustment” example in **Figure 9**. Then press **SET** to turn ramping on. The display will immediately return to the **MAIN** display with “Ramp wTT” at the bottom. The “wTT” indicates that the **TC 1** is now waiting for a new target temperature to be set to start the ramping process. The “return display” in **Figure 9** shows what the **RAMPING** page would look like if you return to it.

If you have been performing the steps for this example, the Sample Holder will be stable at 37 °C (indicated by the LED being on but not blinking) and the **MAIN** display will look like “before starting” in **Figure 10**.

**Figure 10.** The **MAIN** display while running a ramp.



To start the ramping process, press the **right arrow** to open the **TEMPERATURE** display and use the **up arrow** and **down arrow** buttons to adjust the “Target” parameter to 43 °C. Then press the **SET** button to start the ramping process. The LED will begin blinking and the **MAIN** display will look like “during ramping” in **Figure 10**. The ramp will take 6 minutes to complete. When the “Target” is reached, the display will look like “after ramp is completed” in **Figure 10**. The “Holder” temperature may go a little above the “Target” before settling back.

The maximum ramp rate setting is 10 °C/min. However, the maximum rate that can actually be achieved is determined by how fast the cuvette holder can change without ramping. The maximum ramp rate will be much lower going down in temperature compared to going up.

For complex control of the sample temperature, we recommend the use of software. For example, the program **T-App** from Quantum Northwest permits the use of scripts that can greatly simplify temperature ramping and other processes that require multiple commands.

---

## 4.2. Cooling Water for the Peltier

The Peltier element (or “thermoelectric cooler”) is a heat pump. When cooling, it transfers heat from the cuvette tower to the heat exchanger; when heating, electrical polarity is reversed and it transfers heat from the heat exchanger to the tower. When cooling, it is particularly important to transfer this heat away from the Peltier unit. This is accomplished by flowing water through the heat exchanger.

Water (or other cooling fluid) must be flowing through the heat exchangers via the  $\frac{1}{4}$ -inch hose barbs on the side of the **NeutronIQ 3x3**. The QNW **BATH 10** includes a submersible pump, the appropriate fittings for connecting tubing, and a plastic bucket. Connect the pump to the **NeutronIQ 3x3**, place it in the bucket with water, and run a return tube to the bucket. The Peltier unit requires a flow of 100 to 300 mL/min. This flow should require a pressure of about 3 - 5 psi (0.2 - 0.3 bar).

Cooling water may be provided from another source, such as a refrigerated cooling bath for holder temperatures below -15 °C; the water temperature entering the heat exchanger, *ist* If tap water is used keep the pressure below 25 psi or leakage at hose connections (internal as well as external) may result.

The submersible pump included with the QNW **BATH 10** will provide an adequate water flow to attain -15 °C (using ice water) and 0 to 80 °C (using water with temperatures of 25 to 30 °C. For temperatures above 80 °C, see section 4.6 below.

### CAUTION



**Caution** - Do not exceed an input water pressure of 25 psi (1.7 bar), as damage may occur inside the **NeutronIQ 3x3**.

---

**Note:** The heat exchanger and hose barbs are brass, and the tubing inside the **NeutronIQ 3x3** is vinyl. Be sure that any circulating fluid used, other than water, will not corrode these materials.

---

The temperature of the heat exchanger in the **NeutronIQ 3x3** is monitored by the **TC 1**. If this temperature exceeds 82 °C for target temperatures below 80 °C, then temperature control is shut down to prevent damage to the Peltier element and the warning, “check coolant flow,” is displayed on the **TC 1** temperature controller. This will happen if the circulating fluid gets too warm and/or is restricted in flow. The heat exchanger temperature can be as much as 20 °C higher than the coolant temperature entering it, particularly when attempting to attain very low temperatures. The heat exchanger temperature may be accessed by computer through the RS 232 or USB connections on the back of the **TC 1** Temperature Controller.

Temperature increases will be faster when room temperature water is used in the circulator. Temperature decreases will be faster when ice water is used. Only water should be circulated using the **BATH 10**.

---

### 4.3. Dry Gas to Minimize Condensation

Dry gas can be directed into the **SNB 3x2** sample holder via the hose barb labeled “gas” in the base of the each sample holder. Use a flow of at least 100 cc/min into each chamber and allow adequate time for the gas to adequately reduce the humidity inside.

---

**Note:** A flow of dry gas is necessary any time the **SNB 3x2** is controlled below the dew point temperature. For ambient air, this would typically be about 5 °C.

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### 4.4. External Temperature Probe

A ¼-inch phone jack labeled “Probe” can be found on the back panel of **TC 1** Temperature Controller. This jack will accept the plug on a standard Series 400 or Series 500 thermistor probe. When a probe is plugged into the jack, the probe temperature is presented on the display of the **TC 1**. Place the probe in the sample to measure the actual temperature of the sample, which will lag in time from the temperature of the cuvette tower.

---

### 4.5. External Computer Control

All functions may be accessed either through a Serial (RS 232) or a USB located on the back of **TC 1** Temperature Controller. You may write your own program or purchase the QNW application program **T-App**. **T-App** will plot temperatures of the probe, cuvette tower, and even the Peltier element heat exchanger vs time. It will also permit you to set up temperature ramps. If you wish to do your own programming, please see the Appendix for communication instructions and the set of text commands that may be used and their responses to the commands.

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### 4.6. Working at Extended Temperatures

#### **Operation at High Temperatures**

The state of the pump (on/off) provided with the **Bath 10** is controlled by the **TC 1** and is normally off unless temperature control is on.

If the **TC 1** firmware version is 2.51 or earlier, for target temperatures (TT) above 80 °C the **TC 1** will turn the pump off when the temperature exceeds 80 °C. Temperature control can stay on as long as the next TT set is greater than the current sample holder temperature. To change from a higher temperature (such as 115 °C) to any lower setting, the user must turn temperature control off and allowed the sample holder to cool passively until it drifts below the TT. Then temperature control can be turned back on. If the TT is below 80 °C, when the sample holder temperature drops below 80 °C the pump will be turned back on and temperature control can be turned back on and normal operation begins, the holder temperature will decrease rapidly and the new TT will be reached quickly.

If the **TC 1** firmware version is 2.52 or later, the controller handles all of this automatically (no user intervention is required).

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### **Operation at Low Temperatures**

While the **NeutronIQ 3x3** system is capable of operation as low as -50 °C, the minimum useful temperature depends on the conditions under which it is used.

Without special conditions, using room temperature coolant, the minimum is the dew point temperature of the air at the location of use (roughly 5 °C). Below the dew point temperature, condensation on the cuvette windows will interfere with measurements.

With a dry gas flow rate of at least 100 cc/min into the gas inlets of the sample holder and room temperature water, the minimum temperature is about 0 °C. Using ice water as the coolant, the minimum temperature will be about -15 °C..

The minimum temperature of -50 °C can be attained, but only using dry gas flow and coolant temperatures about 25 °C above the target temperature (typically using a refrigerated bath circulating a fluid that will not freeze at the bath temperature – for example methanol / water mixtures).

---

## **4.7. Troubleshooting**

If the submersible pump has operated without being immersed in water, it may not prime properly. If this occurs, place one drop of detergent on the pump intake and try again.

If the **TC 1** display indicates that there is inadequate coolant (see section 4.13), it may be that the circulating water is too warm. Add ice to the water to cool it down.

If a circulating bath is used as the water source, it may not be possible to maintain a flow rate of 200 mL per minute. While this condition will limit the lowest temperature attainable, it may be partially compensated by using colder circulating liquid. Below about -25 °C, 30% methanol in water is generally recommended as the coolant.

If the set temperature is near room temperature and too close to the temperature of the cooling water, the sample holder temperature may oscillate around the set temperature. In this case, changing the temperature of the cooling water (for example by adding ice) should stop the oscillation. Cooler circulating water can result in improved performance of the cuvette holder at low temperatures, whereas warmer water can improve performance at very high temperatures.

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## **4.8. Error Messages**

When errors occur, the line 1 of the **TC 1** Temperature Controller display presents an error code. Line 3 of the display identifies the error and line 4 of the display presents possible solutions. The most common events that cause errors to be displayed are loose cables or inadequate coolant flow. For errors not easily solved, contact QNW at [www.qnw.com/contact-us/](http://www.qnw.com/contact-us/) or [service@qnw.com](mailto:service@qnw.com).

Specific error messages include the following.

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### **5 – cell out of range**

**warnings:** loose cable, sensor failure

The temperature controller is not receiving a reasonable response from the sensor on the cuvette tower. Either the sensor has failed or a cable is not making a good connection.

---

**6 – cell out of range**

**warnings:** loose cable, check cable

The temperature controller is not receiving reasonable responses from either the cell tower or heat exchanger sensors. Since it is very unlikely for both to fail, probably a cable is loose.

---

**7 – heat exchanger error**

**warnings:** loose cable, sensor failure

The temperature controller is not receiving a reasonable response from the sensor on the heat exchanger. Either the sensor has failed or a cable is not making a good connection.

---

**8 – inadequate coolant**

**warnings:** inadequate coolant, water temperature

The sensor on the heat exchanger is reading a temperature above 60 °C. Temperature control has been shut down to prevent damage to the Peltier element. Either the water was too warm or the rate of flow was inadequate to draw sufficient heat from the heat exchanger.

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**9 – Invalid command**

The controller has been sent an invalid text command. This error will be observed only when under computer control.

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## 5. Maintenance and Spare Parts

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### 5.1. Maintenance

The **NeutroniQ 3x3** and **TC 1** Temperature Controller require very little routine maintenance on the part of the user. For routine cleaning of exposed surfaces, use only a cloth dampened with water or diluted detergent. Do not use organic or abrasive solvents.

#### CAUTION



**Caution** – Any action which makes it necessary to open the **NeutroniQ 3x3** or **TC 1** Temperature Controller units must be executed only by QNW technicians or authorized personnel.

The water hoses and their attachments to the **NeutroniQ 3x3**, **TC 1** Temperature Controller, and submersible pump should be inspected prior to each usage to ensure that they are intact. Replace the nylon tubing when it becomes discolored or cracked. Periodically replace the water stored in the bucket to minimize the growth of microorganisms. When not in use, the pump, bucket, and tubing may be stored dry.

---

### 5.2. Spare Parts

Vinyl tubing and magnetic stir bars may be obtained from a variety of commercial vendors. For electrical cables to be used with **NeutroniQ 3x3** and **TC 1** Temperature Controller and submersible pumps, please contact QNW at [service@qnw.com](mailto:service@qnw.com).

# Appendix – Serial Communications for the TC 1 Temperature Controller

This document provides the serial communications protocols for version 1.0 of the firmware on the **TC 1** family of controllers used for the **NeutronIQ 3x3**.

All functions of the temperature controller can be managed from a computer, using the command set described below. If you purchased your unit as a component of a spectrometer from certain manufacturers, this feature may be implemented through traditional RS232 serial connectors on the computer or the spectrometer and on the controller. In this case they will be connected by a standard 15-pin serial extension cable (male connector on one end and female on the other). No driver installation should be needed.

Otherwise the serial linkage will be established through a USB connection between the computer and the controller. In this case the controller includes electronics which convert the USB connection to a serial communications port. However, for the port to be available to programs on the computer it will be necessary to load driver software. It is important that the driver software be loaded before connecting a USB cable between the controller and the computer. Contact Quantum Northwest at support@qnw.com for further information. Quantum Northwest can provide a control program written specifically for control of all functions of the temperature controller. Ask for program **T-App**.

In programming for the **TC 1** controller, one must adhere to the conventional notation: 8/N/1.

```
Baud:          19200
Data Bits:     8
Parity:        None
Stop Bit:      1
Flow Control:  None
```

For many of the commands listed below, the controller returns information in response to the command. All commands and responses are delineated by left and right square brackets ( [ ] ). Any text sent to the controller not enclosed within brackets will be ignored. In this document, an ellipsis (.....) is used to distinguish responses from commands.

<b>Note:</b> [command]	Purpose of the command (sent to the controller)
..... [response]	Meaning of the response (received from the controller)

## **1. Identify**

[F1 ID ?]	What is the ID number of the sample holder being controlled?
..... [F1 ID 14]	Sample holder is a <b>t2, NeutronIQ 3x3</b> , or other single cuvette sample holder.

The ID and version number are shown briefly on the display when the temperature controller is turned on.

### **Assigned Identities:**

```
ID = 00 – specialty sample holder
      14 – t2
      24 – t2x2
```

34 – turret6 or other multi-sample holder

## **2. Controller Firmware Version**

[F1 VN ?]                      What is the version number of the controller firmware?  
 ..... [F1 VN 1.00]            The controller is operating firmware version number 1.00.

## **3. Stirrer**

[F1 MS ?]                      What is the maximum stirrer speed?  
 ..... [F1 MS 2500]            The maximum stirrer speed is 2500 rpm.  
 [F1 LS ?]                      What is the lowest stirrer speed?  
 ..... [F1 MS 300]            The lowest stirrer speed is 300 rpm.  
 [F1 SS S 1000]                Set stirrer speed to 1000 rpm.  
 [F1 SS S 0]                    Turn stirrer off and set the speed to zero.  
 [F1 SS +]                      Turn stirrer on and set it to the most recent non-zero stirrer speed setting.  
 [F1 SS -]                      Turn stirrer off.  
 [F1 SS ?]                      What is the current stirrer speed setting?  
 ..... [F1 SS 1000]            Stirrer speed setting is 1000 rpm.

## **4. Temperature Control**

[F1 TC +]                      Turn temperature control on.  
 [F1 TC -]                      Turn temperature control off.

## **5. Target Temperature**

[F1 TT S 23.10]                Set target temperature to 23.10 °C.  
 [F1 TT ?]                      What is the current target temperature?  
 ..... [F1 TT 71.32]            Target temperature is 71.32 °C.  
 [F1 TT +]                      Turn on automatic reporting of manual changes to the target temperature.  
 [F1 TT -]                      Turn off automatic reporting of manual changes to the target temperature.  
 [F1 MT ?]                      What is the maximum target temperature allowed?  
 ..... [F1 MT 110]            The maximum target temperature allowed is 110 °C.  
 [F1 LT ?]                      What is the lowest target temperature allowed?  
 ..... [F1 LT -40]            The lowest target temperature allowed is -40 °C.

## **6. Instrument Status**

[F1 IS ?]                      What is the current instrument status?



.....[F1 IS 0-+S]	Response is four parameters: -number of unreported errors is 0 (0 to 9) -stirrer is off (+ is on, - is off) -temperature control is on (+ is on, - is off) -temperature is stable (S is stable, C is changing)
[F1 IS +]	Automatically report instrument status whenever it changes (e.g., due to manual changes at the controller)
[F1 IS -]	Stop automatic reports of instrument status.
.....[F1 IS R]	The controller has been powered off and back on again.

### **7. Current Sample Temperature**

[F1 CT ?]	What is the current temperature of the holder?
.....[F1 CT 22.84]	The current temperature is 22.84 °C.
[F1 CT +3]	Periodically report current temperature every 3 seconds.
[F1 CT -]	Stop periodic current temperature reports.

### **8. Error Reporting**

[F1 ER ?]	Report the current error.
.....[F1 ER 0]	No current error.
.....[F1 ER 5]	Cell T out of range (Loose cable? Sensor failure?).
.....[F1 ER 6]	Cell & heat exchanger T out of range (Loose cable?).
.....[F1 ER 7]	Heat exchanger T out of range (Loose cable? Sensor failure?).
.....[F1 ER 8]	Inadequate coolant (check flow). Temperature control has shut down.
.....[F1 ER 9 <<bad command>>]	Syntax error on a preceding command where <<bad command>> is the command that caused the syntax error.
[F1 ER +]	Automatically report errors when they occur.
[F1 ER -]	Stop automatic error reports.

### **9. Probe Status and Temperature**

[F1 PS ?]	Is there an external temperature probe connected?
.....[F1 PR +]	A probe is connected.
.....[F1 PR -]	No probe is connected.
[F1 PS +]	Enable probe status to be sent automatically when a probe is installed or removed. This is the default.
[F1 PS -]	Disable automatic sending of probe status.
[F1 PT ?]	What is the current probe temperature?

[F1 PT +3]	Periodically report the probe temperature every 3 seconds.
..... [F1 PT 22.3]	The current probe temperature is 22.3 degrees.
..... [F1 PT NA]	Probe temperature is not available.
[F1 PT -]	Stop periodic probe temperature reports.
[F1 PA S 0.5]	Set the increment for automatic reporting of the probe temperature to 0.5 degrees during a ramp. (Increment must be positive, without sign in tenths between 0.1 and 9.9 degrees, and will work for ramps going up or down.)
[F1 PA +]	Start automatic reporting of probe temperature every temperature increment (set by the command above).
..... [F1 PT 30.5]	The current probe temperature is 30.5 degrees.
[F1 PA -]	Stop automatic reporting of probe temperature every temperature increment.
[F1 PX +]	Change probe temperature returned to a precision of 0.01 degree.

### **10. Temperature Ramping**

[F1 RR S 2.10]	Set the ramp rate to 2.10 °C/minute.
[F1 RR ?]	What is the current ramp rate?
..... [F1 RR 2.10]	Current ramp rate is 2.10 °C/minute.

For the following three commands, there are no corresponding reference commands ([R1 . . .]; see command class 13).

[F1 TL +]	Ramp the sample and reference identically.
[F1 TL -]	Ramp the sample while the reference remains stable.
[F1 TL 0]	Ramp the sample and reference independently.

The default is to ramp the sample while the reference remains stable. Ramping both the sample and reference at different rates or to different target temperatures (see below) is not possible.

To Ramp the temperature:

1. equilibrate at the starting temperature,
2. set the ramp rate,
3. set a new target temperature (command class 5).

The target temperature may be above or below the current temperature; as soon as it is set, the ramp will begin, up or down, to that new target.

After reaching the target, the controller will hold at that temperature. At any time a new ramp rate and target temperature can be set to start a new ramp.

Once you are done, set the ramp rate to 0. Otherwise, setting a new target temperature later will initiate a ramp to that target temperature.

#### **Notes:**

The minimum settable ramp rate is 0.01 °C/minute.

For higher ramp settings, the observed rate may be lower than that calculated from RT and RS or it may be nonlinear over part of the temperature range because the maximum possible rate of heating or cooling is limited (and dependent on the ramp direction as well as on the temperature).

When the ramping process is completed the controller will send a response of the form [F1 TT #] as a notification of the end of the ramp. This response can be blocked at any time by previously sending the command [F1 TT -] (see command class 5).

### **11. Heat Exchanger Temperature**

[F1 HL ?]	What is the high temperature limit for the heat exchanger?
.....[F1 HT 60]	The heat exchanger high temperature limit is 60 °C.
[F1 HT ?]	What is the current temperature of the heat exchanger?
.....[F1 HT 39]	The current heat exchanger temperature is 39 °C.
[F1 HT +3]	Start periodic heat exchanger temperature reports every 3 seconds.
[F1 HT -]	Stop periodic heat exchanger temperature reports.

### **12. Cell Changing**

**These commands will have an effect only for a sample holder with multiple cuvette positions.**

[F2 DI]	Device initialize: move to home position.
[F2 PI]	Device initialize: move to home position and reply when done.
.....[F2 DL 1]	Device is finished moving.
[F2 DL 3]	Device locate: move to position 3. (Device should be initialized prior to using this command for the first time.)
[F2 PI]	Device initialize: move to home, then to position 1 and reply when done.
.....[F2 DL 1]	Device is in position 1.
[F2 PL 4]	Device locate: move to position 4 and reply when done. (Device should be initialized prior to using this command for the first time.)
.....[F2 DL 4]	Device is now in position 4.
[F2 DD 400]	Set speed to 400 (acceptable range 100 – 900, with a default of 500).

### **13. Reference Cuvette**

These commands will have an effect only for systems with two independently controlled sample holders.

[R1 ...]

To control and monitor the temperature and status of the reference cuvette using a Dual Temperature Controller, use any commands in classes 3-8, 10 and 11, substituting R1 for F1. There are no corresponding [R1 ...] commands for command classes 1, 2, 9 and 12.

If you wish to ramp the temperature of the reference and sample cuvettes together, please note the linking command, [F1 TL +], in command class 10.

#### 14. Specialty Sample Holder

**For a Specialty Sample Holder (ID = 00) controlled by a TC 1, there will be a second response to the [F1 ID ?] command.**

[F1 ID ?]                                      What is the ID number of the sample holder being controlled?  
 ..... [F1 ID 00]                              Sample holder is a **Specialty Sample Holder**  
 ..... [F1 FS (+/-)(+/-)(+/-)(+/-)(+/-)]

This response will automatically follow the [F1 ID 00] response. It defines the Functional Status (functionality) of the holder through its five (+/-) parameters. Each parameter defines whether the holder has (+) or does not have (-) the functionality as defined in the table below.

parameter #	functionality
1	Temperature Control
2	Stirring Control
3	External Probe
4	Multiple Sample Positions
5	Reference Holder

As appropriate to the functionality, the commands and responses in command classes 2 – 13 will also be available.

#### 15. Control of Automatic Reports

**The TC 1 can be controlled manually using the buttons and the display on the front. When a computer program is in use, the TC 1 will automatically send reports to the program whenever a manual change has been made. The commands in this section are intended to allow the program to prevent the TC 1 from sending those automatic reports.**

[F1 XX R(+/-)]                                      Stop (R-) or start (R+) reporting all manual changes, including those starting with [R1 and [F2.  
 By replacing the XX with individual command codes, a program can selectively stop or start reporting of manual changes.

[F1 SS R-]    Stop reporting manual changes for the sample stirrer.

[F1 TC R-]    Stop reporting manual changes for the sample temperature control.

[F1 TT R-]    Stop reporting manual changes for the sample target temperature.

[F1 PR R-]    Stop reporting manual changes for the external probe connection.

[F1 RR R-]    Stop reporting manual changes for the sample ramp rate.

[F2 PL R-]    Stop reporting manual changes for the sample position.

[R1 SS R-]    Stop reporting manual changes for the reference stirrer.

---

[R1 TC R-]	Stop reporting manual changes for the reference temperature control.
[R1 TT R-]	Stop reporting manual changes for the reference target temperature.
[R1 RR R-]	Stop reporting manual changes for the reference ramp rate.