

## Linkam (10002L) Procedure Manual (for users) 2020.07.08

### 1. Introduction.

Linkam is a device that can heat or cool samples from  $-190\text{ }^{\circ}\text{C}$  to  $600\text{ }^{\circ}\text{C}$ . This instruction manual describes the measurement procedure.

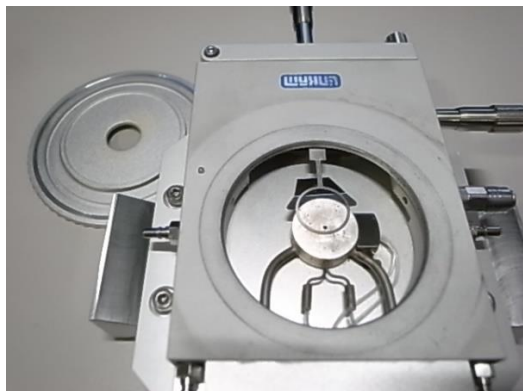
Since the beamline uses heating stages from several manufacturers, we refer to the heating stages by their manufacturer names for convenience. In the procedure, the heating stage is referred to as the main body of the stage.

Linkam consists of a stage body, holder, programmer, high-cooling unit, PC and Dewar bottle. The programmer is responsible for temperature control, while the high-cooling unit is responsible for sucking in liquid nitrogen and discharging dry nitrogen. Please leave the hooks on the Dewar bottle lids off for safety.

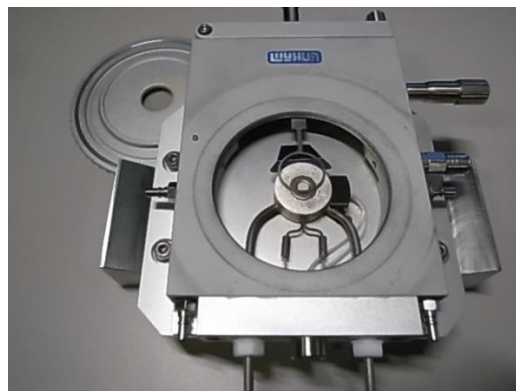
To avoid exposure during measurement, the instrument is operated remotely from outside the hatch.

### 2. Sample Setup

- Open the lid of the stage body and secure the sample with the ring.

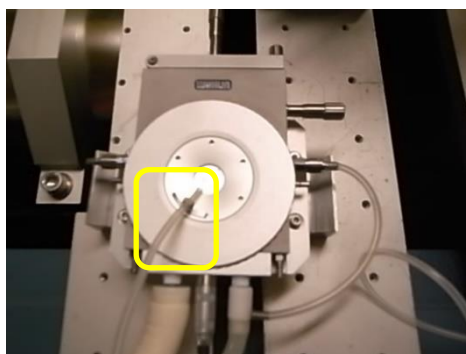


Before sample fixation

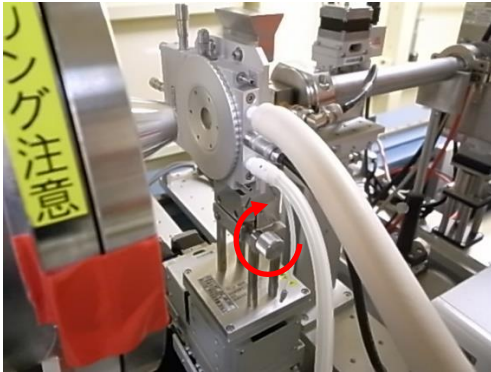


After sample fixation

- When cooling below room temperature, nitrogen should be sprayed from the strong cooling unit to the window of the stage unit to prevent condensation.



- Close the lid and make sure that the sample does not fall out when the stage body is in the vertical position.
- Insert the stage body toward the back of the holder.
- The side to which the cables are connected on the stage itself is the side that people normally access.
- Turn the holder screw firmly to the right to secure the stage body.



BL-6A



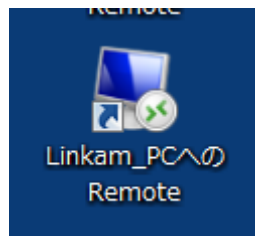
BL10C



BL-15A2

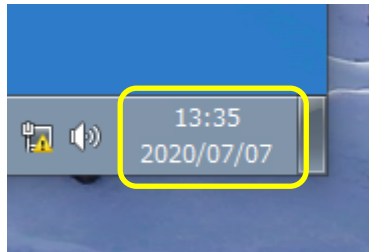
### 3. Time synchronization and operation via remote access

- From a terminal outside the experimental hutch (measurement PC), left-click on the Remote shortcut to Linkam\_PC.



- When the remote connection window appears, check to see if the PC is on the same time as the measurement PC to which you connected. If there is a match,

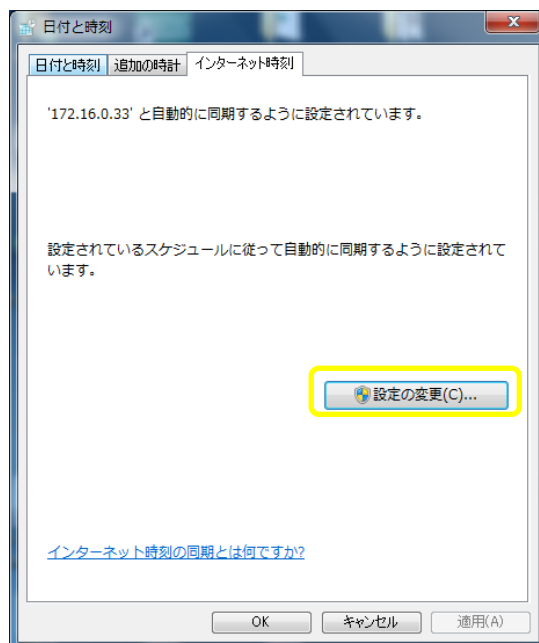
proceed to activate "LINK". If they do not match, match them according to the following procedure. Right-click on the clock in the lower right corner of the remote window and select "Adjust Date and Time".



•Date and Time will launch. From Date and Time, select the Internet Time tab.

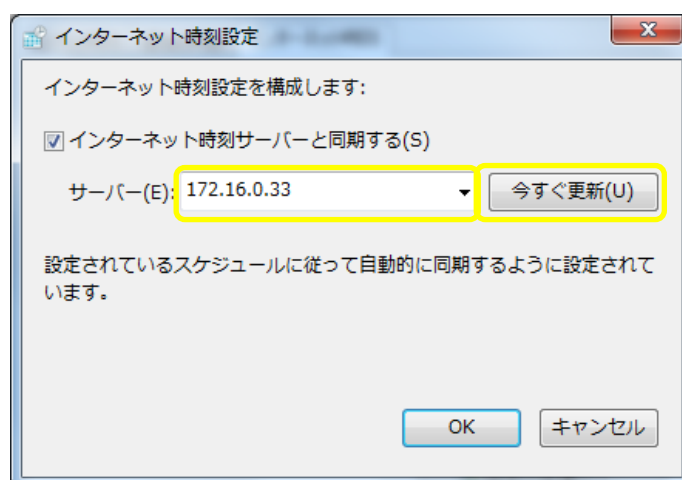


- Press Change Settings from Internet Time.



- Enter the IP address of the time server for each beamline shown below in Internet Time Server and press Update Now.

BL-6A	172.16.0.33
BL-10C	172.16.0.66
BL-15A2	172.16.0.1

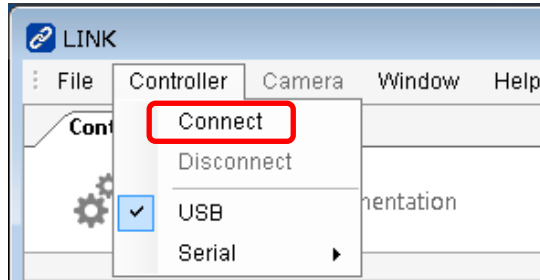


- Time synchronization is now complete.

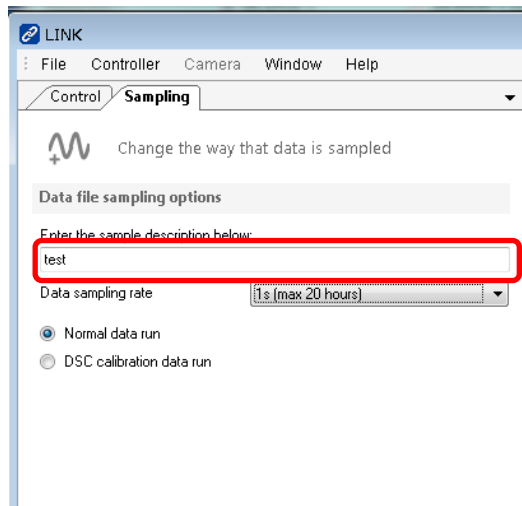
- Left-click on LINK to activate.



- Once LINK is activated, left-click Controller, then left-click Connect.



- The stage body and PC are now connected.
- Enter the file name of the temperature data in the red box below on the Sampling tab. In the following example, the file will be named test dd-mm-yy hh-mm-ss-ss.ldf and related to the date and time.

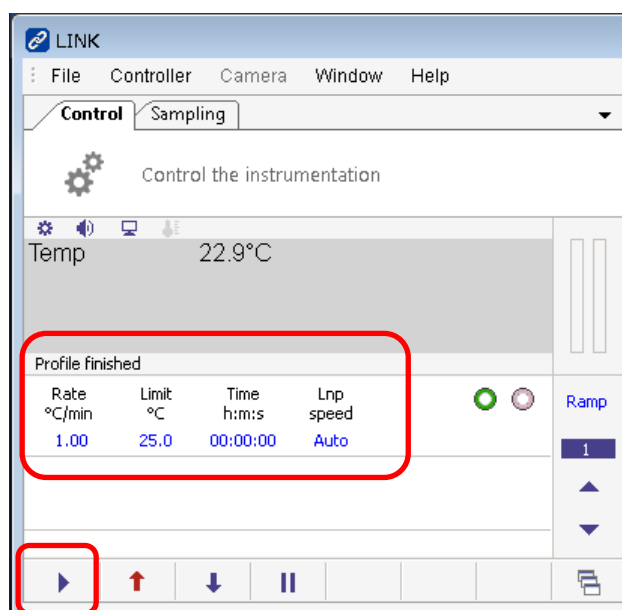


- If nothing is specified, it will be Unknown sample dd-mm-yy hh-mm-ss-ss.ldf.
- The folder where it will be stored is  
C:\Users\PFSA\My Documents\Linkam\link\DataFiles.
- Temperature sampling interval can be selected from 50 msec.(max. 1 hour) to 60 sec.(max. 50 days).

•Profile finished controls the temperature. Each parameter is shown below. You can enter values by right-clicking.

Rate °C/min: temperature gradient  
Limit °C: Attained temperature  
Time h:m:s: Attained temperature holding time  
Lnp speed: Liquid nitrogen pump speed

•The right arrow starts temperature rise or fall.



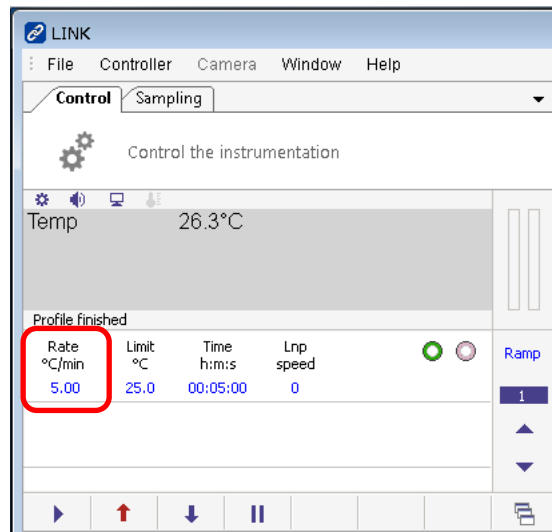
#### 4. Temperature setting profile

•This section describes an example of creating and executing a temperature change profile shown below.

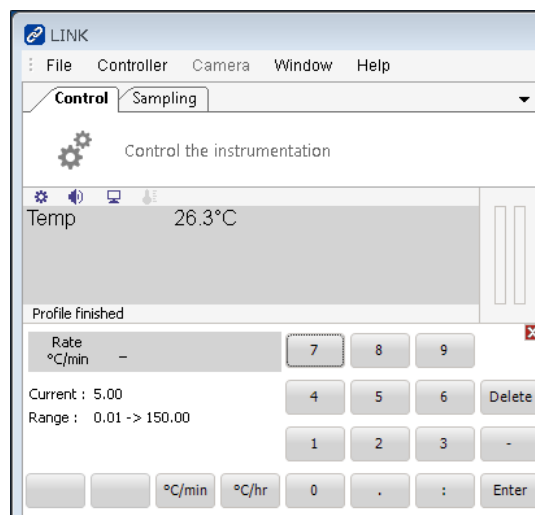
- The temperature gradient is set to 25 °C at 5 °C /min and maintained for 5 minutes.
- The temperature gradient is set to 100 °C at 5 °C /min and maintained for 10 minutes.
- The temperature gradient is set to 200 °C at 5 °C /min and maintained for 10 minutes.
- The temperature gradient is set to 25 °C at 5°C /min and maintained for 10 minutes.
- Set Ramp cycle to 1. Use the up and down arrows to change it.



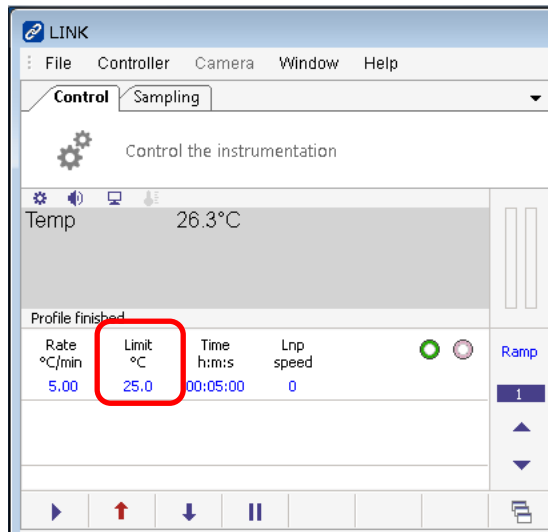
•Left-click on Rate °C /min.



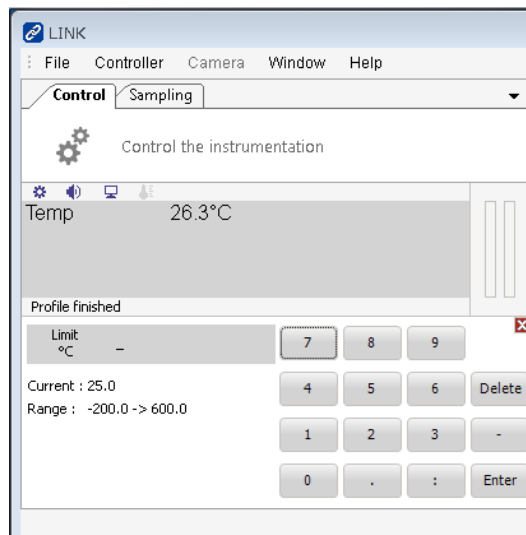
•Left-click on "5" and "Enter."



•Left-click on Limit °C.

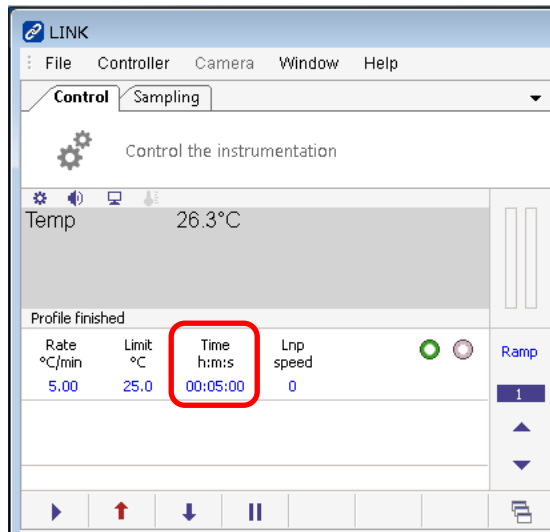


•Left-click on "2," "5," and "Enter."

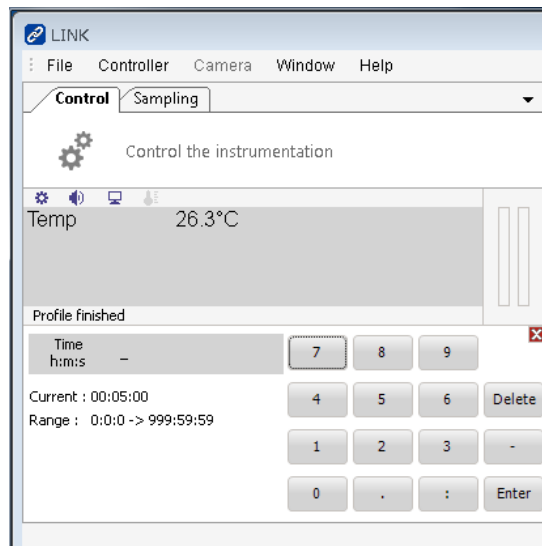


•Left-click on Time h:m:s.

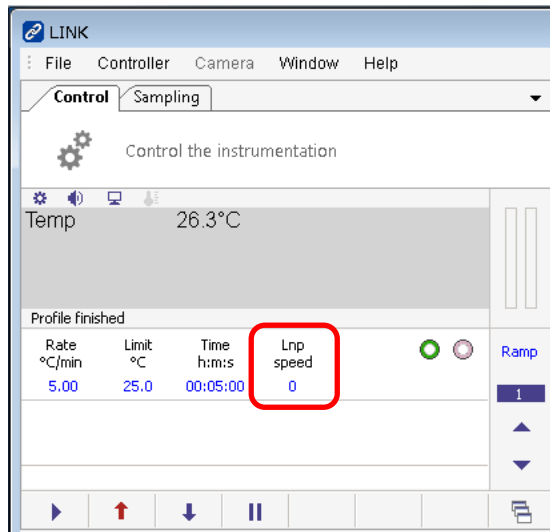




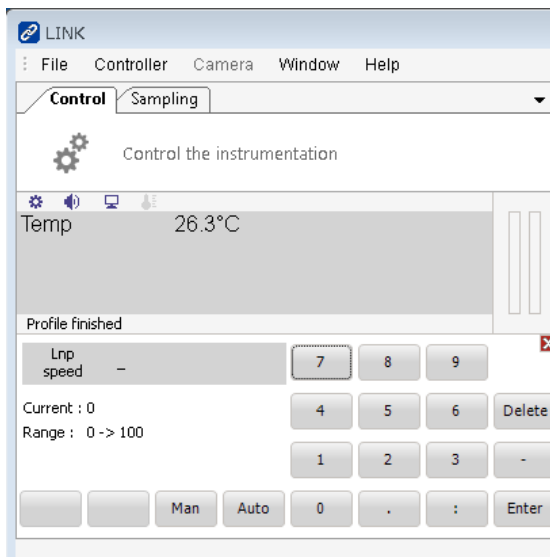
•Left click on "5", ":", "0", and "Enter".



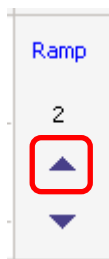
•Left click on Lnp speed.



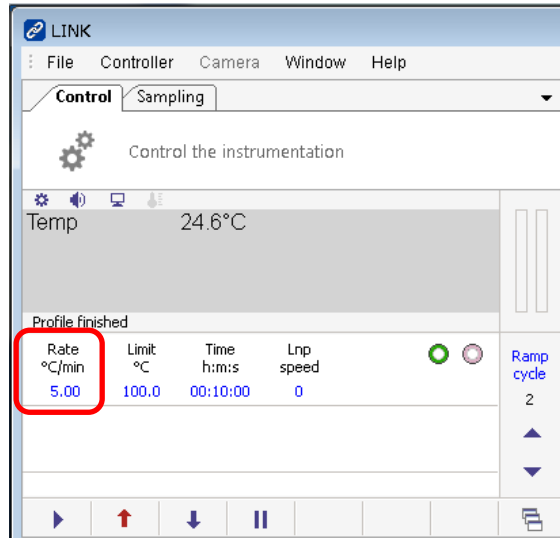
•Left click on "Auto" and "Enter".



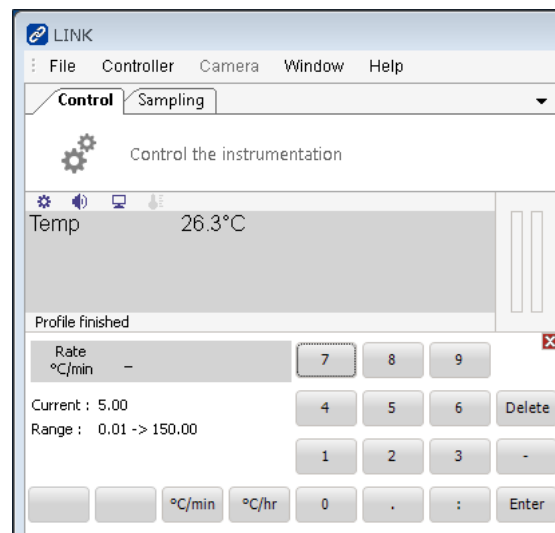
•Change Ramp cycle to 2 with the up arrow.



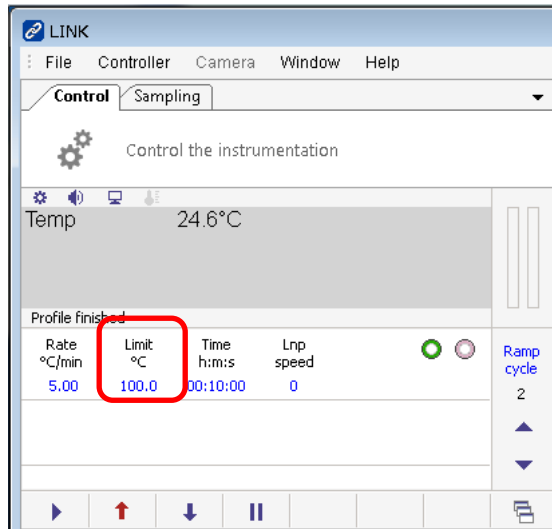
•Left-click on Rate °C/min.



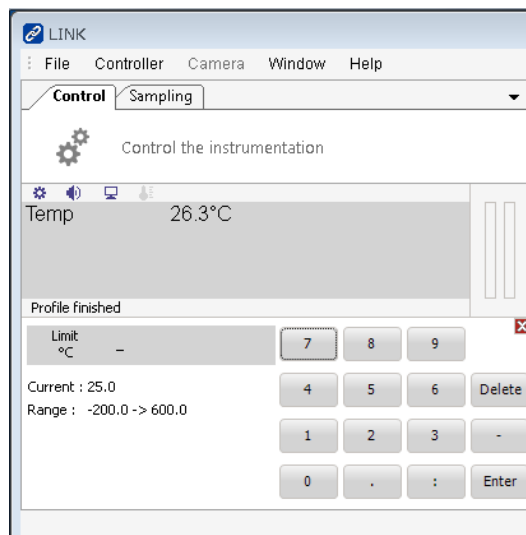
•Left-click on "5" and "Enter."



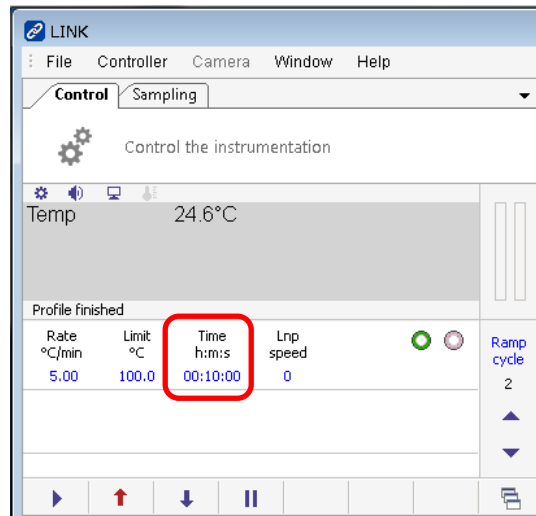
•Left-click on Limit °C.



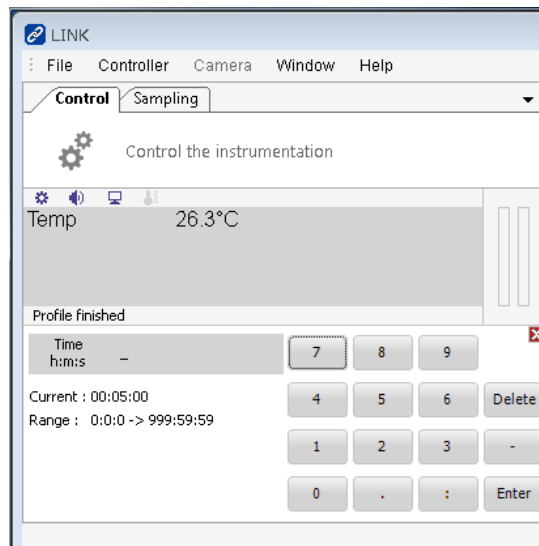
•Left click on "1", "0", "0", and "Enter".



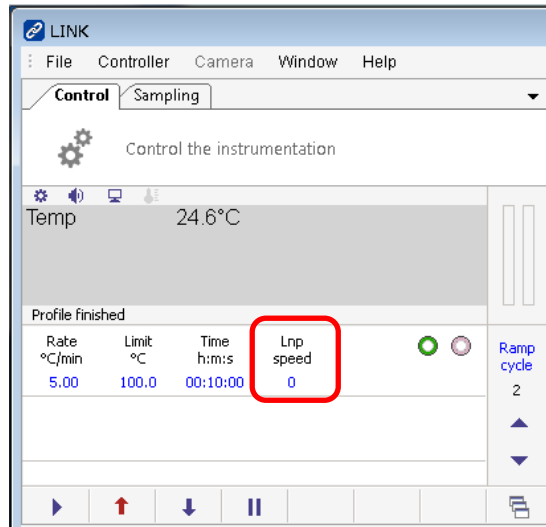
•Left-click on Time h:m:s.



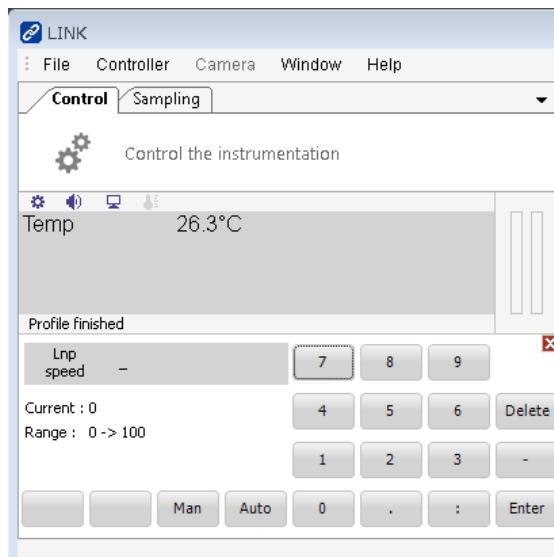
•Left click on "1", "0", ":", "0", and "Enter".



•Left click on Lnp speed.



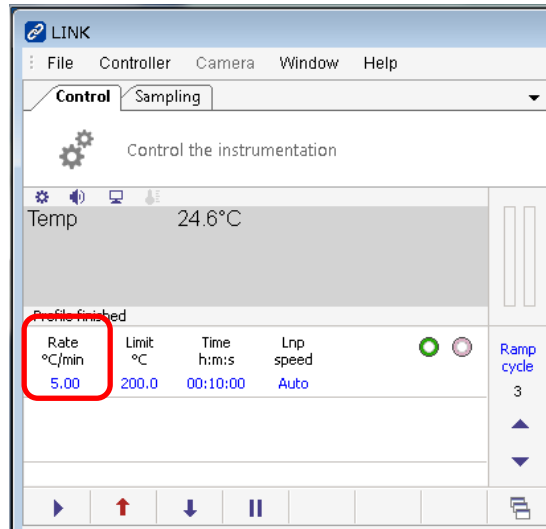
•Left click on "Auto" and "Enter".



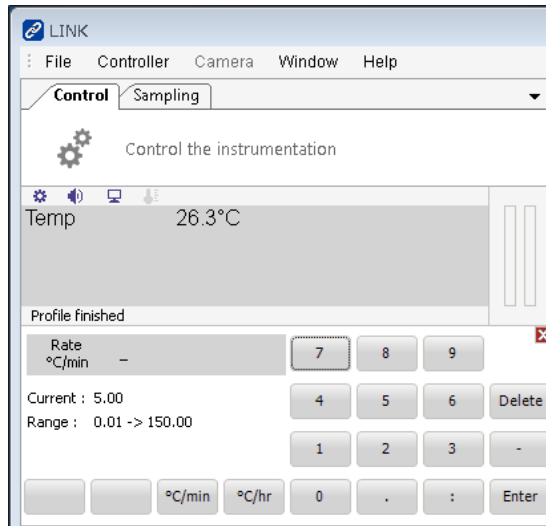
•Change Ramp cycle to 3 with the up arrow.



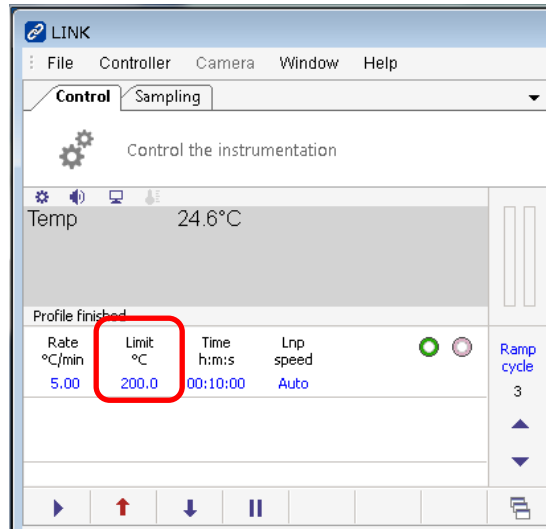
•Left-click on Rate °C/min.



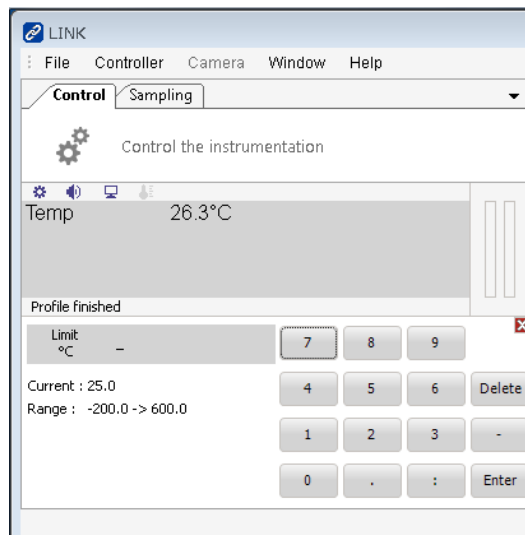
•Left-click on "5" and "Enter".



•Left-click on Limit °C.

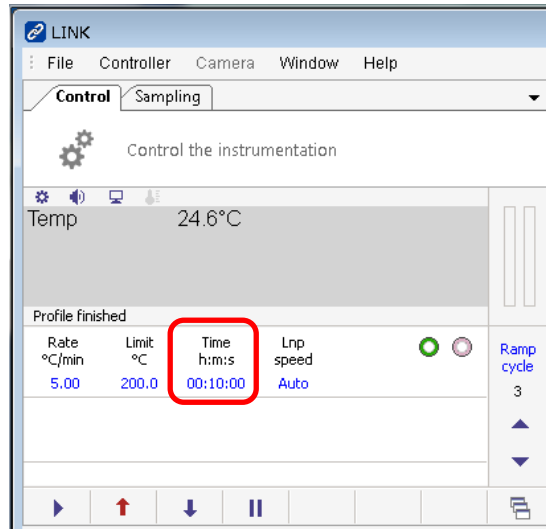


•Left-click on "2," "0," "0," and "Enter."

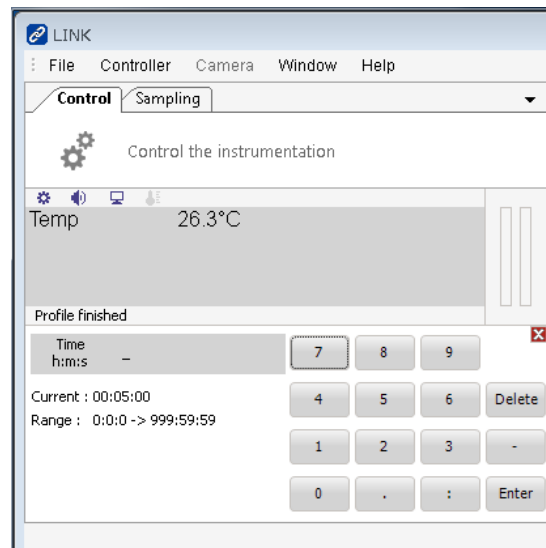


•Left-click on Time h:m:s.

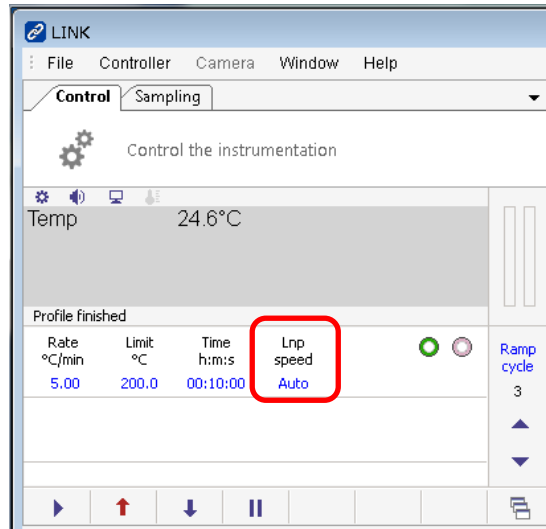




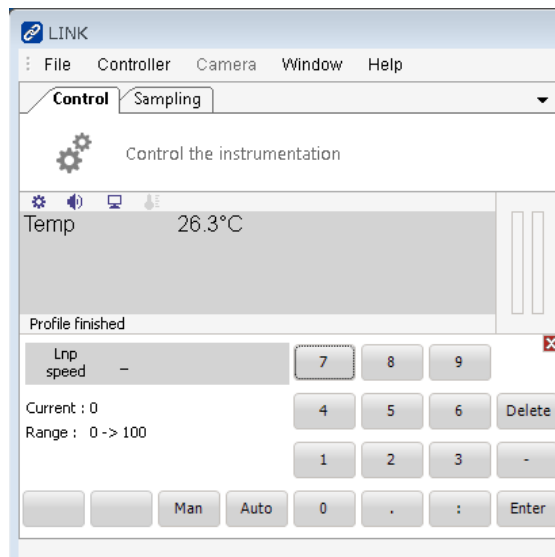
•Left click on "1", "0", ":", "0", and "Enter".



•Left click on Lnp speed.



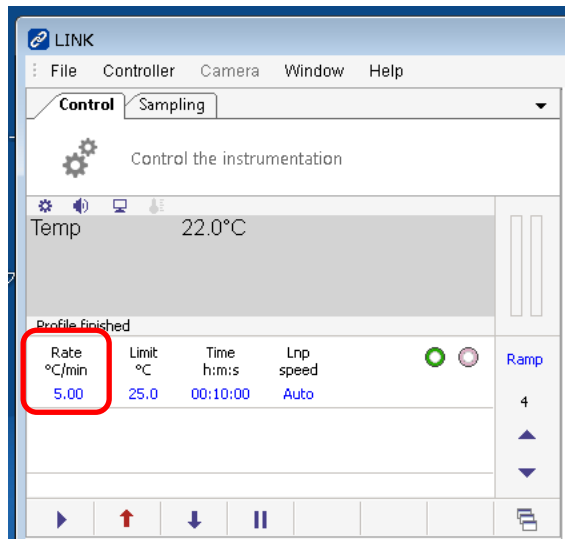
•Left click on "Auto" and "Enter".



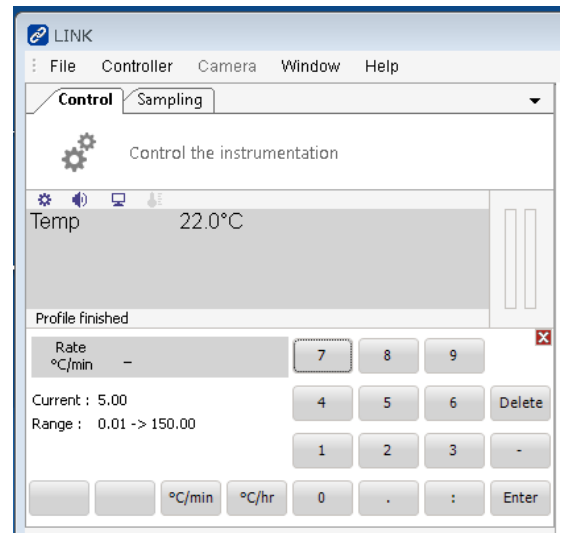
•Change Ramp cycle to 4 with the up arrow.



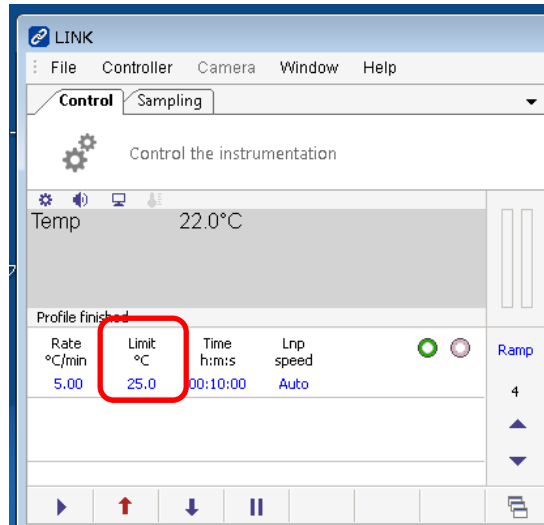
•Left-click on Rate °C/min.



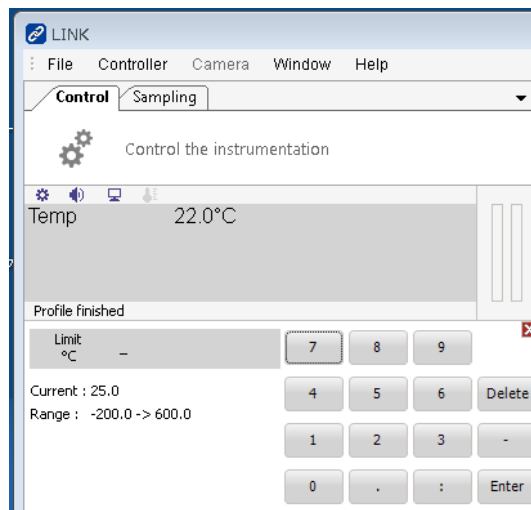
•Left-click on "5" and "Enter."



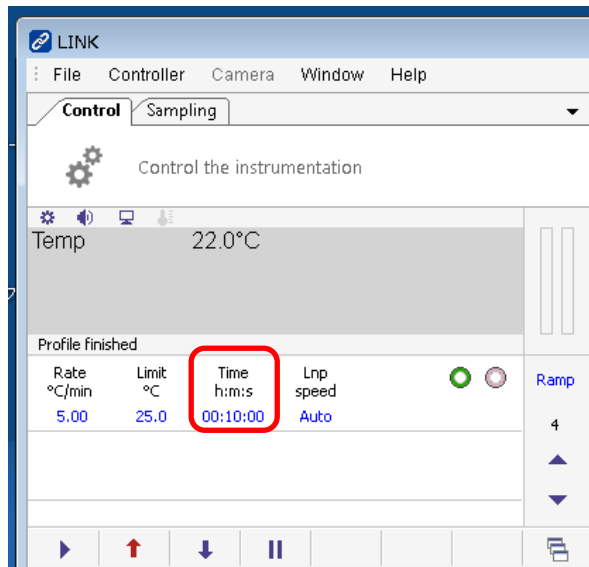
•Left-click on Limit °C.



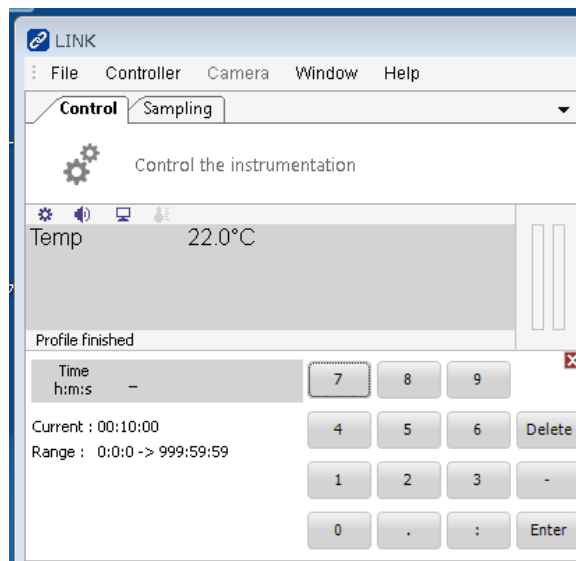
•Left-click on "2," "5," and "Enter."



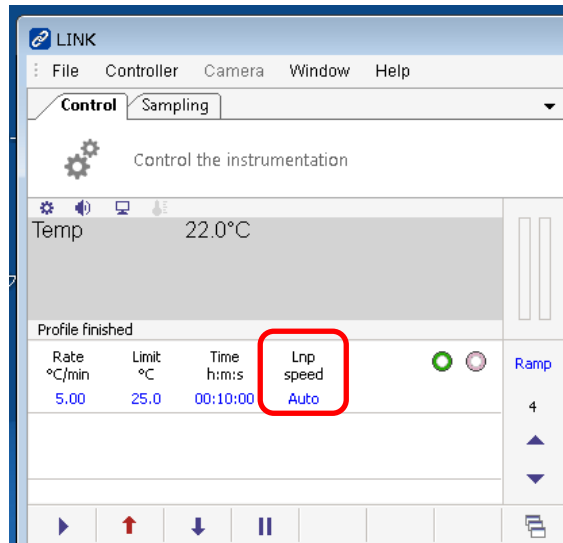
•Left-click on Time h:m:s.



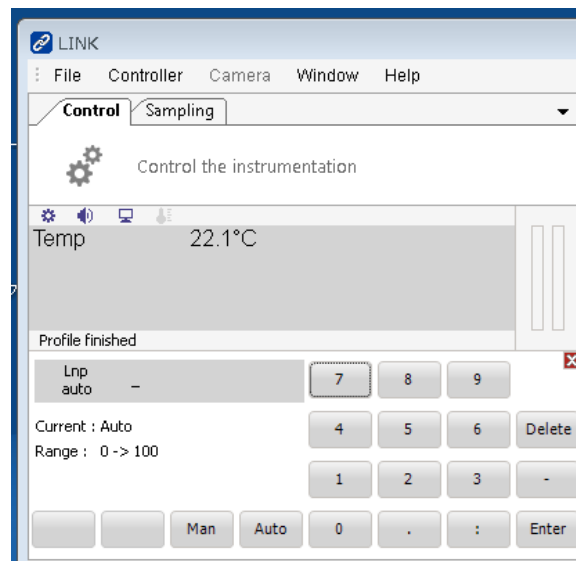
•Left click on "1", "0", ":", "0", and "Enter".



•Left click on Lnp speed.



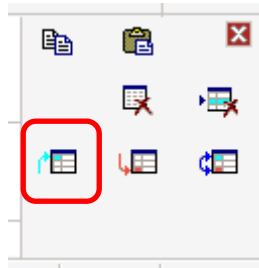
•Left click on "Auto" and "Enter".



•Select the start ramp; set Ramp to 1 with the up and down arrows.



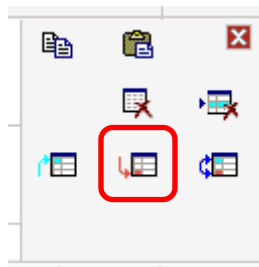
- Left-click on Ramp cycle.
- Right-click on the icon surrounded by the red line below.



- Select the end ramp; use the up and down arrows to set Ramp to 4.



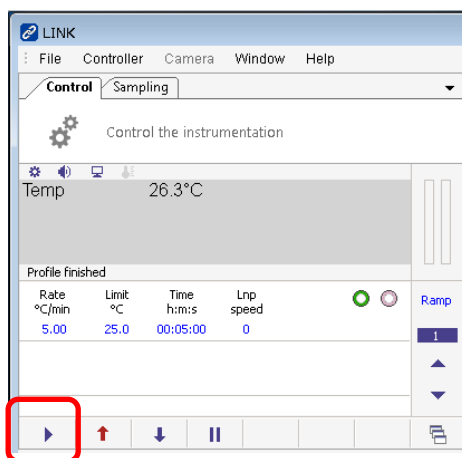
- Left-click on Ramp cycle.
- Right-click on the icon framed in red below.



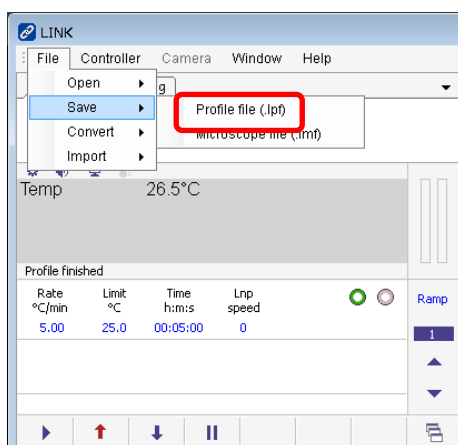
- The profile is now complete. By changing the Ramp cycle with the up and down arrows, you can check the Ramp cycles 1 through 4 that you have entered so far.



- The right arrow allows the temperature to be raised or cooled according to the profile.

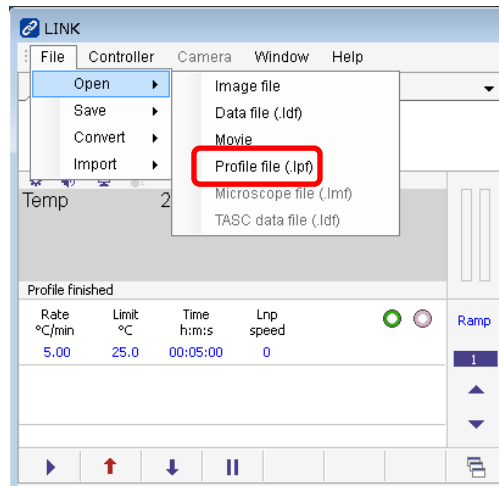


- You can save the profile by going to File>Save>Profile file(.lpt). Save the file to the file server of each beamline.



- You can load a profile by going to File>Open>Profile file(.lpt).

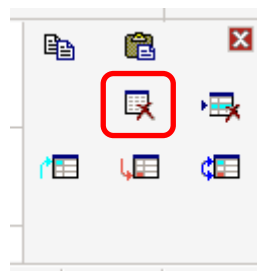




- When the measurement is complete, left-click on Ramp cycle.



- Left-click the icon framed in red below to delete the profile.



## 5. Liquid nitrogen replenishment

- Since Dewar bottles do not have liquid level sensors, occasionally lift the Dewar bottle to check the remaining level.
- Liquid nitrogen is located in the room behind BL-7, the CE room.
- To pump out liquid nitrogen, refer to the manual supplied with the 250L Dewar bottle.

