INSTEC 1st Unit / 2nd Unit Procedure Oct. 13 2021
User Manual

1. Introduction

- INSTEC is a device capable of heating or cooling from -190 ℃ to 400 ℃. This manual provides instructions for operating the equipment.
- In the beamline, several manufacturers' heating and cooling stages are used, so we refer to the heating stage by the manufacturer's name for convenience. In this manual, we will call the heating and cooling stage the "stage main body."
- INSTEC consists of 1st Unit and 2nd Unit, each comprising the Stage Main Body (mk2000 for 1st Unit / mk2000B for 2nd Unit), LN2-P, PC, and Dewar flask. The mk2000/mk2000B is responsible for temperature control, while LN2-P functions to extract liquid nitrogen and expel dry nitrogen.
-There are differences in nomenclature between the 1st Unit and 2nd Unit, as shown in the table below:

|	1st Unit	2nd Unit
Current Temperature of the Stage	TC	PV
Target Temperature (Set during Ramp or Control)	TT	CSP
Final Set Temperature	TF	TSP
Temperature Measured by Thermocouple (Actual Sample Cell Temperature)	TM	MV
Controller	mk2000	mk2000B

- To avoid exposure to radiation during measurements, we operate remotely from outside the hatch.
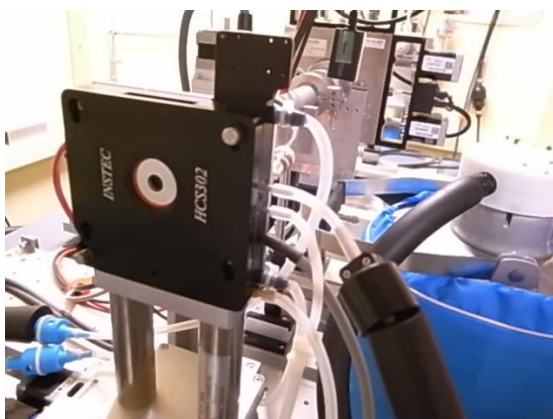
Unit 1 Stage Main Body and Dewar Flask

Unit 1 PC, MK-2000 (left), and LN2-P"

1
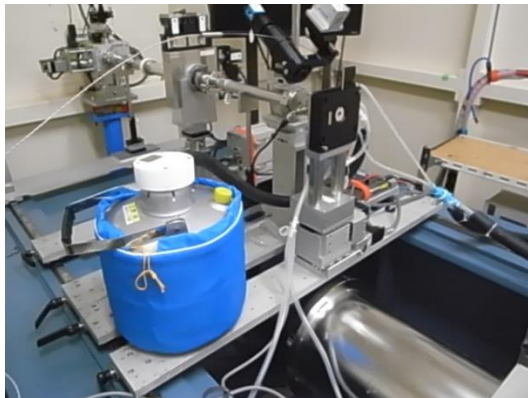
Unit 2 Stage Main Body and Dewar Flask   Unit 2 PC, MK-2000 (left), and LN2-P

2. Dispensing Liquid Nitrogen

- Please make sure to attend the PF refrigerant training (once per fiscal year).
- Liquid nitrogen is located in the room behind BL-7.
- Please wear gloves when dispensing.
- Insert injection tube ① into the Dewar flask.

-Turn extraction valve ② to the left to fully open it.
-Turn discharge valve③ to the right to fully close it.
-While observing the pressure gauge, gradually boost valve④ to the left to open it and adjust it to around 0.05MPa.
-When the replenishing Dewar flask starts to fill up, liquid nitrogen may spray out. Stay calm and follow the steps below to complete the process.
- Turn boost valve④ to the right to fully close it.
- Turn discharge valve③ to the left to fully open it.
- Turn extraction valve② to the right to fully close it.

- Remove injection tube ① from the Dewar flask.
- Finally, please double-check that the extraction valve② is fully closed, the discharge valve③ is fully open, and the boost valve④ is fully closed.

- Please record the amount dispensed on the paper.
- When taking the Dewar flask outside, please write your name, container number, etc., on the whiteboard on the wall.

-When replenishing liquid nitrogen in the Dewar flask provided with INSTEC, please use the funnel and plastic stand as shown in the photo (left). When topping up, remove the rubber plug at the top and directly refill using the funnel (right).

- With the supplied Dewar flask, if the stage body is used at temperatures ranging from room temperature to 100°C, the liquid nitrogen will last for approximately 24 hours.

3. Oxygen Monitor

- The Oxygen Monitor is used to prevent asphyxiation accidents when using liquid nitrogen to heat or cool the sample. The Oxygen Monitor is pre-installed by the staff. The display unit is usually placed beside the hatch used for entry and exit. An alarm will sound when the oxygen

concentration drops below 18%

4. Sample Set-up

- Please secure the samples in the long cell, as shown below, using Mendel's tape or similar. The side with notches should be positioned downstream.

-Please set up the stage body as shown in the photos below. The thermocouple from the INSTEC unit is temporarily inserted into the adapter hole used to secure the cell, as shown in the photo. However, for accurate temperature measurements near the sample, it is necessary to insert the thermocouple into the upper hole of the cell or directly contact the sample (as shown in the photo, using a washer to hold the sample). This allows us to measure the temperature of the sample cell (≒sample). If you are not using the thermocouple, please inform the staff. (We do not use Graphtec's thermocouples.)
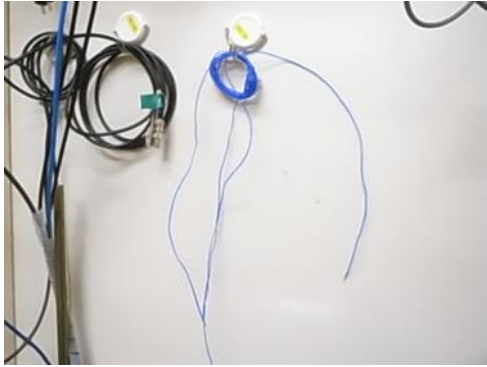
*There are two basic types of thermocouples.

(1) Metal Sheath: This type has a wide operating temperature range. It is used for temperatures above 100 ℃.
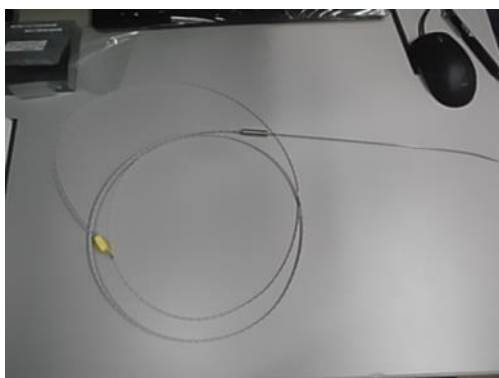
(2)Vinyl Sheath: This type is suitable for temperatures below 100 ℃. It can also be used for temperatures below 100 ℃. To do so, remove the tip from Graphtec's

thermocouple and replace it with the metal sheath.
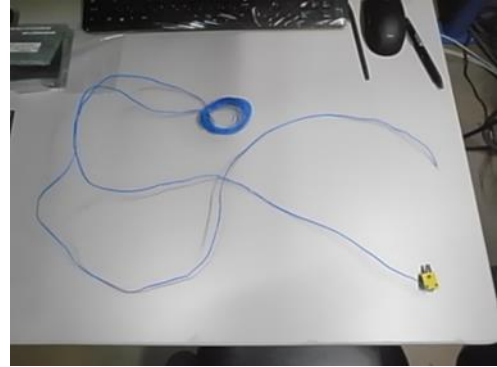
The screw hole next to the insertion port of the sample cell. If you are not measuring the temperature of the cell, you can leave it inserted here.

In practice, for each sample, the thermocouple is inserted into the sample cell (through the upper hole, for example) or placed in contact with a washer holding the sample to measure the temperature at a position as close as possible to the actual sample temperature.
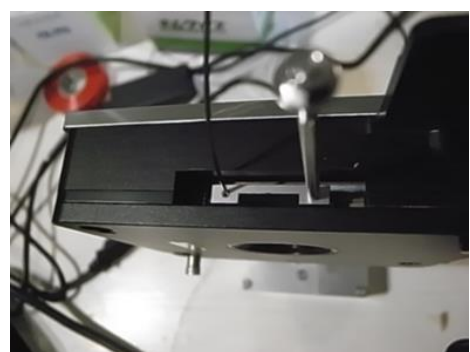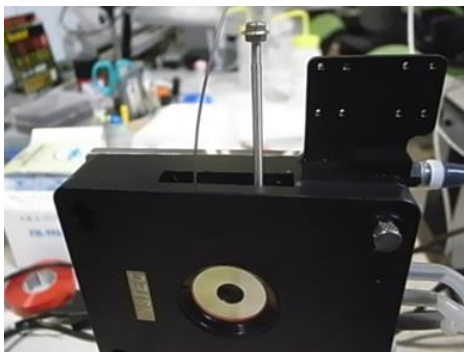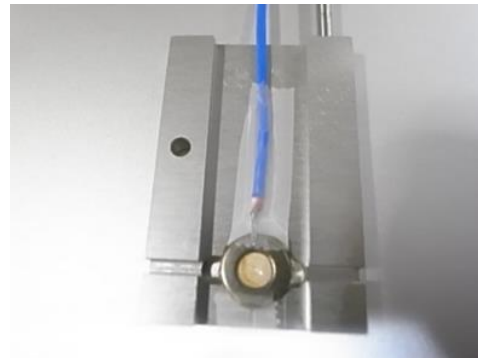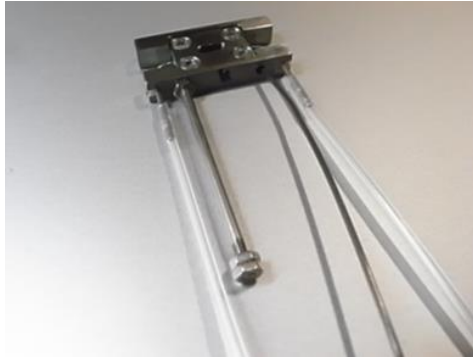
The flow rate of liquid nitrogen in LN2-P is automatically controlled, so please do not touch it with your hands.
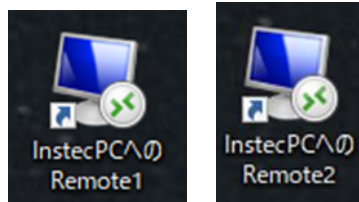

| Unit 1 | Unit 2 |

5. Time synchronization and remote access operation

- From the terminal (measurement PC) outside the experimental hatch, please double-click on the shortcut "Remote1 to Instec_PC (InstecPC への Remote1)" for Unit 1, or "Remote2 to Instec PC (InstecPC への Remote2)" for Unit 2 .


-Once the remote connection window appears, please check if the time on the connected measurement PC is synchronized. If the time is synchronized, proceed to launch the "New Instec App." If the time is not synchronized, follow the steps below to adjust it for Unit 1. For Unit 2, the instructions will be provided later. Right-click on the clock in the bottom right
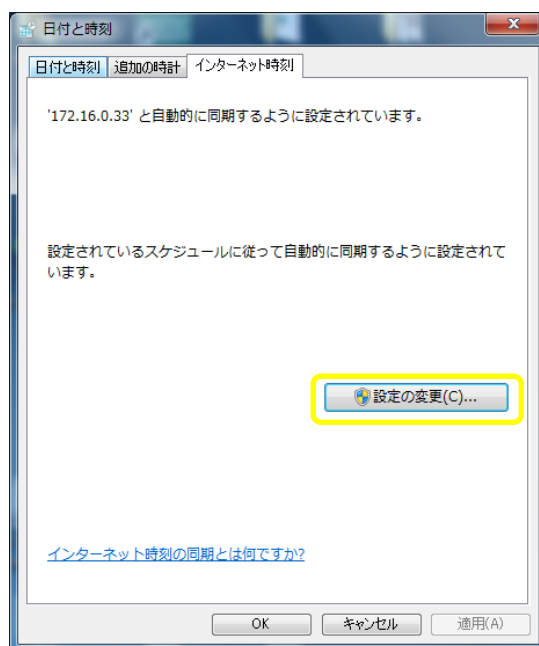
corner of the remote window and select "Adjust Date/Time (日付と時刻の調整)."
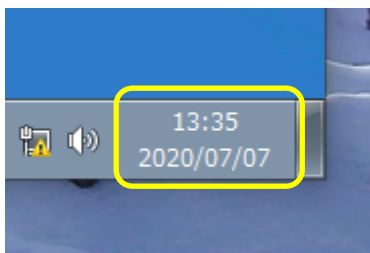
-Open Date and Time settings (日付と時刻). From Date and Time, select the Internet Time tab(インターネット時刻).
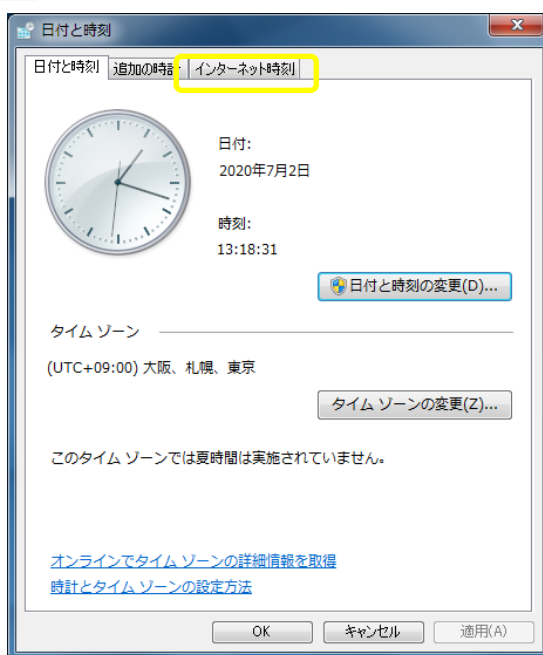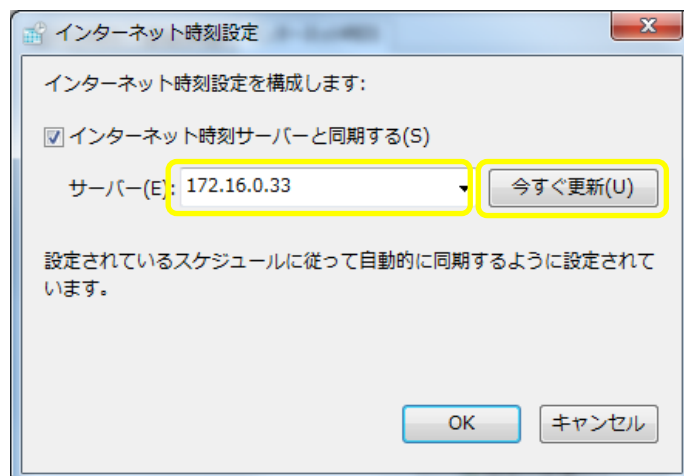

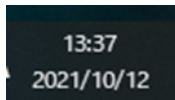-Please press "Change settings (設定の変更)" in the Internet Time (インターネット時刻) section

-Please enter the IP address of the time server for each beamline shown below in the Internet Time Server (インターネット時刻サーバー) field, and then click 'Update Now (今すぐ更新)'.

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

-Synchronization of Unit 1's time is now complete.

-For Unit 2, follow the steps below to synchronize the time: Right-click on the clock in the bottom right corner of the remote window and select 'Adjust Date/Time (日付と時刻の選択)'.
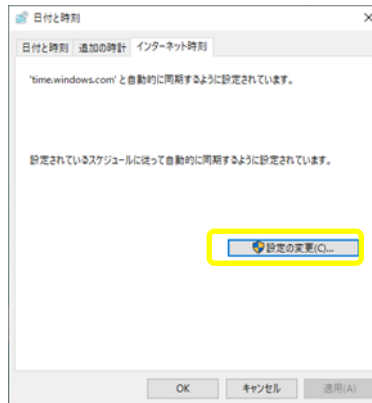
-Date and Time (日付と時刻) settings will open. Please left-click on 'Add clocks for different time zones (別のタイムゾーンの時計を追加する)'.

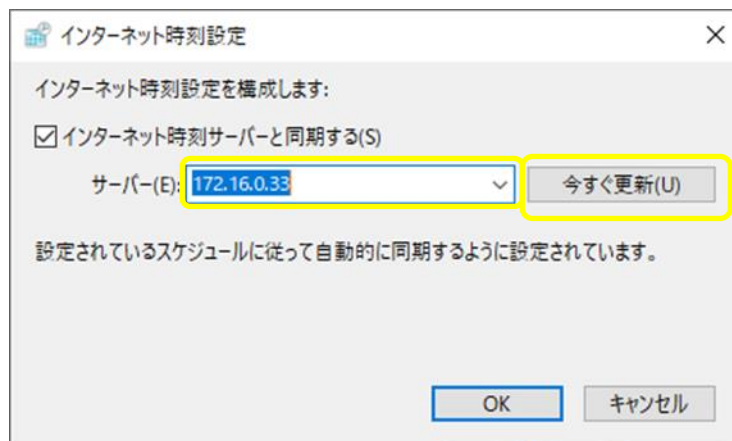-Please left-click on "Internet Time (インターネット時刻).
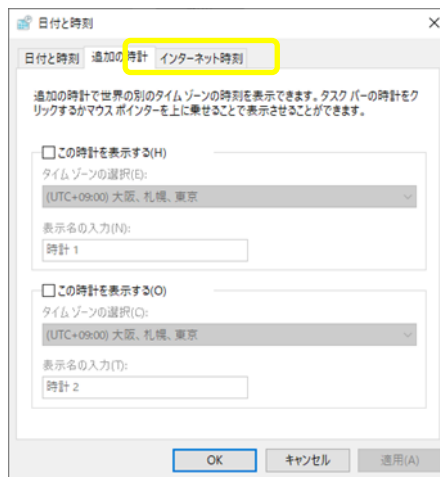
-Please left-click on "Change settings(設定の変更)."

Please enter the IP addresses of the time servers for each beamline shown below in the Internet Time Server field and click "Update now (今すぐ更新)":

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

-Synchronization of Unit 2's time is now complete.

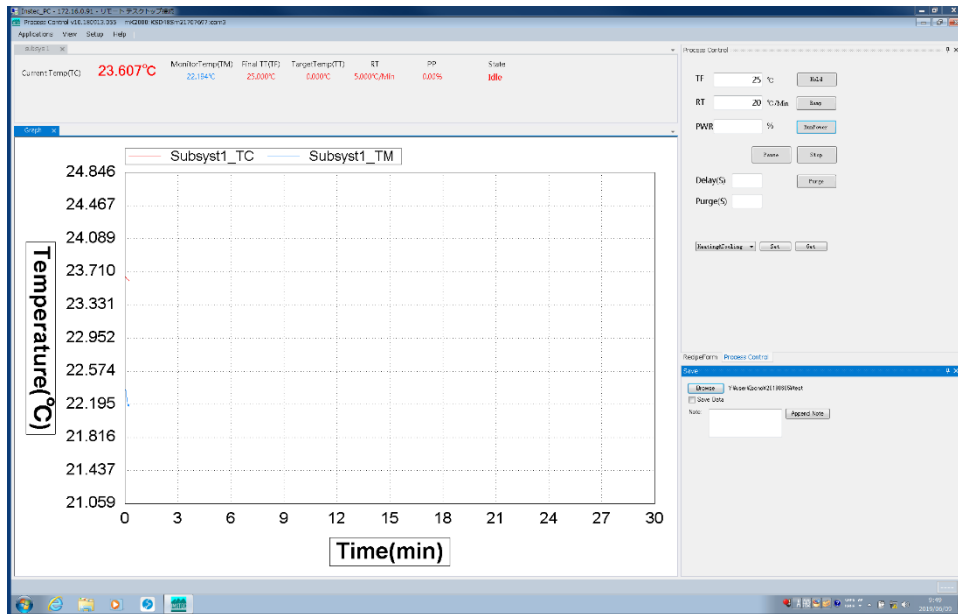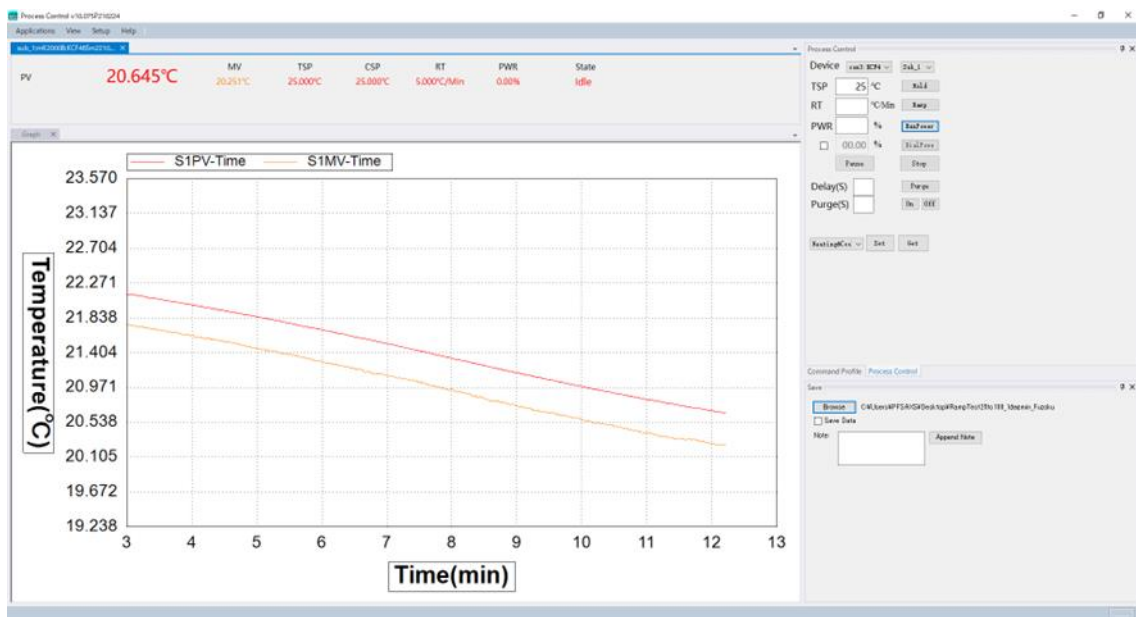-Please double-click on "New InstecApp" or "InstecApp.exe."

Unit 1	Unit2

-Please select "Process Control."

-The control panel for mk2000/mk2000B will be launched.
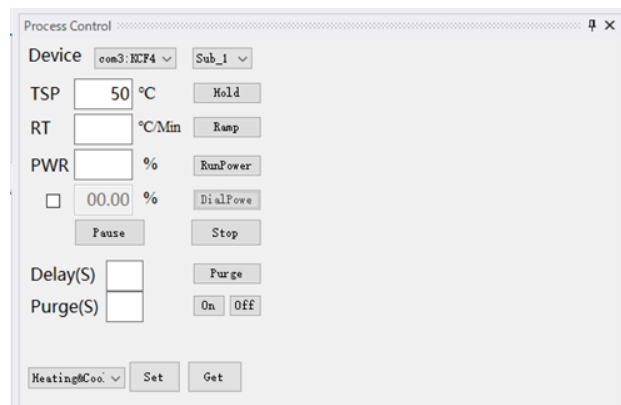

Unit 1

Unit 2

-In "Process Control," you control the temperature. The main buttons and parameters are as follows:

Hold:	Rapidly heats or cools to the target temperature (TF/TSP), but may cause overshoot.
Ramp:	Heats or cools with a specified temperature change rate (RT).
RunPower:	Heats or cools using the power input (PWR) level.
Pause:	Temporarily pauses temperature control.
Stop:	Stops temperature control.
TF/TSP:	Target temperature (°C).
RT:	Temperature change rate (°C/min).
Power/PWR:	Output power level (%).

-Here's an example of using "Hold" to heat up to 50 °C. Input 50 for TF/TSP and press the "Hold" button.
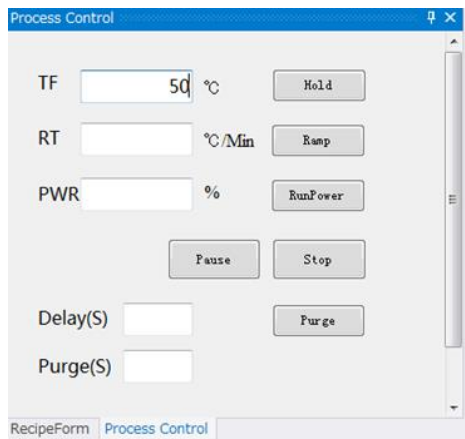
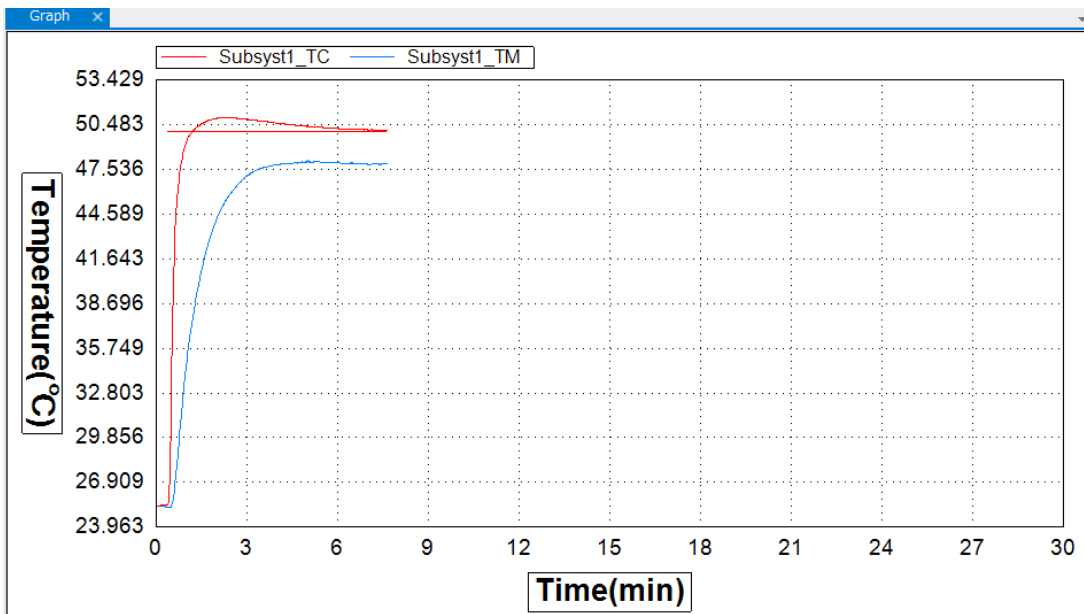Unit 1   Unit 2

-In Unit 1, the temperature change will follow the graph below. The red line represents TC (current temperature around the sample cell, showing overshoot), and the blue line represents TM (temperature of the thermocouple inserted outside the sample cell).
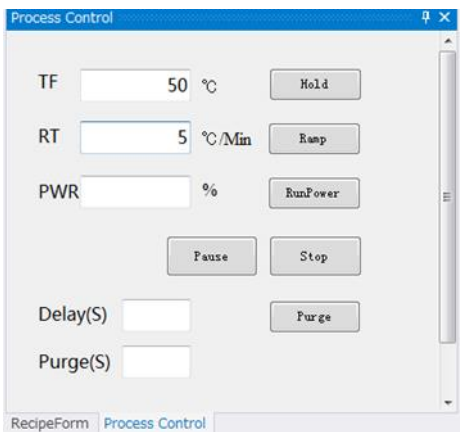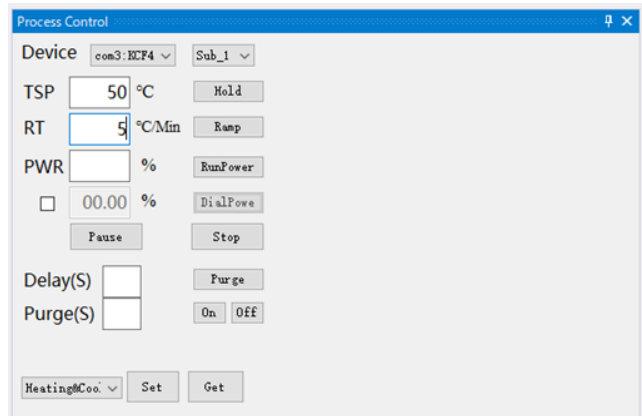
-In Unit 2, the temperature change will follow the graph below. The red line represents PV (current temperature around the sample cell, showing overshoot), and the orange line represents MV (temperature of the thermocouple inserted outside the sample cell).
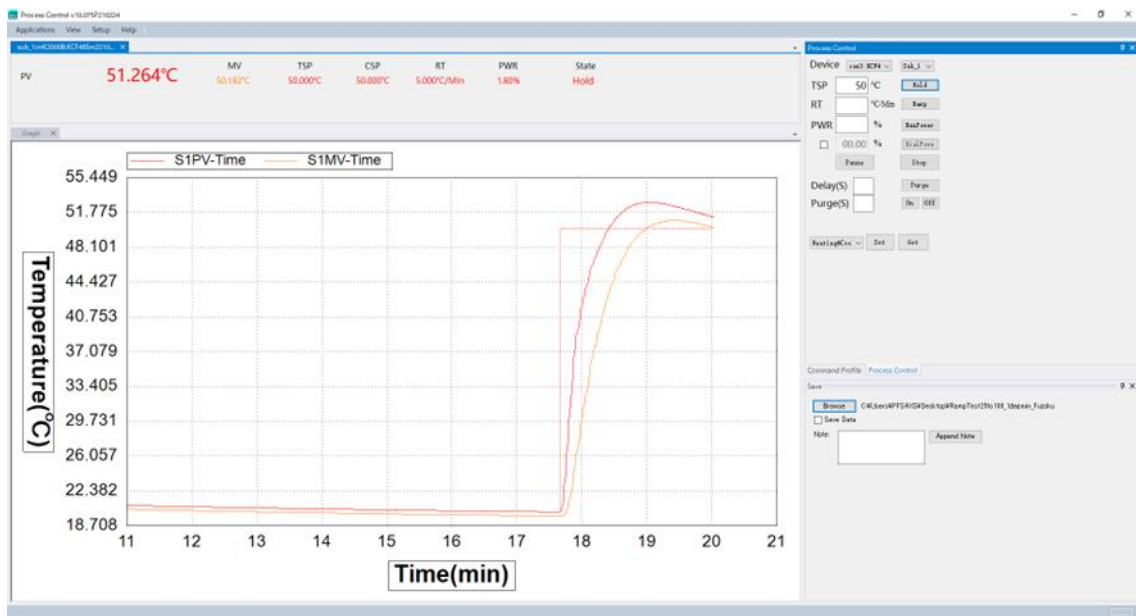
Here's an example of using "Ramp" to heat up from room temperature to 50 °C at a rate of 5 °C per minute. Input 50 for TF/TSP and 5 for RT, then press the "Ramp" button. Please make sure to enter the temperature change rate as an absolute value (adding a minus sign will prompt a pop-up message).
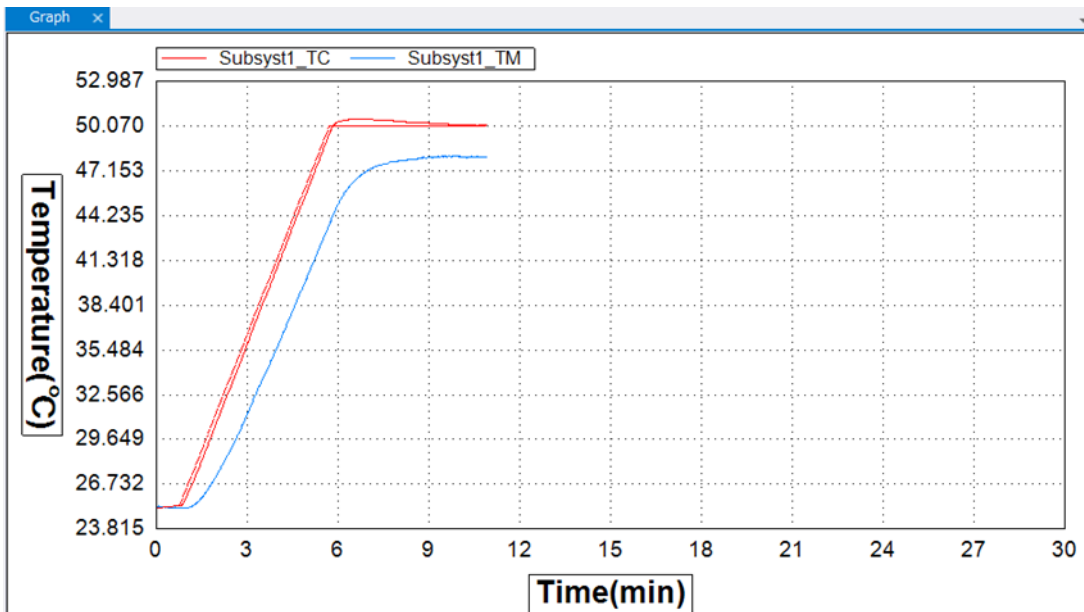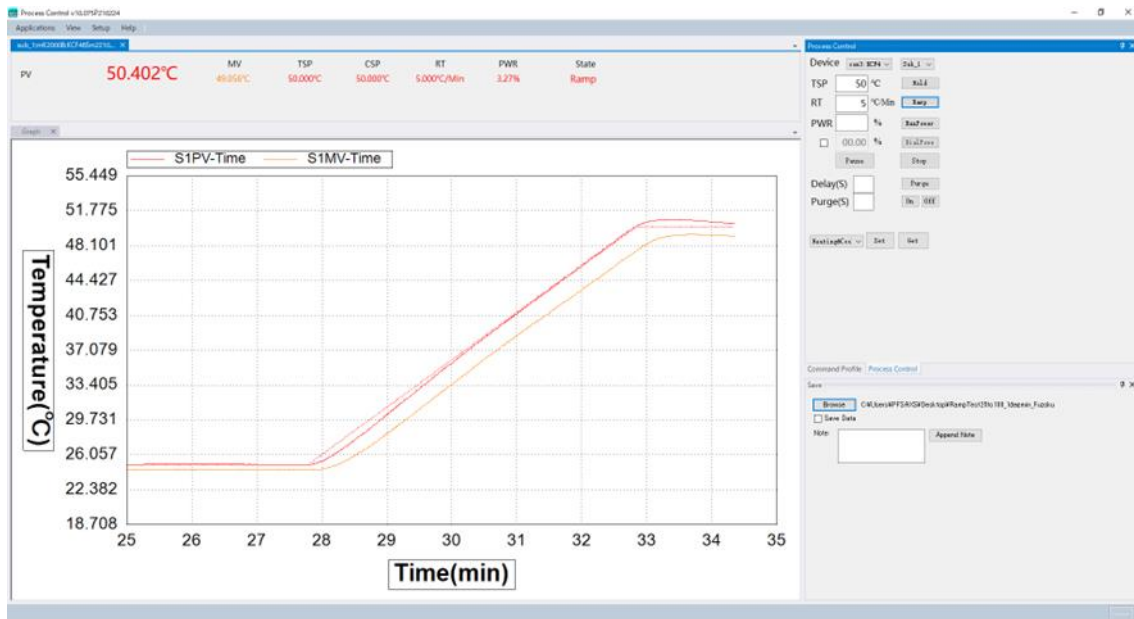
Unit 1   Unit 2

- The temperature change will follow the graph below.

Unit 1

Unit 2

The method to save temperature logs.

- Use "Browse" to determine the saving folder and file name.

- When you check the "Save Data" checkbox, it will start recording every second. The recorded temperature and values include TC/PV: the current temperature around the sample cell, TT/CSP: the target temperature at the moment, TF/TSP: the final set temperature, TM/MV: the temperature of the thermocouple inserted outside the sample cell, and Status.

- In addition to the data recorded every second, when you press "Append Note," the temperature and time at that moment will be added to the file. Along with this information, any notes you entered in the "Note" field will also be saved.
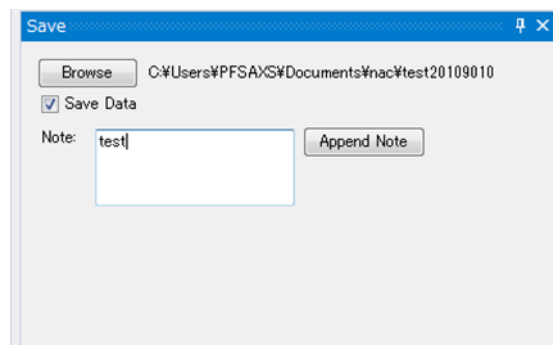
-When you right-click on the graph and select "Graph Setup...," the settings window will appear, allowing you to change the colors of the graph lines and other options.
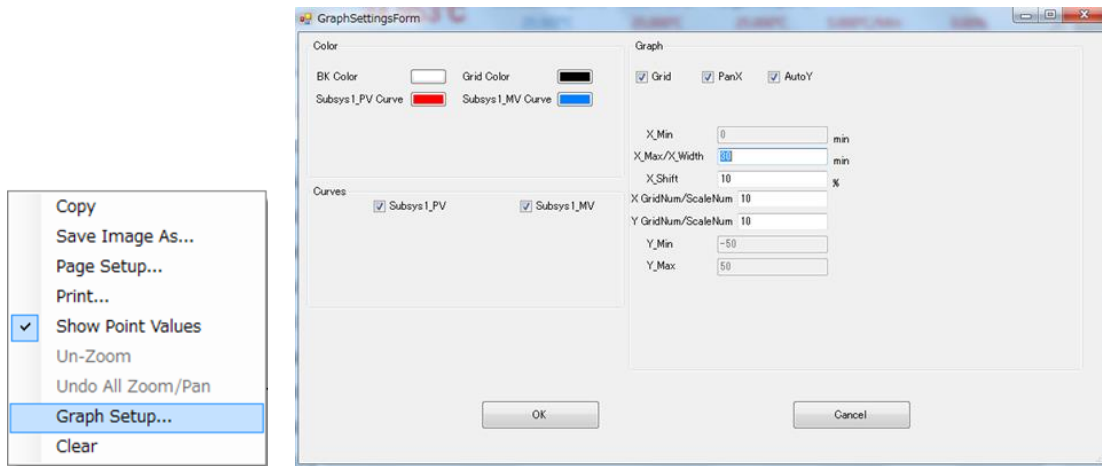
-By selecting the "RecipeForm/Command Profile" tab on the right side of the control screen for mk2000/mk2000B, you can configure the temperature change profile.
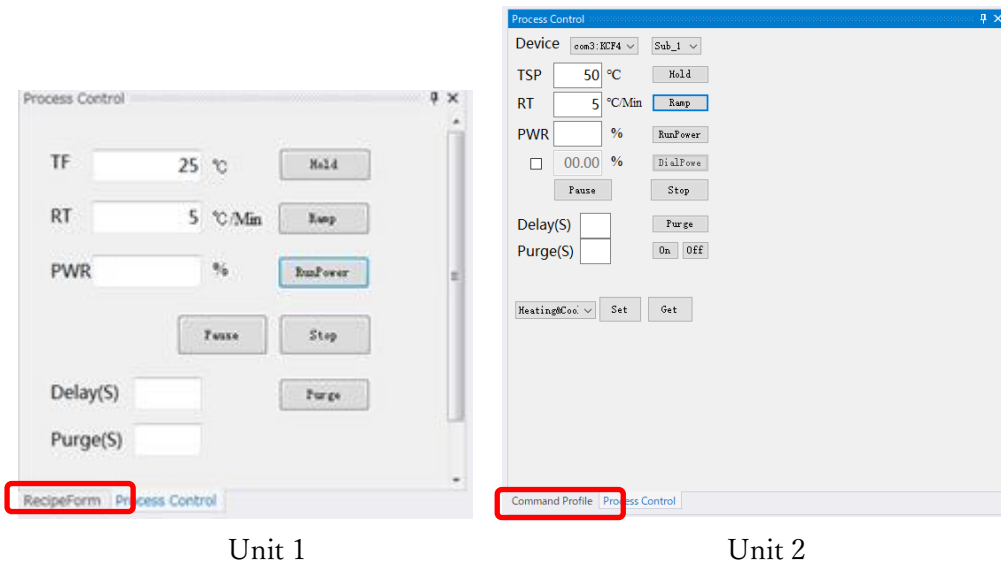
Unit 1   Unit 2

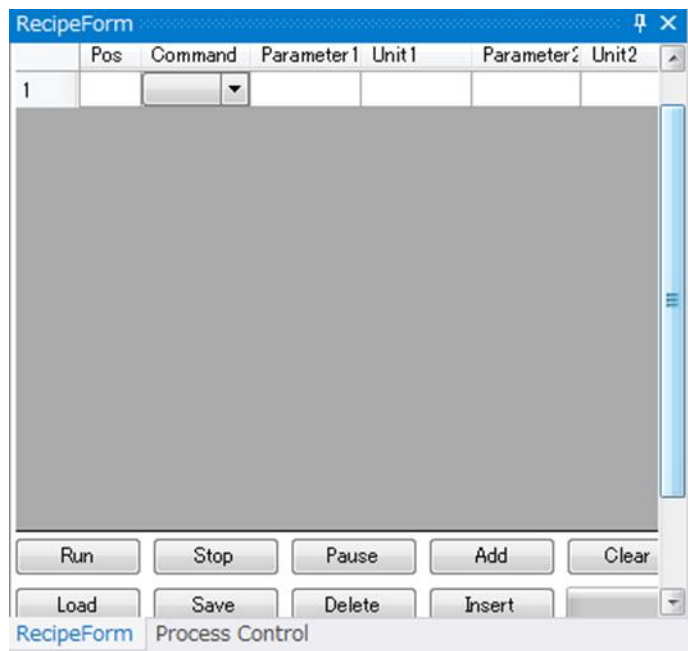-When you select "RecipeForm," the following screen will be displayed. Each button is as follows:

      Run: Execute the profile.

      Stop: Stop the profile.

      Pause: Pause the profile.

      Add: Add a new line to the profile.

      Clear: Delete all lines from the profile.

      Load: Load a profile.

      Save: Save the profile.

      Delete: Delete a line from the profile.
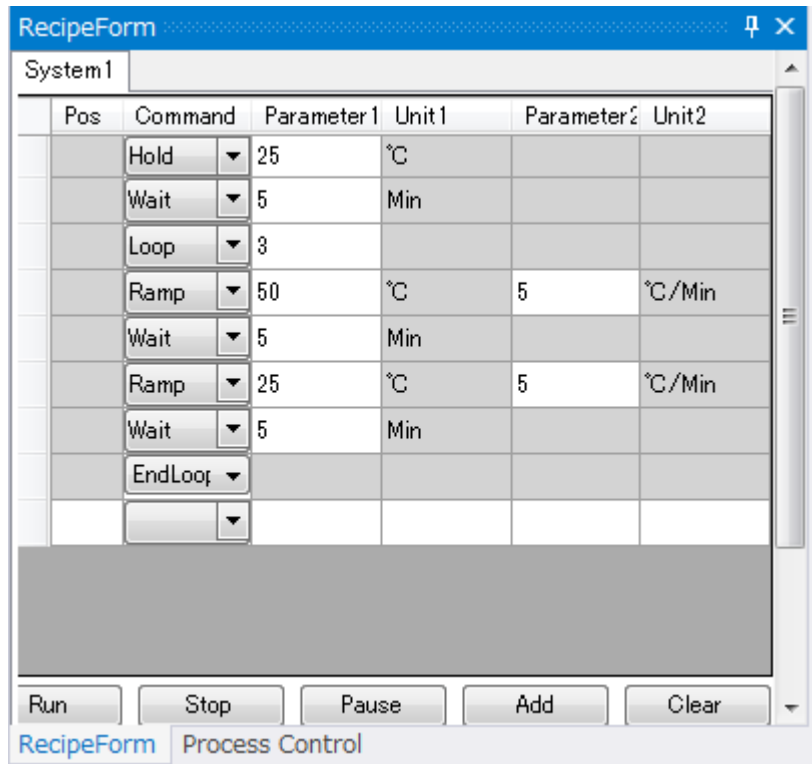
Insert: Insert a profile line between rows.

-The main commands and parameters of the profile are as follows:

Command	Parameter1	Parameter2	Remarks
Hold	Set temperature (°C)	None	
Ramp	Set temperature (°C)	Rate of temperature change (°C/min)	
Wait	Wait time (minutes)	None	
Loop	Number of loops		
End Loop	None	None	End of the loop

-As an example, the following profile shows temperature control under the given conditions.
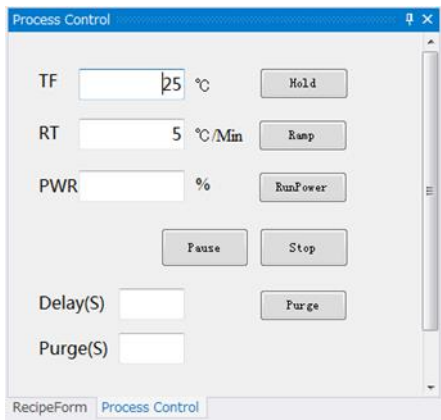
Please input the rate of temperature change as an absolute value.

       (1) Set the temperature to 25 °C.

       (2) Hold for 5 minutes.

       (3) Repeat the following loop 3 times.

       (4) Ramp up to 50 °C at a rate of 5 °C/min.

       (5) Hold for 5 minutes.

       (6) Ramp down to 25 °C at a rate of 5 °C/min.
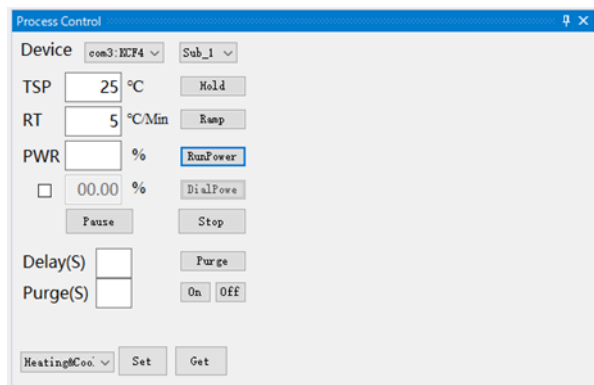
       (7) Hold for 5 minutes.

       (8) Go back to (3).

	Pos	Command	Parameter 1	Unit 1	Parameter 2	Unit2
		Hold ▼	25	℃		
		Wait ▼	5	Min		
		Loop ▼	3			
		Ramp ▼	50	℃	5	℃/Min
		Wait ▼	5	Min		
		Ramp ▼	25	℃	5	℃/Min
		Wait ▼	5	Min		
		EndLoop ▼				
		▼				

Run Stop Pause Add Clear

RecipeForm Process Control

6. Shutdown procedure.

- Please input 25 for TF/TSP and press the "Hold" button.

Unit 1	Unit 2

-Once it returns to room temperature, please leave it as it is.

7. The points to be noted are as follows:

-For Unit 1:

When heating from 25 ℃ to 100 ℃, at a temperature gradient of 6 ℃/min or higher,

there is a delay of approximately 2 to 3 seconds during Ramp. When the temperature gradient is reduced to 5 ℃/min or lower, the delay becomes more significant, reaching around 17 to 18 seconds at 1 ℃/min. (Please note that this is the current "specification" and is under investigation by the domestic representatives, Tokyoy Technica, and Instec.)

-For Unit 2:

When heating from 25 ℃ to 100 ℃, at temperature gradients ranging from 1 ℃/min to 10 ℃/min, the delay during Ramp falls within a range of approximately 2 seconds early to 3 seconds late.

- The Instec stage cannot move in sync with the measurements. It is necessary to set the measurement conditions to precisely measure according to the programmed temperature profile without any excess or deficiency.