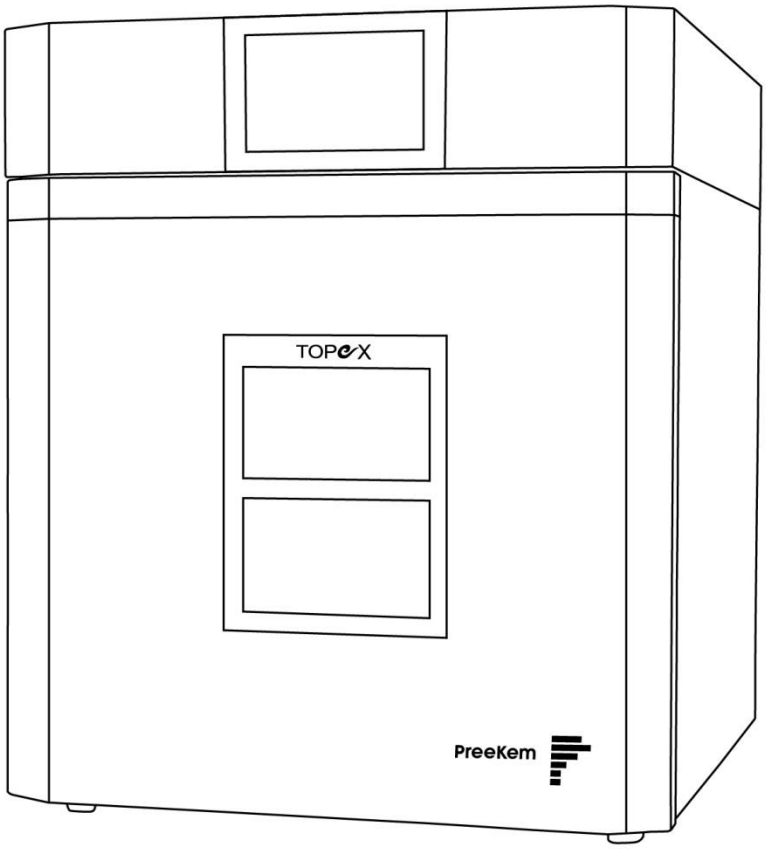
PreeKem

Operation Manual



TOPEX

Preface

Safety Declaration

Please read the entire manual carefully before operation. The operator needs follow up with the safety declaration paragraph strictly. Otherwise PreeKem should not be responsible for all the accidents.

ATTENTION	Symbol ATTENTION means danger exist, to remind you that you must pay attention. Improper operation or failed to comply with the appropriate procedures may result in equipment damage.
DANGER	Symbol DANGER means danger exist , to remind you that you must pay attention. Improper operation or failed to comply with the appropriate procedures may result in injury or death.

Symbol

Anytime when you operate or maintain instrument, you should observe the operation manual and warning symbol on the instrument, otherwise you will violate the designed safe standards and the correct usage of instrument, the result of accident caused by violating rules referred above won't be afforded by our company.

Leaving is banned when experiment is running

Be careful of microwave

Be careful of electric

Be careful

Must connect an earth terminal to the ground

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TOPEX Microwave Digestion System

Operation Manual

1 Safety guidance

Be sure to operate and maintenance the instrument strictly following the Operation Manual, PreeKem will not be responsible for any accident for the violation of the operation standards.

ATTENTION

1.1 Before using, please read all contents of this manual thoroughly and keep it carefully for further use.

DANGER

1.2 This instrument exists high voltages and microwave radiation. For uncommon troubles, the maintenance should be operated by the professional person.

ATTENTION

1.3 Never install the instrument inside the laboratory fume hood to avoid it being corroded by acid gas. The appropriate installation is to place the instrument out of the fume hood and insert the vent-pipe of the instrument into the fume hood.

DANGER

1.4 The outer shell of the instrument is connected with the ground pin of the electrical power socket on the laboratory wall through the ground pin of the tri-core electrical power plug. If the outer shell is electrified, the short circuit current will melt down the fuse inside the instrument automatically to avoid the person getting an electric shock. So the tri-core plug with reliable ground pin must be needed, and the electrical power capacity of the tri-core plug should not be lower than 220 V/16 A.

ATTENTION

1.5 In normal working conditions, this instrument will not generate electromagnetism interference, but it is not suitable to be put nearing other equipment which can be easily interfered by electromagnetism.

DANGER

1.6 Keep the door and the doorframe of the instrument clean. The instrument must not be started to work when there are some objects, such as paper or dishcloth being nipped between the door and the doorframe. Otherwise, the microwave may leak out and cause personal injury.

DANGER

1.7 Don’t start the microwave heating before the objects to be heated being put into the cavity of the microwave instrument, and also don’t use the metal-made container to avoid the magnetron being damaged due to operation without loading and large amount of microwave being reflected.

ATTENTION

1.8 The highest working pressure is 6 MPa, highest working temperature is 260°C. Never operate the instrument over that pressure or temperature to avoid damaging of vessels or other accessories. Furthermore, when using the instrument under the limited condition, follow the guidelines specified in the following figure.

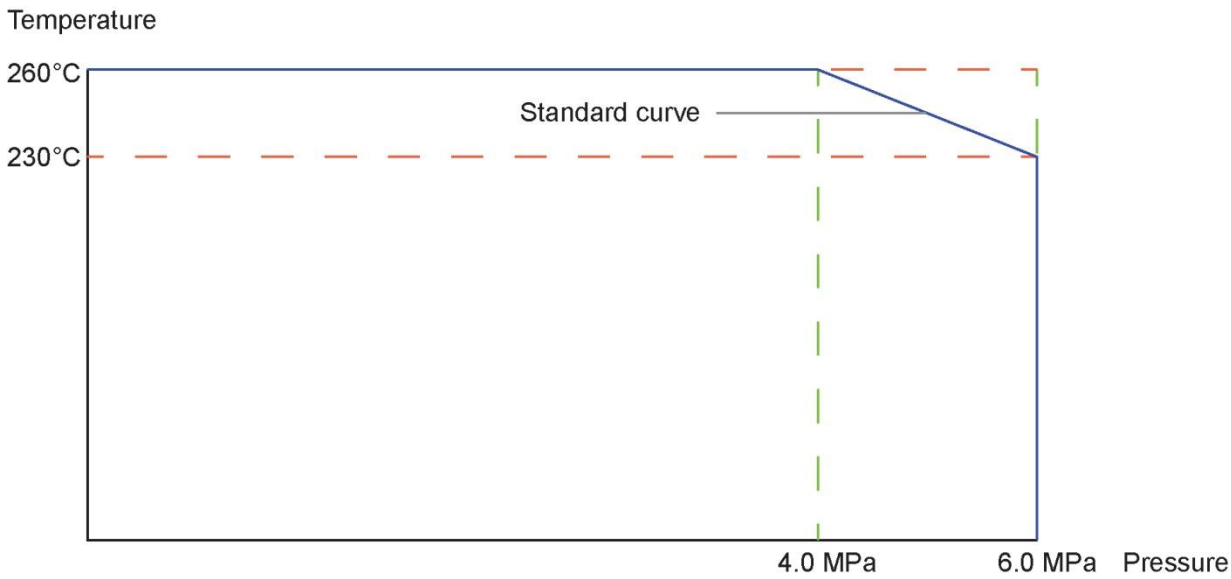


Figure 1-1: The relation between the working temperature and pressure under the limited condition schematic

DANGER

1.9 Before starting the instrument, make sure the door is closed. During the operation, the operator should not leave and should observe the running of the instrument. If abnormal conditions happened, press "Stop", and cut off power immediately.

ATTENTION

1.10 After the instrument being started, the operator should be more than 1.5 m away from the instrument to avoid potential injury to the operator.

ATTENTION

1.11 Don't leave when the instrument running to avoid damaging of vessels caused by localized heating and instrument faults.

DANGER

1.12 Microwave chemistry instrument is adapted to heating some polarity chemicals. Non-polarity or low polarity chemicals heated in microwave instrument for long time may damage the instrument seriously. Be sure never do that.

ATTENTION

1.13 Only those who has been trained by our company, has passed examination and obtained Certification of Instrument Operation can operate this instrument. We will not take the responsibility of instrument damage or personal injury caused by those operations without Certification of Instrument.

2 Instrument Introduction

2-1 Overview

TOPEX adopts the most up-to-date exterior designing, and high-tech wide LCD enables user to touch the colorful interface freely.

The real-time image technology makes the lab working convenient and comfortable. As a pioneer in microwave focusing technology, which is based on the design of roof reflection of microwave cavity, changing the field strength in a certain direction and focusing the energy onto the sample, greatly improved the reaction efficiency.

Figure 2-1: TOPEX Microwave Digestion System

2-1-1 Application field

The instrument can be applied to: accurate procedure control of high/ extremely-high pressure microwave digestion, air-tight organic solution extraction, air-tight organic synthesis etc.

At present, microwave extraction technique is applied in the preparation of environmental samples, biochemical, food, industry and the extraction of natural products and so on. The application of microwave synthesis covered the area of medicine, organic, material, polypeptide and so on.

This system has a very wide application range and can digest the following samples:

- Food, e. g. powdered milk, tea, coffee, flour, sea-mussel, beef, chocolate, alcohol, yeast, cocoa nut, fish, peanut ,capsule and so on.
- Cosmetics, e. g. lipstick, rouge, cold cream, hair dye and so on.
- Biomaterial, e. g. hair, blood, bio-tissue, bone dust and so on

- Agriculture, e. g. forage, corn flour, clover blossom, banana tree leaf, orange tree leaf, wood oar and so on.
- Environment, e. g. polluted mud, waste water, paint fragment, kiln ashes and so on
- Petrochemical Product, e. g. bitumen, gasoline, kerosene, Vaseline, petroleum and so on.
- Metal and Non-metal, e. g. coal cinder, iron ore, copper sulphide, firebrick, ceramic and so on.

2-1-2 Features of the instrument

- Special microwave resonant chamber
- Microwave cavity (56L)
- Cavity coated with as many as 5 layers modified Teflon PFA (modified FTFE)
- Dual-magnetron stagger structure
- Independent floating cushion locking system
- Genuine non-pulse continuous microwave output
- High-frequency closed-loop feedback control
- Large LCD screen display
- Platinum resistor temperature measure and control system
- High precision high temperature melt pressure measure and control system
- Strong convective fast-cooling system

2-2 Hardware introduction

The following paragraphs will introduce you about positions and functions of core component.

2-2-1 Front

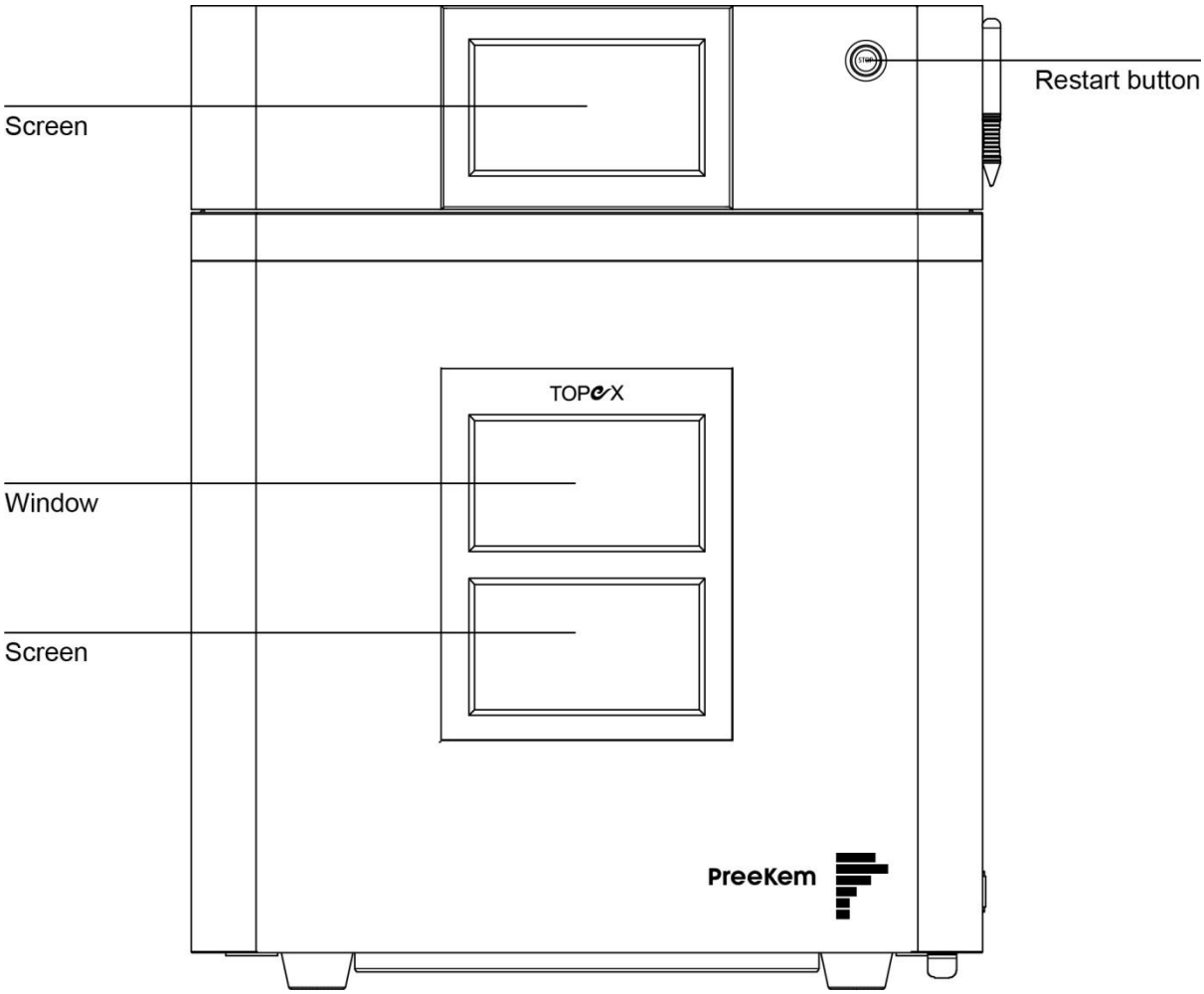


Figure 2-2: TOPEX Microwave Digestion System Front

2-2-2 Right

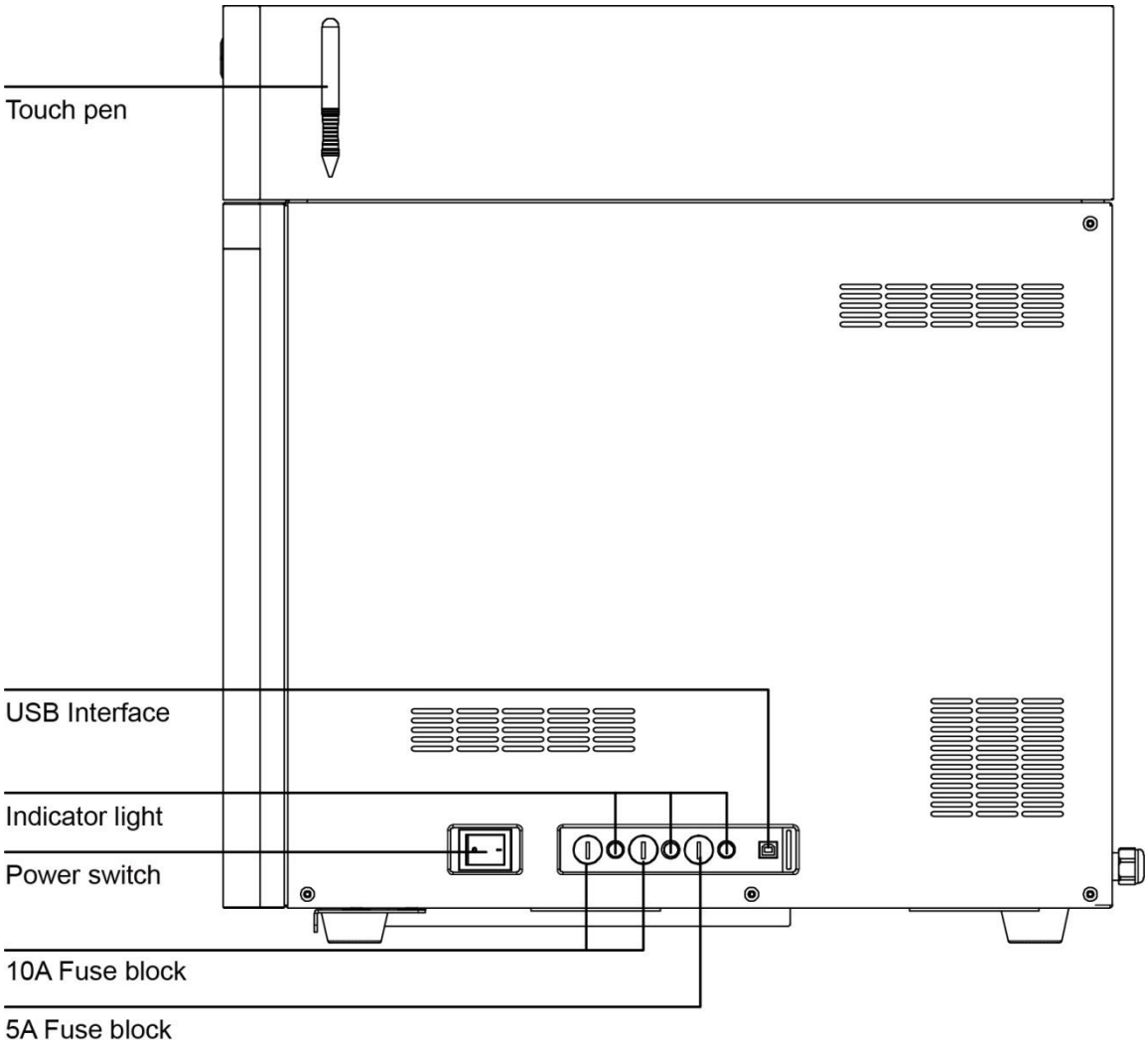


Figure 2-3: Switch and Fuse schematic

2-2-3 Back

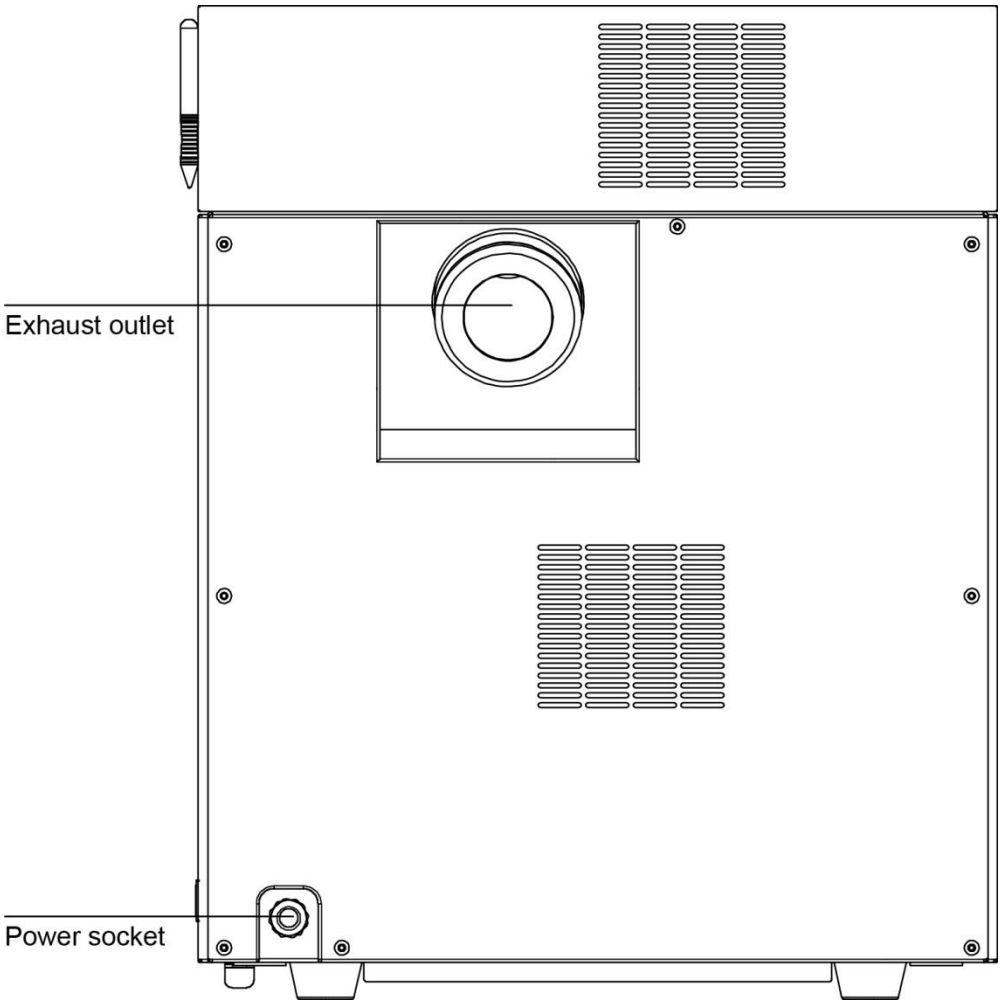


Figure 2-4: Power socket and exhaust outlet schematic

2-3 Specification

Power requirement: AC220-240V, 50 Hz, 16 A

Maximum output power: 1800 W

Power rating: 3200 W

Microwave power adjust: non-pulse continuous microwave, high-frequency closed-loop feedback control

Microwave frequency: 2450 MHz

Microwave leak power density: ≤0.5 mw/cm^2

Size of cavity: 400mm width×380mm depth×350mm height, net volume 56 L

Electric motors: speed 5 r/min

Power setting: Setting number of vessels by touch screen, and the heating power will be regulated automatically by the program

Timer: The time set by touch panel, showed on the screen, calculated by the timer

Timing range: Single step 0-99 min

Pressure measure and control system

- Measuring range: 0-10MPa
- Special pressure sensor, measuring precision ±0.01MPa
- Pressure control stability ±0.05MPa, display the pressure of vessels in real time

Temperature measure and control system

- Temperature measuring range: 0-300°C
- Temperature measuring precision: ±1°C
- Temperature control stability: ±1°C

Safety measures

- The microwave heating is controlled in real-time by the temperature and pressure inside the vessels, the heating will be auto stopped when exceeding the setting value.
- The safety diaphragm or skirt in seal lid will be broken for pressure release if the pressure can't be stopped rising after the microwave heating stopped. If those two safety measures didn't happen because of improper operation, special designed frame structure will vertically explode to reduce the horizontal impact.
- Self-locked safety door: The door will spring out to reduce the impact of the buffer if vessels explode when microwave heating not suitable for digestion and cause a sudden or explosive chemical reaction. The door, made of high strength stainless steel and explosion-proof glass visible window, can stop fragments effectively and avoid personal injury.

Oven chamber ventilation system: Adopt centrifugal fans, maximum airflow capacity 5 m^3/min

Working environmental temperature: 5-40°C

Working environmental relative humidity: 15-80%

Size of mainframe: 550 width×650mm depth×655 mm height

Weight: 72kg

3 Use of instrument

3-1 Installation

The following paragraphs will present you how to install the components and to place the instrument.

3-1-1 Placement of instrument

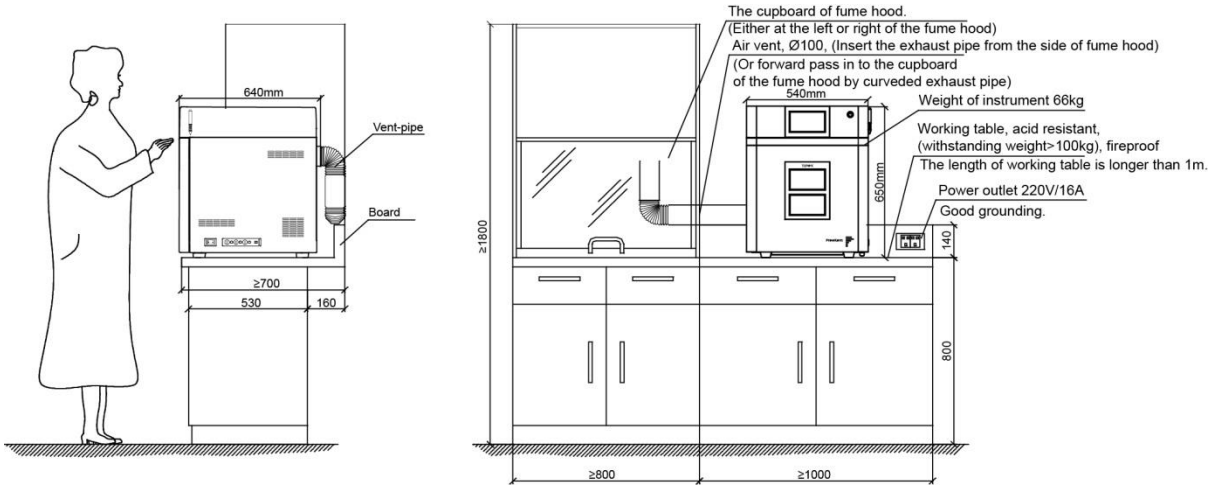


Figure 3-1: Installation of instrument schematic

Put the main instrument on the working table, which is able to withstand at least 100 kg, in the laboratory nearing the fume hood. The distance between the main instrument and the fume hood should not exceed 1 meter, make sure the end of the exhaust pipe can be put into the fume hood. The back cover of the main instrument should be 10 cm away from the wall for ventilation. On the wall nearing to the main instrument, there should be a tri-core electrical power socket. The power voltage should be AC 220V-240V. The socket should have the capability to output 16 A current.

The surrounding temperature of the laboratory should be 5-40°C, and the humidity should be 15-80%.

3-1-2 Installation of the exhaust duct

Related Parts:

- Mainframe
- Exhaust pipe (09fa001)
- Hoop (00dy002)

Flow:

1. Adjust the hoop with flat-blade screwdriver in order to fit exhaust pipe.
2. Insert exhaust pipe into hoop.
3. Connect the exhaust pipe installed with hood to the exhaust hole at the back of the instrument.

ATTENTION **There should be no leak or gap after the exhaust pipe installation in order to avoid leakage of acid gas.**

4. Tightly screw the regulating screw of the hoop to fix the exhaust pipe.

ATTENTION **Ensure the exhaust pipe will not fall off.**

3-1-3 Power connection

Related parts:

- Mainframe
- Power line (00cd304)

Flow:

1. Connect the power supply. Make sure the power is grounded. The grounding resistance should be equal to or smaller than 4 Omega. Also the electric capacity should not lower than 220 V/16 A.

ATTENTION **Ensure the switch is off before connecting to the power supply.**

ATTENTION **Please cut off the power if you don't use the instrument for a long time.**

3-1-4 The Installation of the temperature sensor assembly and air-guide tube assembly

ATTENTION **Please follow the following steps to install the temperature sensor assembly and the air-guide tube assembly for the KJ-100 and KJ-160 vessel, there is no need to install the two parts for GT-400 vessel.**

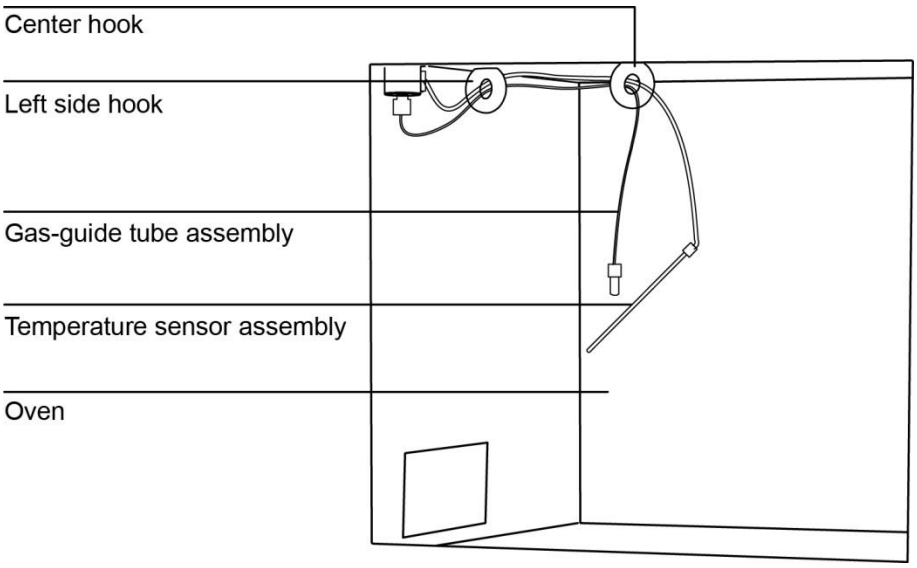


Figure 3-2: Installation of sensors schematic

Related parts:

- Temperature sensor assembly (26tj203/26tj209)
- Air-guide tube assembly (26tj204/26tj210)
- 10-16 adapter (08ca106)

The matching table for temperature sensor assembly and air-guide tube assembly with vessels:

	KJ-100 vessels	KJ-160 vessels
Temperature sensor assembly (26tj203)	√	×
Temperature sensor assembly (26tj209)	√	√
Air-guide tube assembly (26tj204)	√	×
Air-guide tube assembly (26tj210)	×	√

Illustration: “ √ ” means suitable, “×” means not suitable.

ATTENTION **When using temperature sensor assembly (26tj209) with KJ-100 vessels, you should install adapter (08ca106) firstly.**

Flow:

1. Insert the connector of air-guide tube assembly into the socket at the top of cavity and spin it tightly.

ATTENTION **Air-guide tube assembly (26tj210) has two different ends, please insert the bigger one into the socket at the top of cavity.**

2. Pass the smaller end through the side hook and center hook
3. Pass the probe of air-guide tube assembly through the side hook.
4. Make sure the red point on temperature sensor assembly align with the red point on socket.
5. Install the cooper bush onto the cooper seat.

ATTENTION **Do not entangle the temperature sensor assembly and air-guide tube assembly**

3-2 The use of the instrument

The following paragraphs will introduces you the basic operations of the instrument, including turning on/off the instrument, open the safety door.

3-2-1 Start /Stop the instrument

Press the power switch on the right side to start the instrument. Press the power switch once again to stop the instrument after the experiment finished. There is an indicator light in the switch. It lights when connected to the power.

3-2-2 Open/Close the safety door

Click the open key, the door will open automatically.

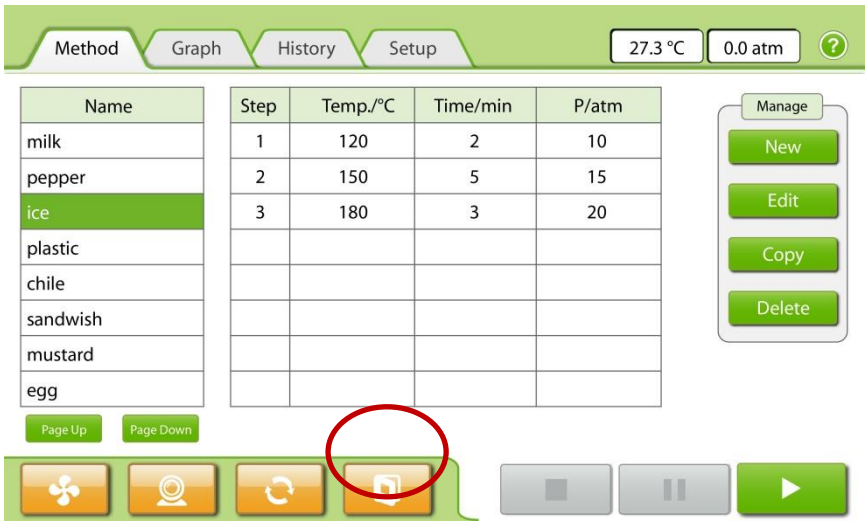


Figure 3-3: Open key

Push the safety door to close it.

ATTENTION **Make sure the door hook is locked, and the door can't be pulled out after closed.**

ATTENTION **The door can only be opened when the temperature is under 80°C and the microwave is shut off.**

3-2-3 Turntable control

Click the turn key, the rotor will turn forth and back.

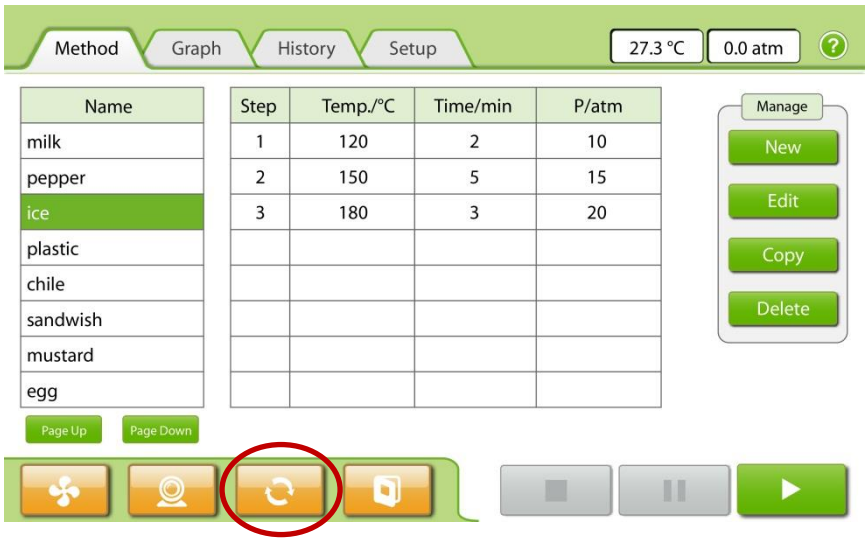


Figure 3-4: Turn key

If you need to stop the running of the rotor, please press the turn key again to stop it.

3-2-4 Top fan control

Click the fan key, the fan will start to work.

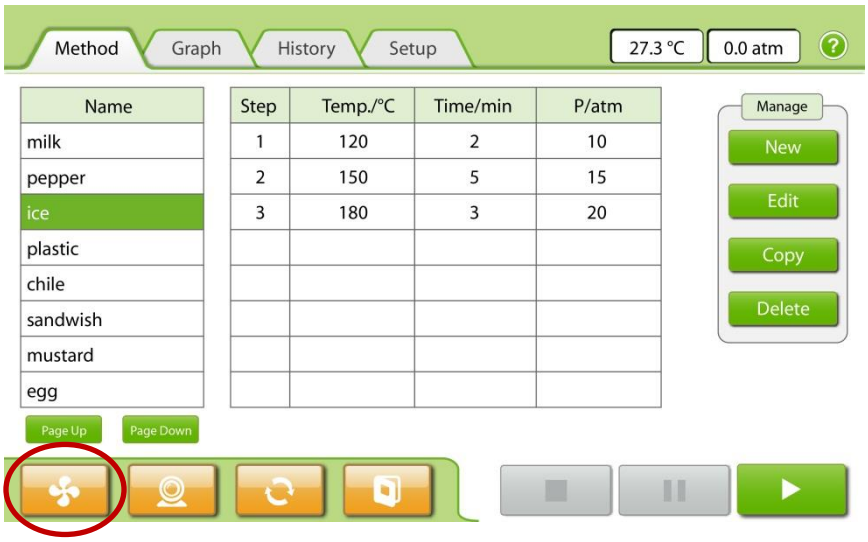


Figure 3-5: Fan key

If you need to stop the working of top fan, please press the fan key again to stop it.

3-2-5 Monitor system control

When you press the monitor key, the screen will start to work, the working conditions can be observed via the LCD.

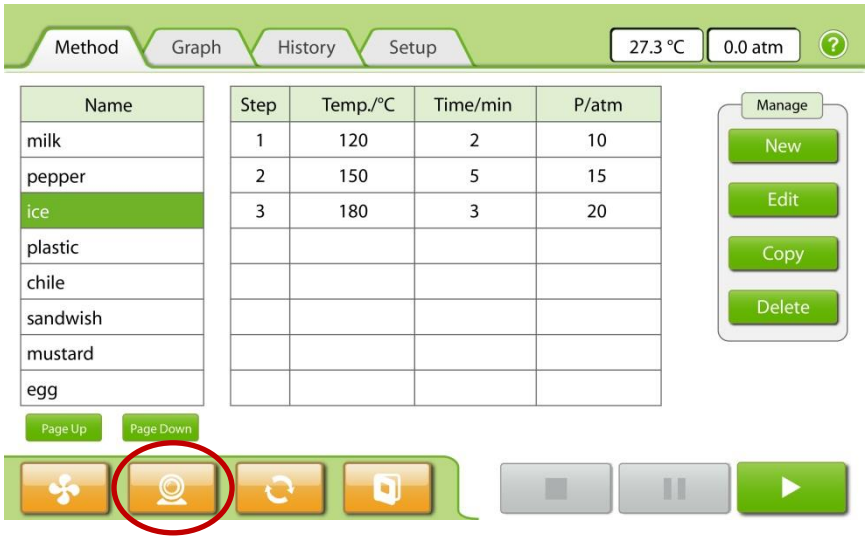


Figure 3-6: Monitor key

If you need to turn off the screen, please press the monitor key again.

3-3 The use of the software

The following content will present you the function and the operation of the software in detail.

The words related to touch pen.

- Click: tap the screen and open the program.

3-3-1 Interface illustration

Figure 3-7: Software interface illustration

Menu bar: It contains method, graph, history and setup. Real time temperature and pressure will be displayed on the right of window. You can also ask for help via “?” key.

Content: It displays core content of software and details of parameters.

Function key: It includes fan key, monitor key, turn key and open key.

Microwave control key: It used for controlling microwave including start key, pause key and stop key.

3-3-2 Illustrations of keys for main programs

Button	Name	Illustration
Method	Method	Set and save the reaction method
Graph	Graph	Display the graph and parameter value
History	History	View the recent experiment record and graph.
Setup	Setup	Basic setup, calibration, trouble-shooting

	Door	Open the cavity door
	Fan	Open or close the cooling fan
	Monitor	Open or close the monitoring system
	Turn	Start or stop the turntable
	Start	Start the microwave
	Pause	Pause or restart to work
	Stop	Stop heating and go the down to cooling process
2	Choose the number of vessel	Choose the number of vessels
	Help	Get the help information
Page Up	Page up	Turn back to previous page
Page Down	Page down	Turn to next page
Page turning	Page turning	To display the record list of history
New	New	Create new method，including the name and the parameters
Edit	Edit	Edit the chosen method
Copy	Copy	Copy the current method
Delete	Delete	Delete the current method
Review	Review	View the selected record graph

ATTENTION **You can't operate the interface, before the grey key becomes to colorful one.**

3-3-3 Method set-up

Function description: The software management of TOPEX available for add, edit, copy, delete of the method.

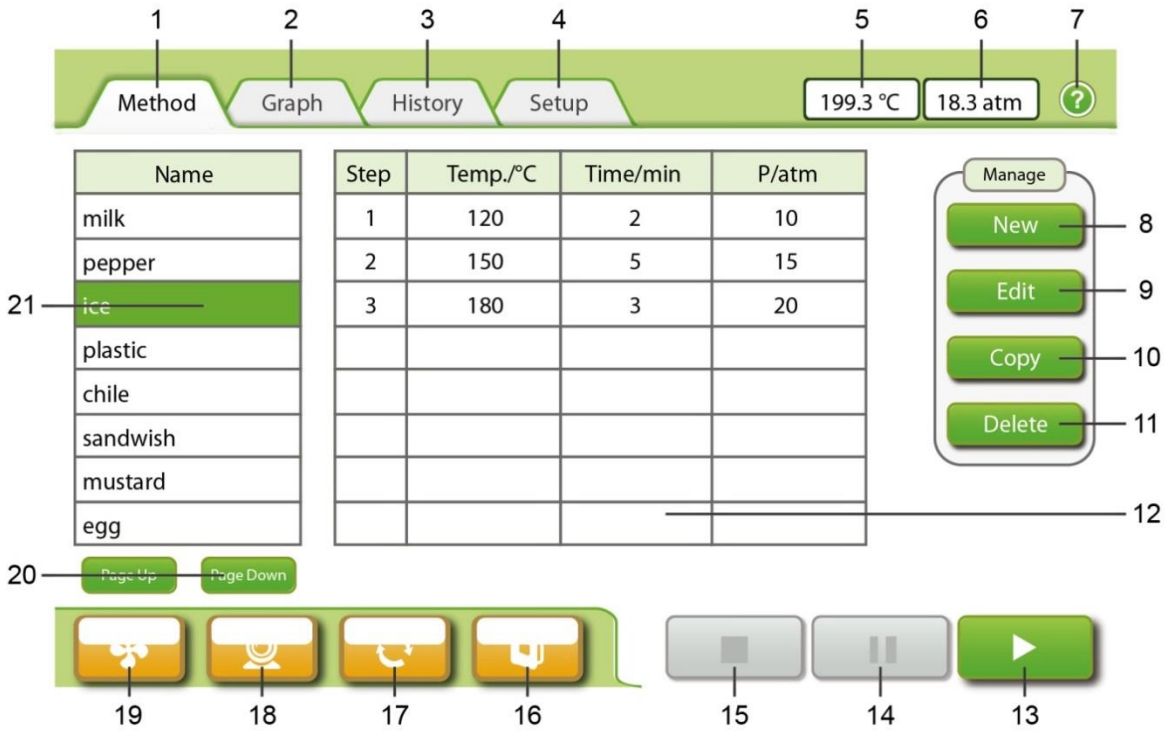


Figure 3-8: The interface of method

1. Method tag: Enter into interface of method set-up
2. Graph tag: Enter into interface of graph.
3. History tag: Enter into interface of history.
4. System tag: Enter into interface of system setup.
5. Display the real time temperature.
6. Display the real time pressure.
7. Help key: Get help and more information when you click this key.
8. New key: Create a new method
9. Edit key: Edit and modify parameter values of selected method.
10. Copy key: Copy the selected method.
11. Delete key: Delete the selected method.
12. Display parameter values.
13. Start key: Click this key and you will see a pop-up prompt window. Set the number of vessels, then start the microwave working click the enter key on the pop-up window.
14. Pause key: Pause the working of microwave. Restart it when click start key again.
15. Stop key: Stop the working of microwave.
16. Open key: Click it to open the safety door.
17. Turn key: Click it to start or stop the turntable
18. Monitor key: Click it to open or close the monitoring system
19. Fan key: click it to start or stop the cooling fan.
20. Page up key and page down key: Click it to turn up or down the page.
21. Display the method name.

Interface of setting method name

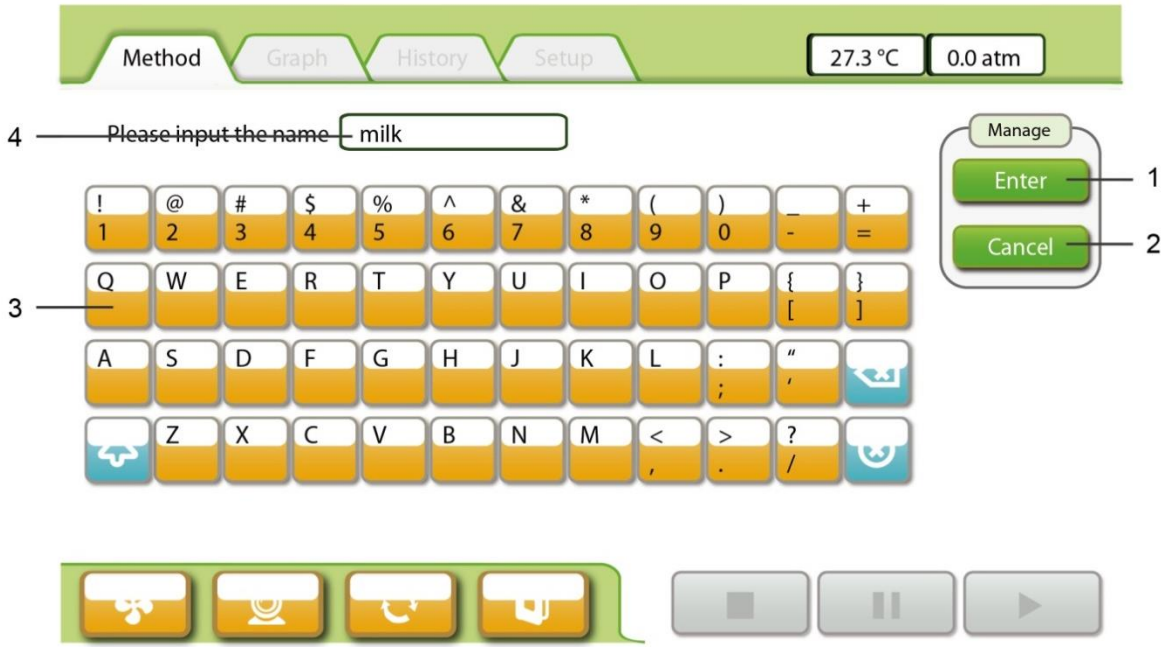


Figure 3-9: The Interface of method name set-up

1. Enter key: Click it to save the name of method and enter into interface of parameters set-up.
2. Cancel key: Return to the interface of method.
3. Input keyboard: Use keyboard to edit the information.
4. Method name: Display the method name.

Interface of setting parameters

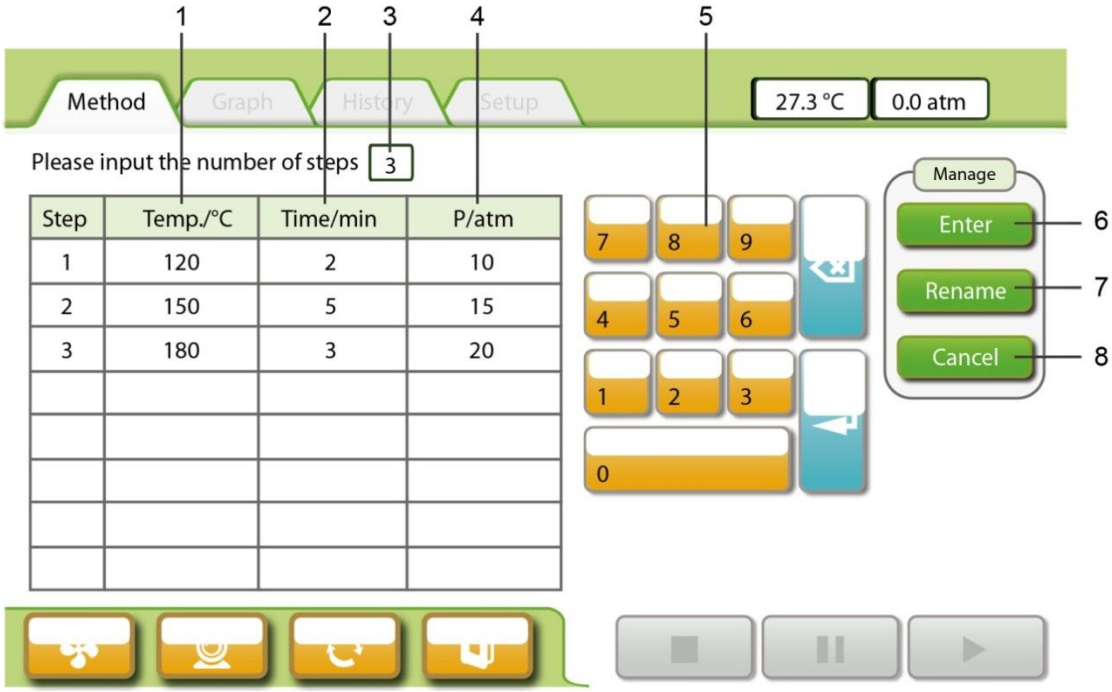


Figure 3-10: The interface of parameters set-up

1. Temperature: Reaction temperature, maximum 260°C.
2. Hold time: Holding time, maximum 99 min.
3. Steps: Total reaction steps, maximum 8 steps.
4. Pressure: Limit pressure, maximum 6 MPa.
5. Numeric keyboard: Input the parameter values through the key on the screen.
6. Enter key: Click it to save the method and enter into interface of method.
7. Rename key: Click it to save the method and return to interface of method name set-up.
8. Cancel key: Return to interface of method.

Operating method:

Add method

Click new key on the function zone to add a new method.

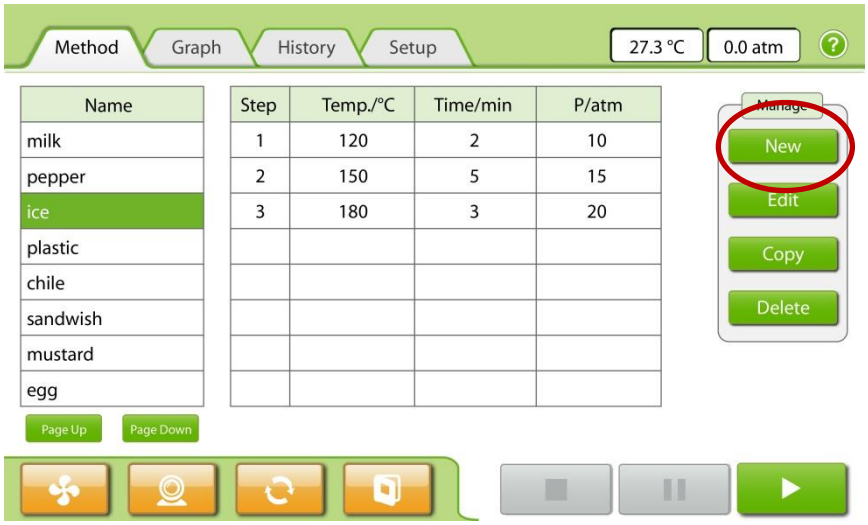


Figure 3-11: Add method

Enter into the interface of method name set-up, input the method name you need with keys on the soft keyboard.

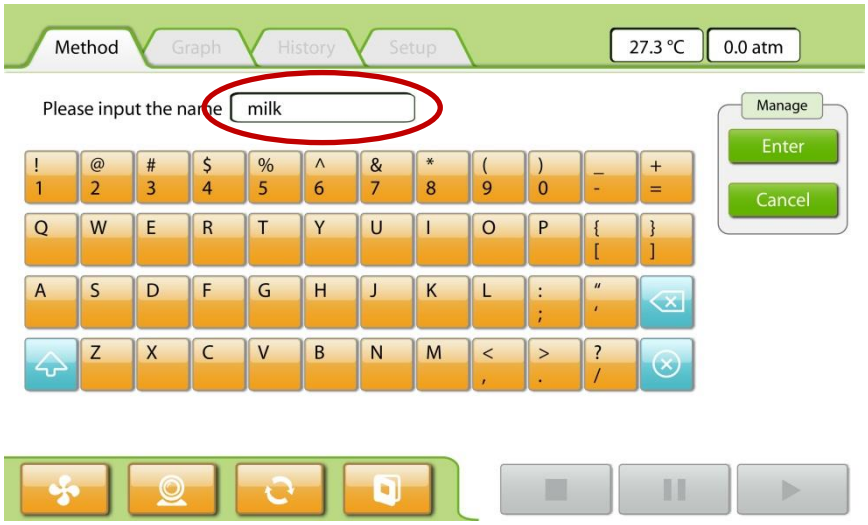


Figure 3-12: The methods name setup

ATTENTION **The method name must be between 1 and 14 characters. Over 14 characters, clicking the key is invalid.**

The Illustration of the function key on the soft keyboard:

Key	Illustration
	Caps lock
	Delete the last character in the input box
	Delete all characters in the input box

Click enter key on the function zone you can save method name.

Figure 3-13: Method name set

Enter into the interface of setting parameters, setting the reaction steps via the numeric key.

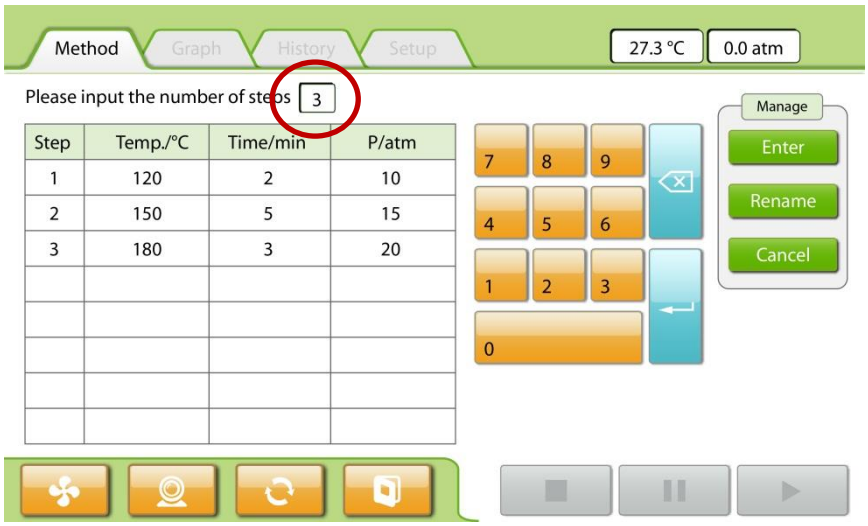


Figure 3-14: Step set-up

Click the numeric key to input the number of steps. The selected step will also be shown on the parameter table.

ATTENTION **Step range is from 1-8. the limit is shows on the top of the numeric key in red.**

The illustration of the function keys:

Key	Illustration
	Delete the last character in the input box
	Enter into next parameter set-up

Move cursor to the input box of temperature, and you can click the number key to input the temperature.

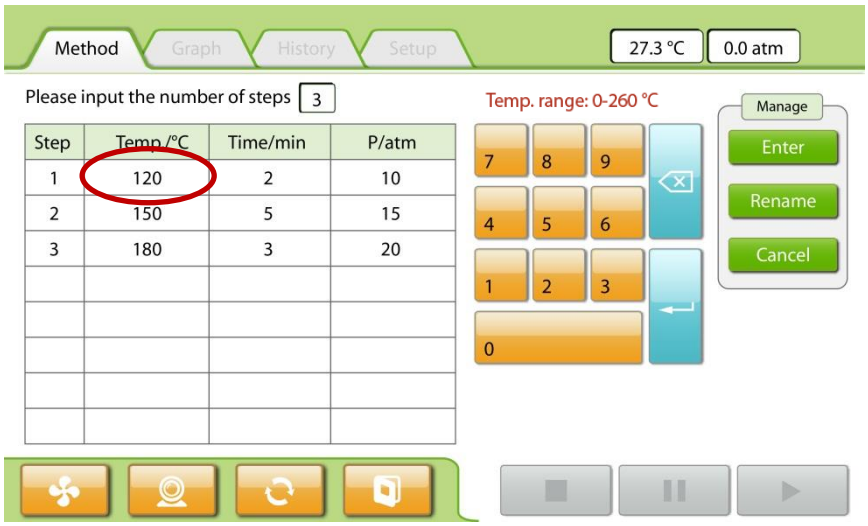


Figure 3-15: Temperature se-up

ATTENTION **The temperature setting must be between 0°C and 260°C, the precision setting is ±1°C. The requirement of setting temperature is displayed above the soft keyboard.**

The cursor moves to the input field of time, and you can click the number key to input the hold time.

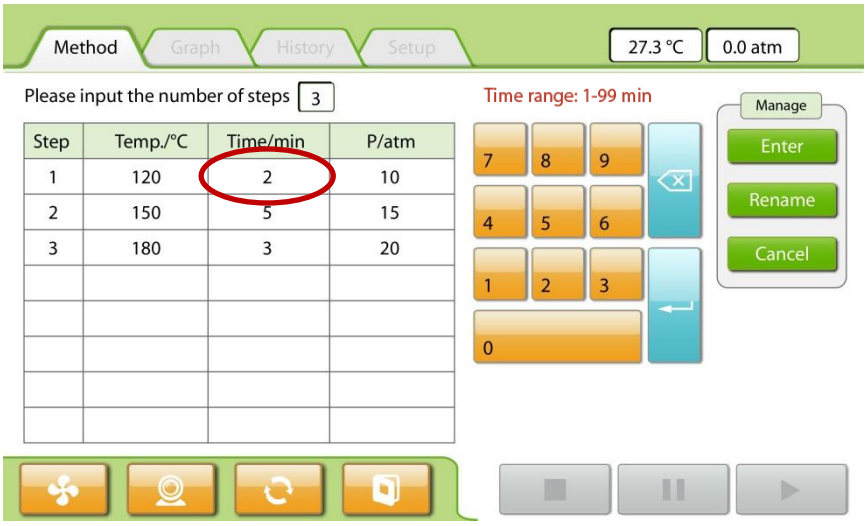


Figure 3-16: Holding time set-up

ATTENTION **Hold time setting must be between 1 minute and 99 minutes, the precision set-up is 1 minute, the requirement of setting hold time is displayed above the soft keyboard.**

Move the cursor to the input box of pressure, and you can click the number key to input the pressure.

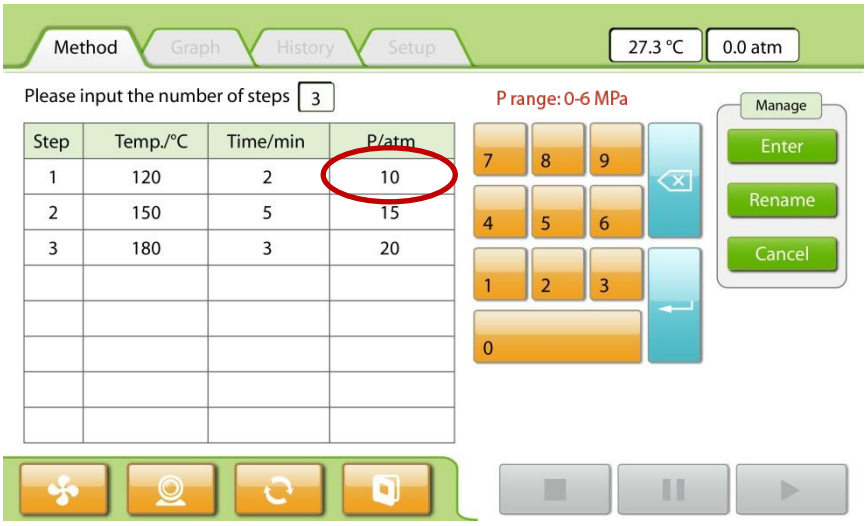


Figure 3-17: pressure set-up

ATTENTION **Pressure setting must be between 1 MPa to 6 MPa, the precision is ±1 MPa, the requirement of setting pressure is displayed above the soft keyboard.**

ATTENTION **The pressure setting range is different based on different pressure unit.**

Click the enter button on the function zone, you can finish setting.

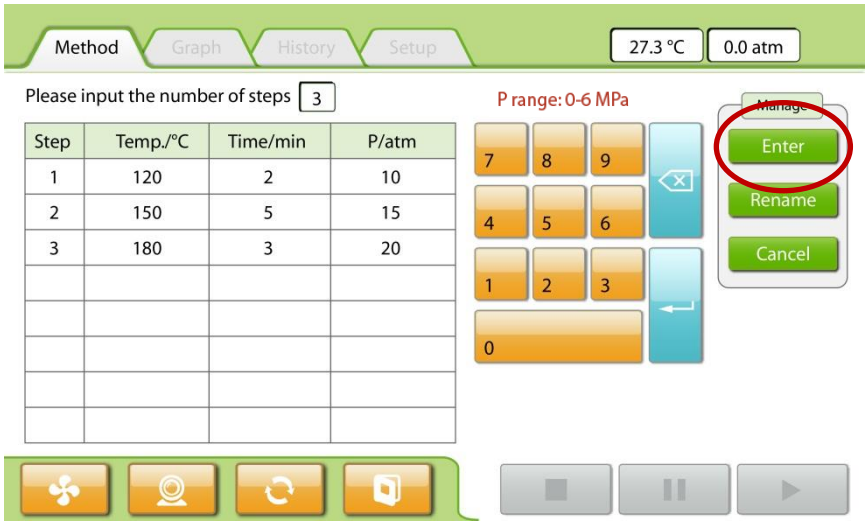


Figure 3-18: Method set-up finished

ATTENTION

If there is no response when press the enter key, please make sure the method is correct. The heating process should stick to the principle of the Gradient ascent, which the temperature value set-up should be higher than the previous one.

When setting the parameters, click the rename key on the function zone and you can set the method name again.

Figure 3-19: Rename the method

Click the cancel key to back to the method interface.

Figure 3-20: Exit the method setup

ATTENTION **All the input information will be deleted after click the cancel key.**

Rename the method

If you want to enter an existed method name, there will be a hint to tell you that the method name has existed. you should modify the method name.

Figure 3-21: Method name exists

Rename the method, if the name is valid, click the enter key to the interface of parameter set-up.

Edit method

Choose the method, click the edit key to revise it.

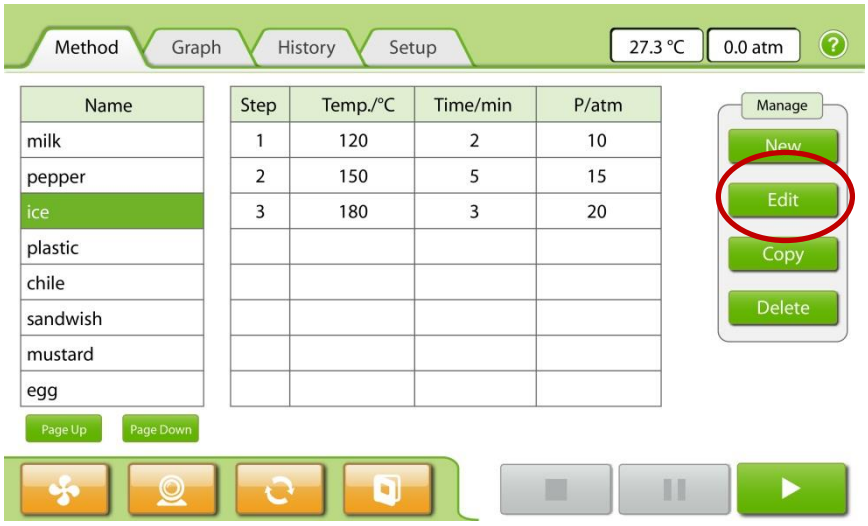


Figure 3-22: Edit the method

Enter into the method set-up interface, where you can edit the parameter by directly input the parameter and press the numeric key to edit it.

When finished, click the enter button to save the method.

Copy method

If you need to copy a method, please select a method you need and click copy button on the function zone.

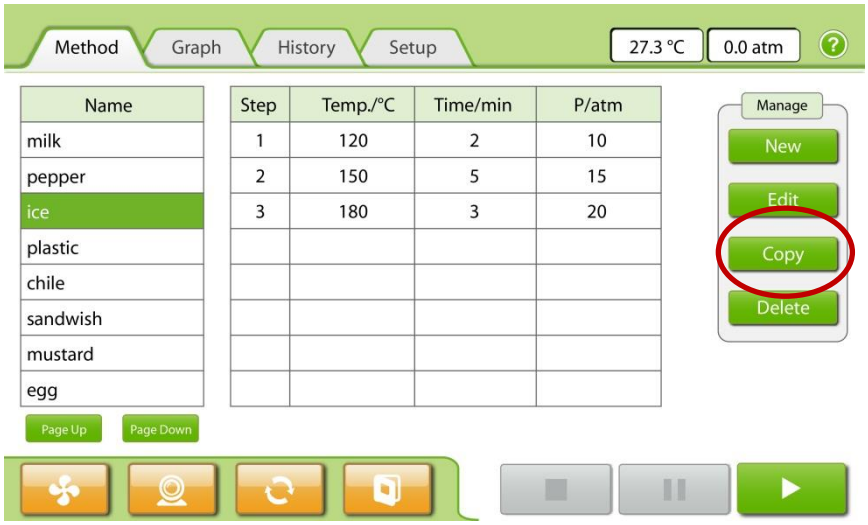


Figure 3-23：Copy method

There is a hint window to tell you to confirm your copy step on the screen.

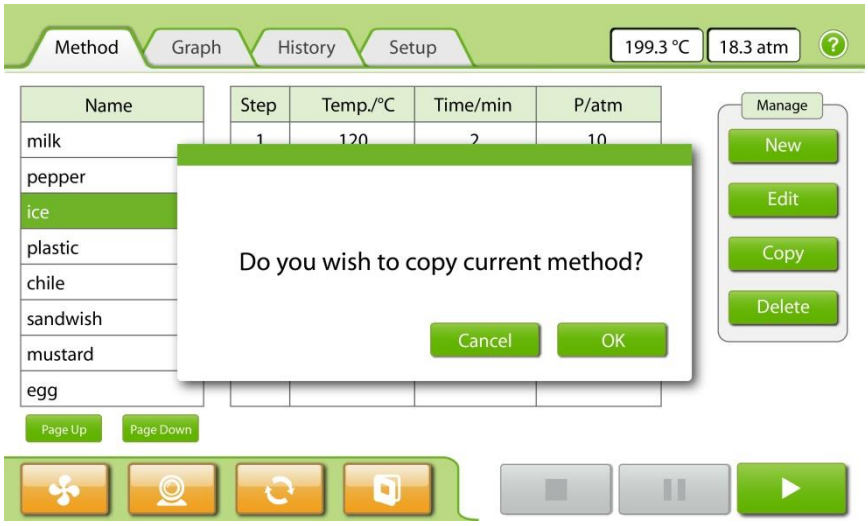


Figure 3-24: Copy confirm window

The method will be copied when you click the OK key. Which the duplicated method is named COPY- is located as first item in the list.

ATTENTION **The first 14 characters will make up as the method name.**

If the selected method already has been copied, further copy can't be operated. Click the cancel key back to the method set-up interface.

Delete method

If you need to delete a method, please select a method you don't need and click delete key on the function zone.

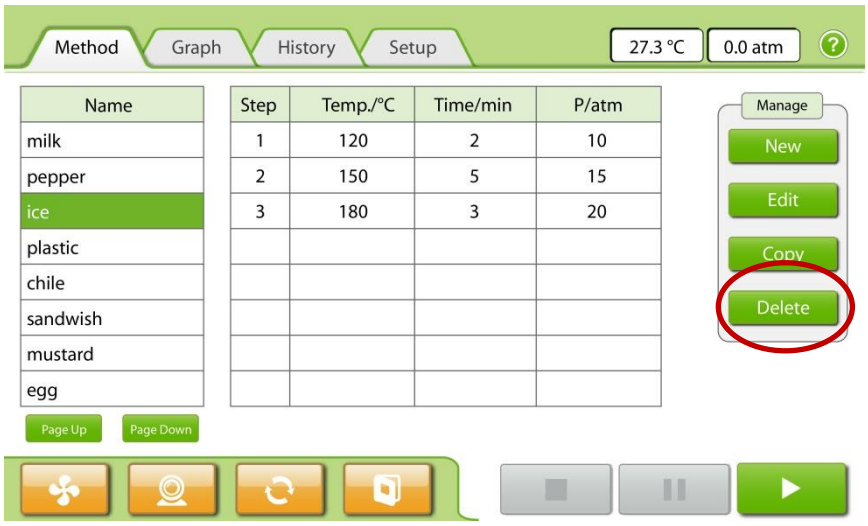


Figure 3-25: Delete method

Method delete confirm window.

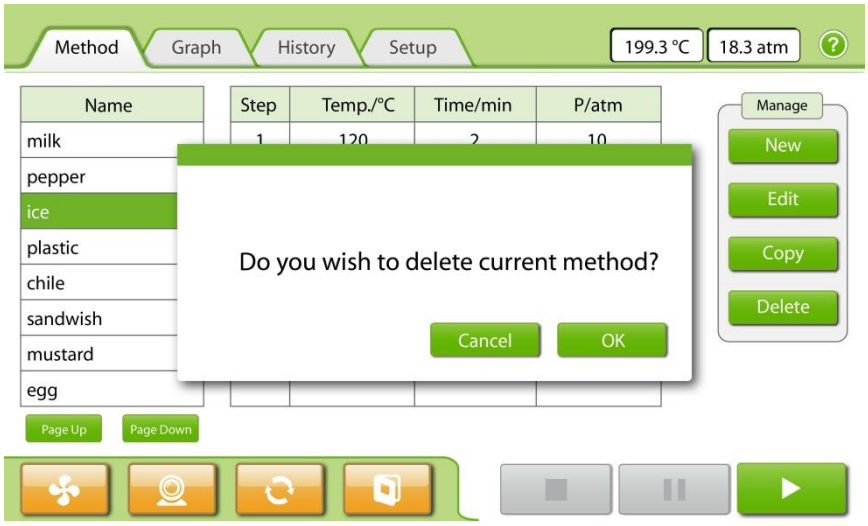


Figure3-26: Method delete confirm window

Click the Ok key to delete the selected method.

Click the cancel key back to the method interface.

3-3-4 Microwave heating

Function: Control the microwave reaction process including start, pause and stop.

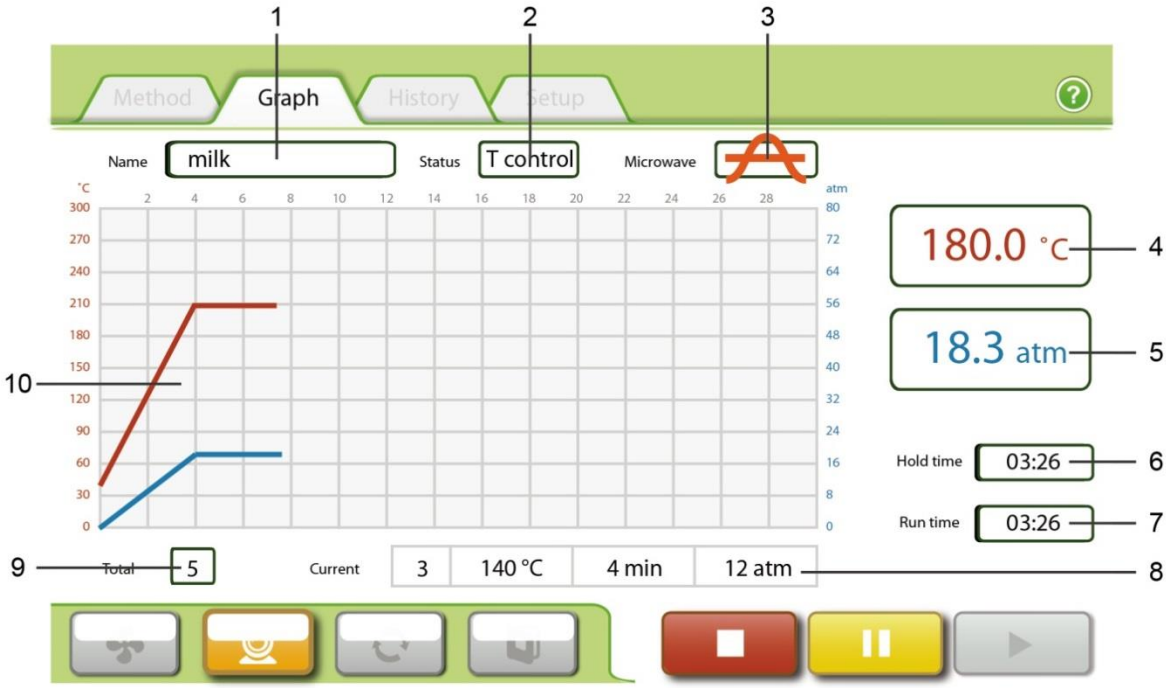


Figure 3-27: Running interface

The curve displays in real-time on the screen automatically after starting the microwave

1. Display the current method name.

2. Operating status, display the status in real-time, including 8 status, temperature rising, temperature holding, temperature control, pause, cooling, finish, stop and fault.

3. Microwave symbol, which indicate the status of the microwave operating, the straight line indicates no microwave output, the variation waveform indicates the microwave is working.

4. The red number shows the real-time temperature, the precision is ±0.1°C.

5. The blue number shows the real-time pressure, the precision will be different based on different unit. When choosing atm as the unit, the precision is ±0.1atm. when choosing MPa as the unit, the precision is ±0.01MPa, when choosing psi as unit ,the precision is ±1psi, when choosing bar as unit, the precision is ±0.1bar.

6. Temperature holding time, the timing starts from the actual measured time reached the setting temperature, when the temperature holding time over, the reaction will enter to next step. The holding time will be displayed as Hour, Minute, Second, such as 00:00:18.

7. Operating time, The operating time will be started after press the Start button and will be stopped when the cooling process finished（Cooling down to 80° C） The operating time will be displayed as Hour, Minute, Second, such as 01:12:23.

8. Current step, showing the current detailed parameters, including step number, target temperature, holding time, pressure.

9. Display number of steps.

10. Working curve, including the temperature curve and the pressure curve.， The temperature curve set time as abscissa, set temperature as ordinate. The time range for a screen is 30 minutes, if exceed 30 minutes, the curve will auto short in half, the maximum time line is 960 minutes. The temperature ordinate range is 0-300° C。The pressure curve set time as abscissa, set pressure as ordinate , the time range for a screen is 30 minutes, if exceed 30 minutes, the time will auto short in half, the maximum time line is 960 minutes. The pressure ordinate range is 0-8MPa.

Operating method:

Start microwave

Press the start key to operate the selected method.

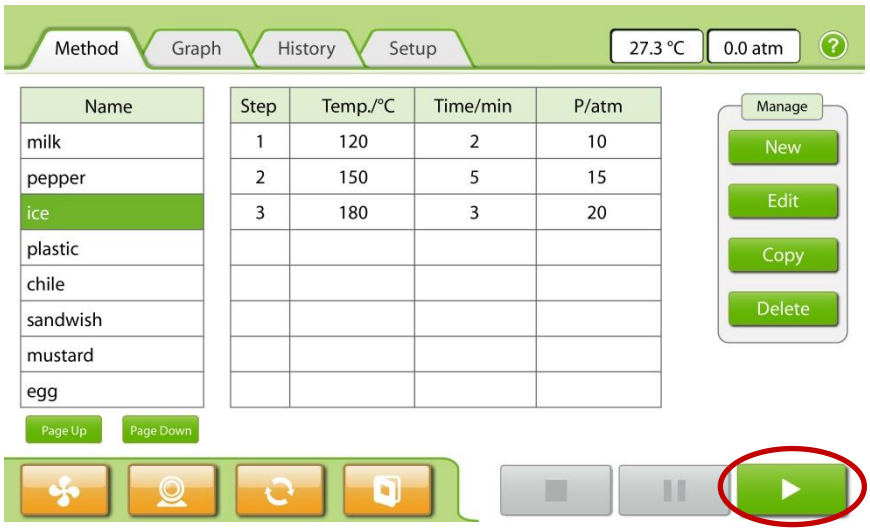


Figure 3-28: Start key

Vessel number selection window.

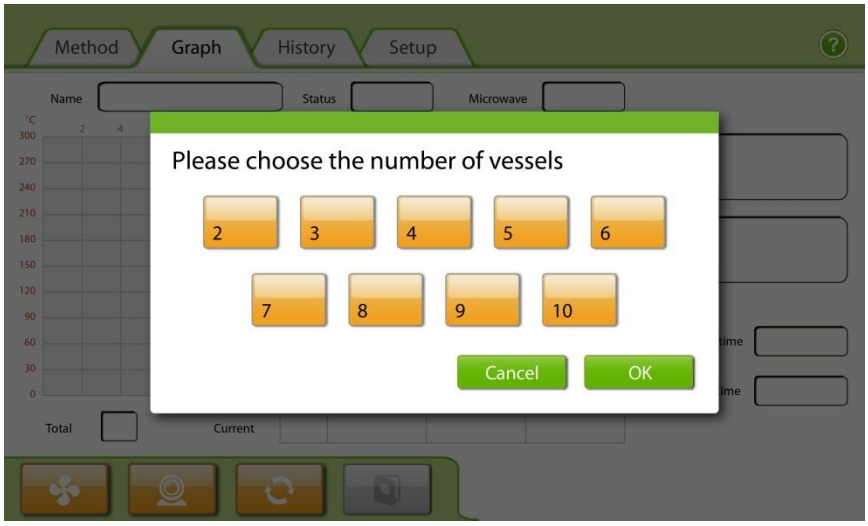


Figure 3-29: Vessel number selection window

Choose the desirable number.

ATTENTION

The available vessels are different according to different rotors. The above figure shows the available vessel numbers are from 2-10 for KJ-100's rotor. While for KJ-160's rotor, the Choice vessels numbers are 2-15. If for GT-400's rotor, the Choice vessel numbers are from 8-40.

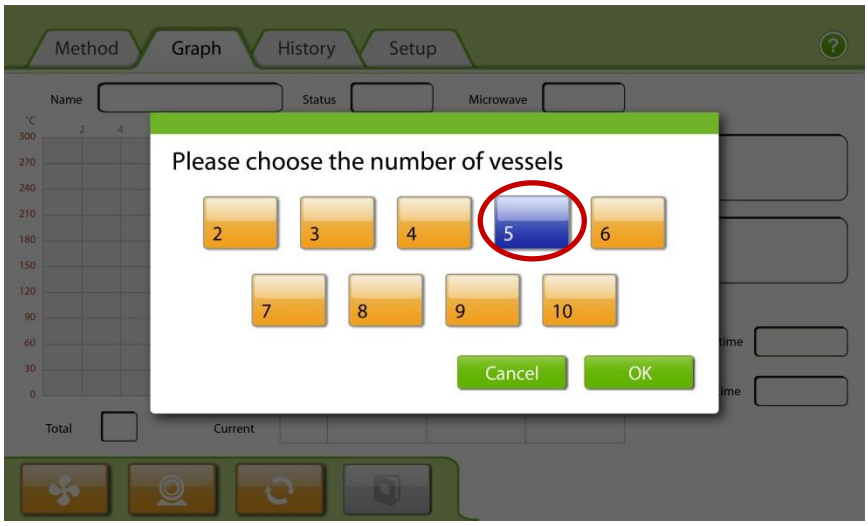


Figure 3-30: Choose the vessel numbers

Press the Enter button of the lower right corner to start the microwave and enter into the curve interface.

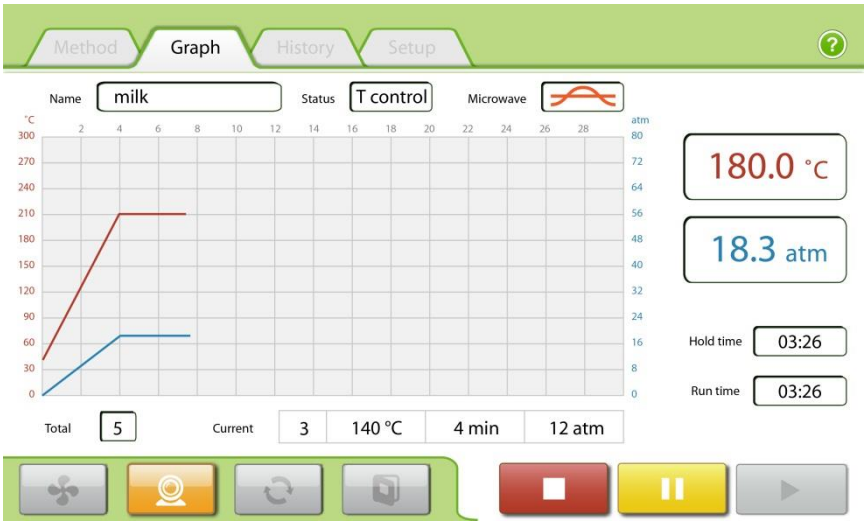


Figure 3-31: Operating interface

The system will auto draw the curve of temperature- time, pressure-time and display them on the screen. On the right side of the operating interface, it shows the actual temperature value, pressure value, holding time and operating time.

Pause microwave

Press the pause key on the lower right corner of the interface.

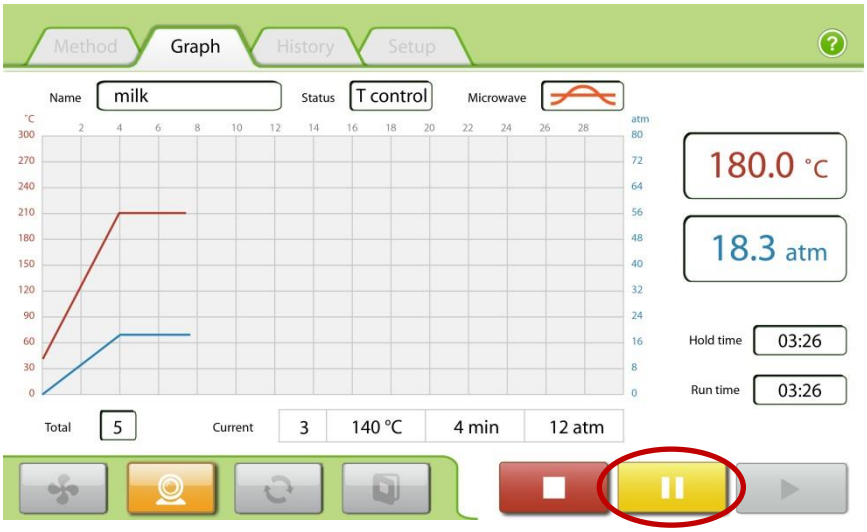


Figure 3-32: Pause microwave

Pause the operation, to restart the operation, please press the start key.

Stop operating

Press the stop key on the lower right corner of the interface.

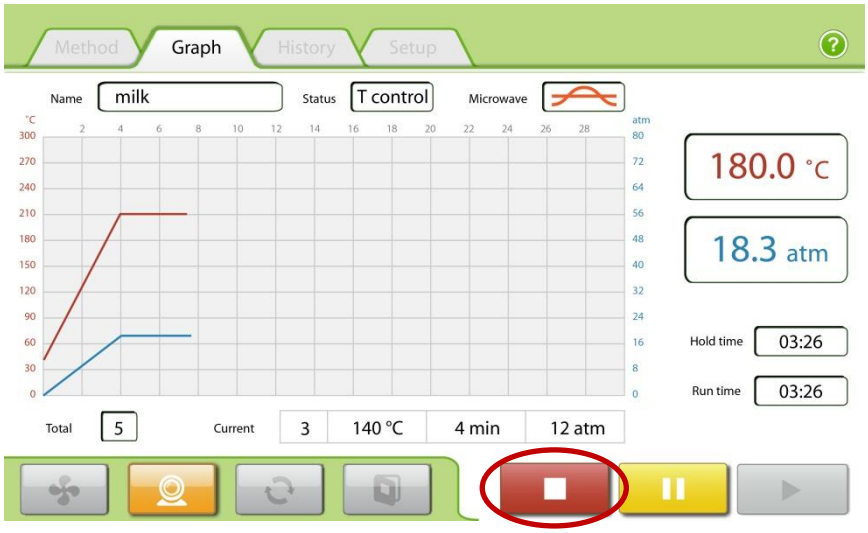


Figure 3-33: Stop microwave

Error message

During the operation, if the instrument detects out the rotor function is abnormal, the screen will pop up an error message and will send out a beep warning sound.

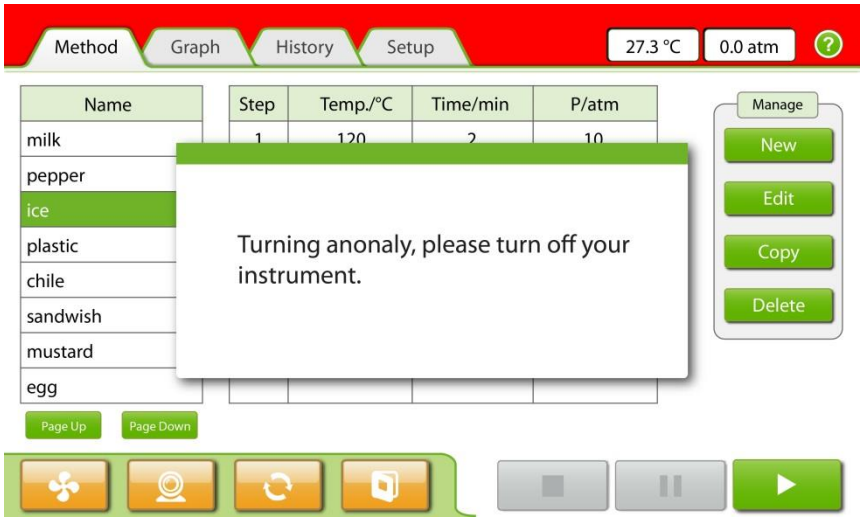


Figure 3-34: Rotor abnormal

If abnormal occur, please refer to the chapter of Trouble Shooting to tackle this problem, and then restart the instrument to try again, if still not working please contact the PreeKem service engineer.

During the operation, if the system detects any abnormal phenomenon, it will send out a warning sound and an error message will appear on the upper right corner.

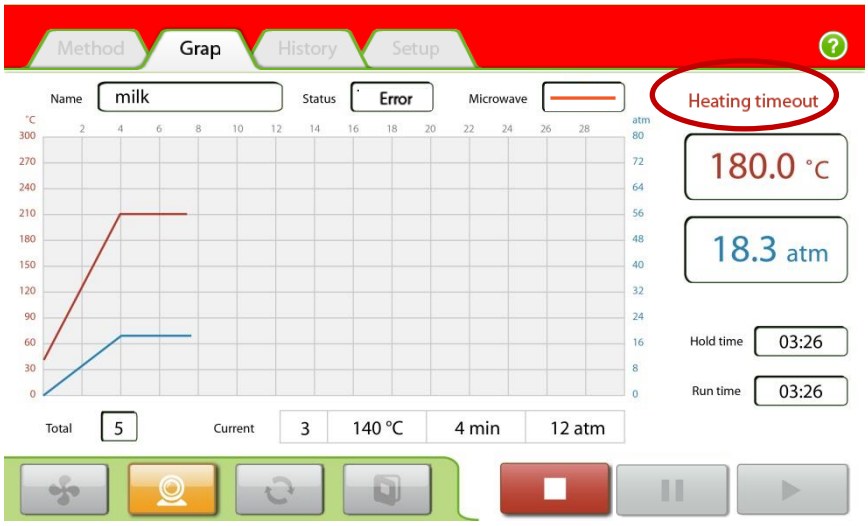


Figure 3-35: Over heating

Table for error message to related reasons:

Error message	**reason**
Temperature anomaly	Temperature rising too slow
	Temperature rising too fast
	Temperature jump substantially
Pressure anomaly	Pressure exceeds the Maximum working pressure
	Control the pressure more than 2min in succession
Door open	The cavity door is open
Heating timeout	The temperature hasn't risen to target temperature within 10 minutes.

Under the abnormal situation, press the stop button to shut the warning signal, for the detailed trouble-shooting, please refer to the related chapter- Trouble Shooting.

To start the microwave, the system will auto detects the rotor condition and the method, after the detection, if the rotor is matched with the method, the system will start to work, if not matched, an error message will appear on the right corner.

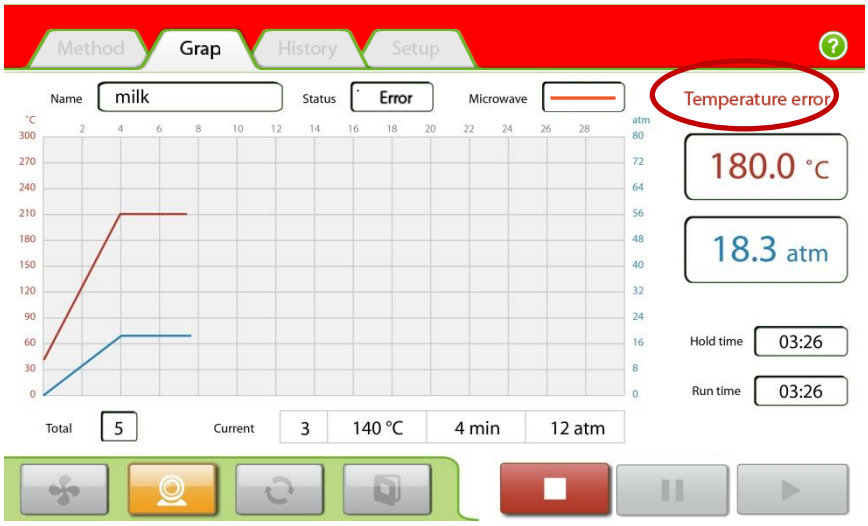


Figure 3-36: Temperature exceeds the limit

Error message, the related reason and the methods:

Error message	Reason	Solution
Temperature error	The maximum temperature in method is higher than the vessel maximum working temperature.	Back to the method interface to reset a suitable method.
Pressure error	The maximum pressure in method is higher than the vessel maximum working pressure.	Back to the method interface to reset a suitable method.

3-3-5 Check the history

Function description: TOPEX management control software enables you review and record the history record.

Figure 3-37: History interface

1.The history record list displays the latest records, the newest one is in the front of the list, this instrument can record as much as 20 records, the record is including of two information: method name and the operating time, which is the system time showing the year, month, date, hour, minute, second , for example: 2012-04-13 13:27:44.

2. View key, press the view key to enter into the history curve.

3. Page key, press page key to check the history list.

History curve interface

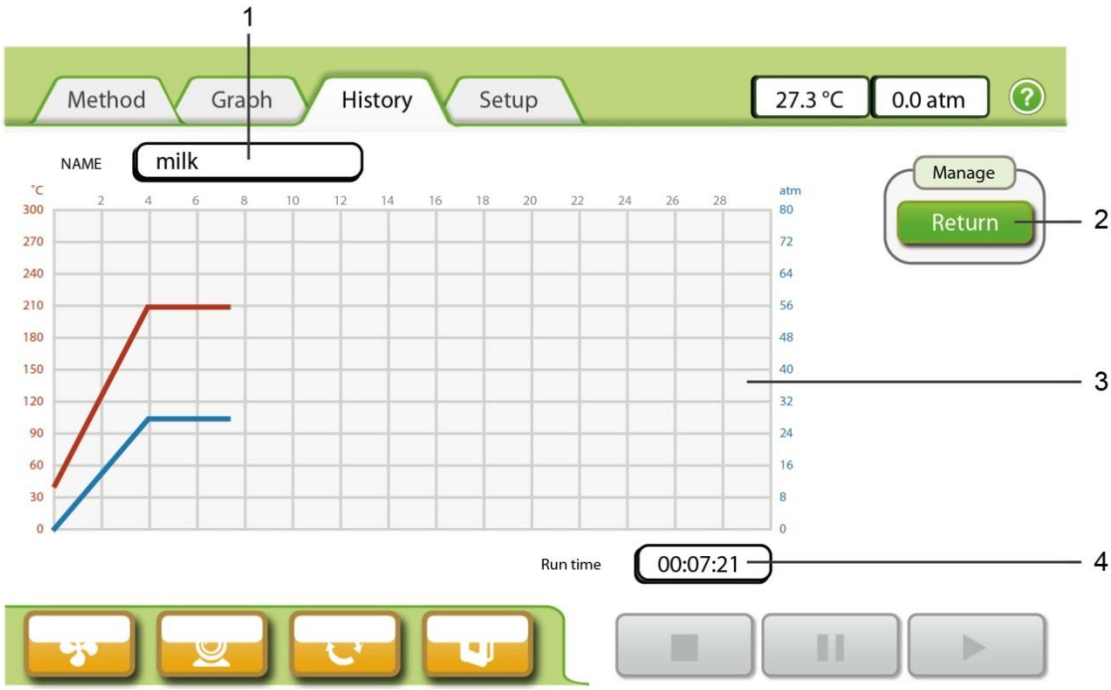


Figure 3-38: History curve interface

1. Shows the working method and name

2. Back key, press to back to the history interface

3. Shows the temperature-time curve and the pressure-time curve.

4. Shows the total working time.

Operating method:

Enter into the history

Press the history key

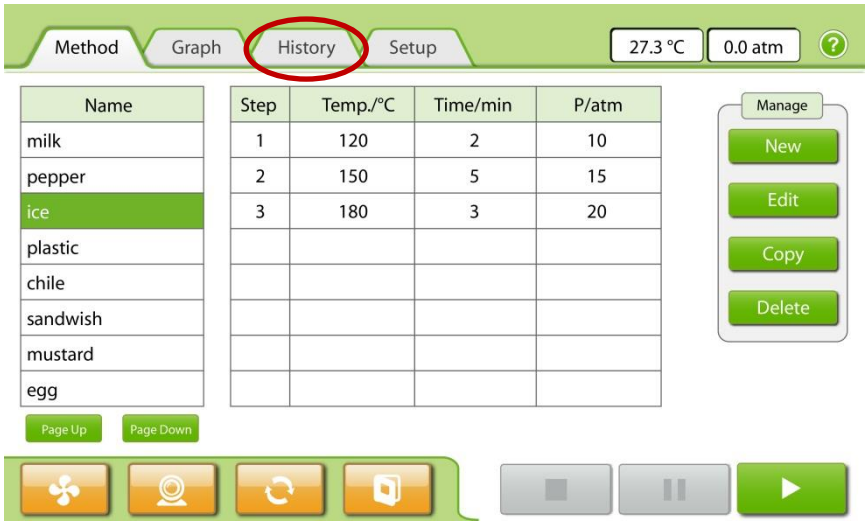


Figure 3-39: History interface

View the history

In the history interface, find out the desirable history and enter it.

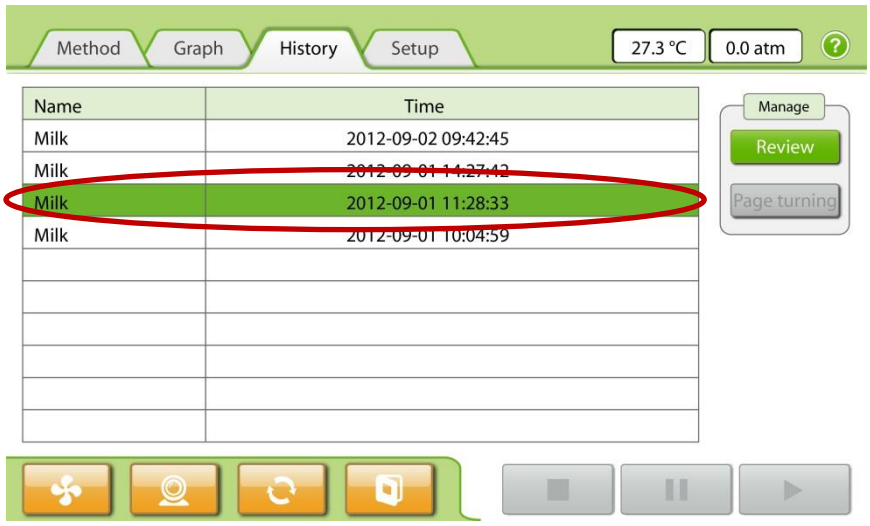


Figure 3-40: Choose the history record

Press the review key.

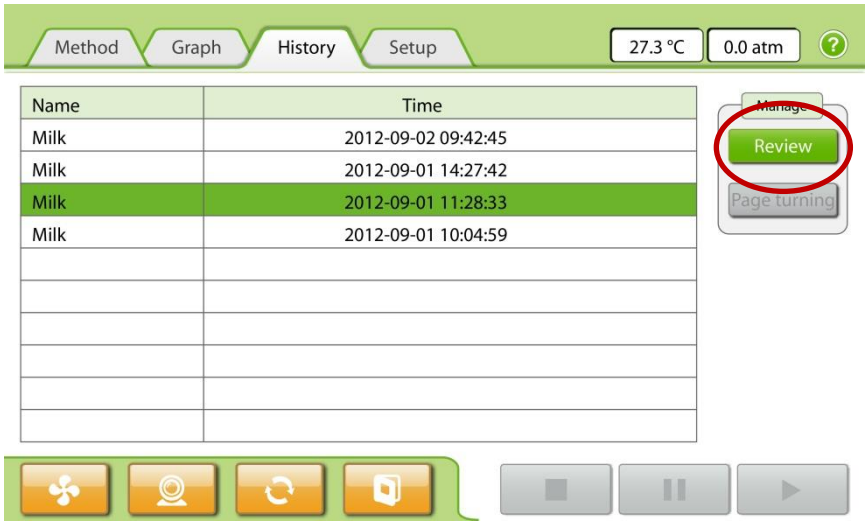


Figure3-41: View the history

Enter into the curve interface.

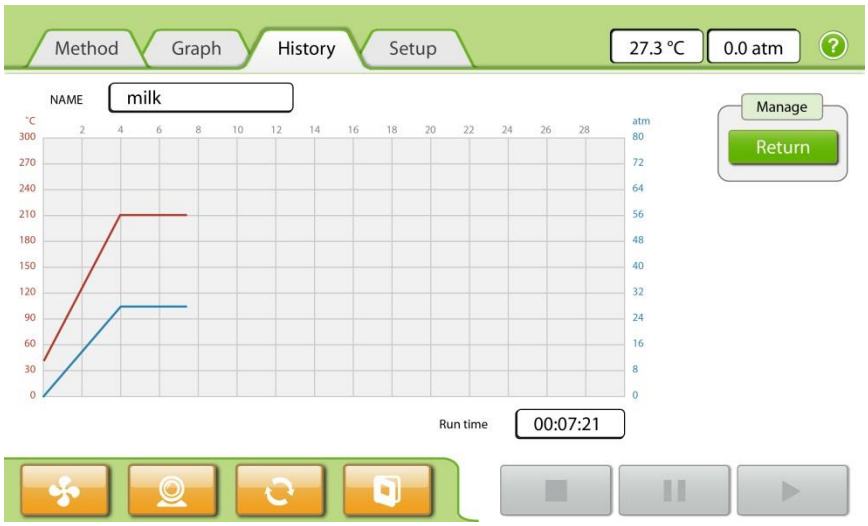


Figure 3-42: History curve interface

Press the return key.

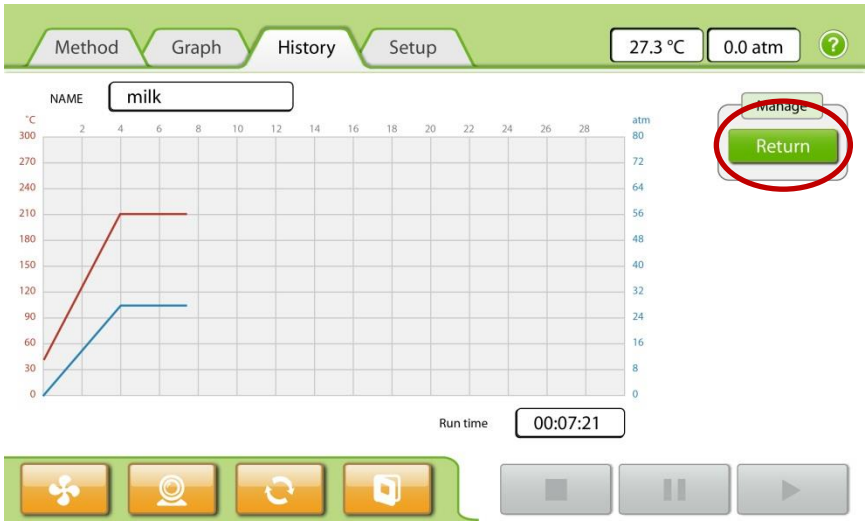


Figure 3-43: Back to history

Enter into the interface of history.

Turn page

Press the page turning key to view the next page.

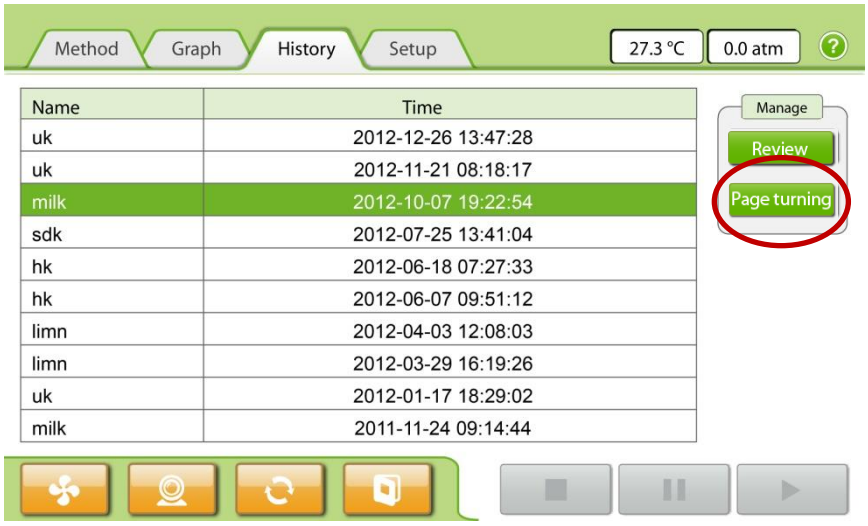


Figure 3-44: History interface

20 records can be found out in two pages.

3-3-6 System set-up

Function description: shows the basic information of the instrument, the operating environment.

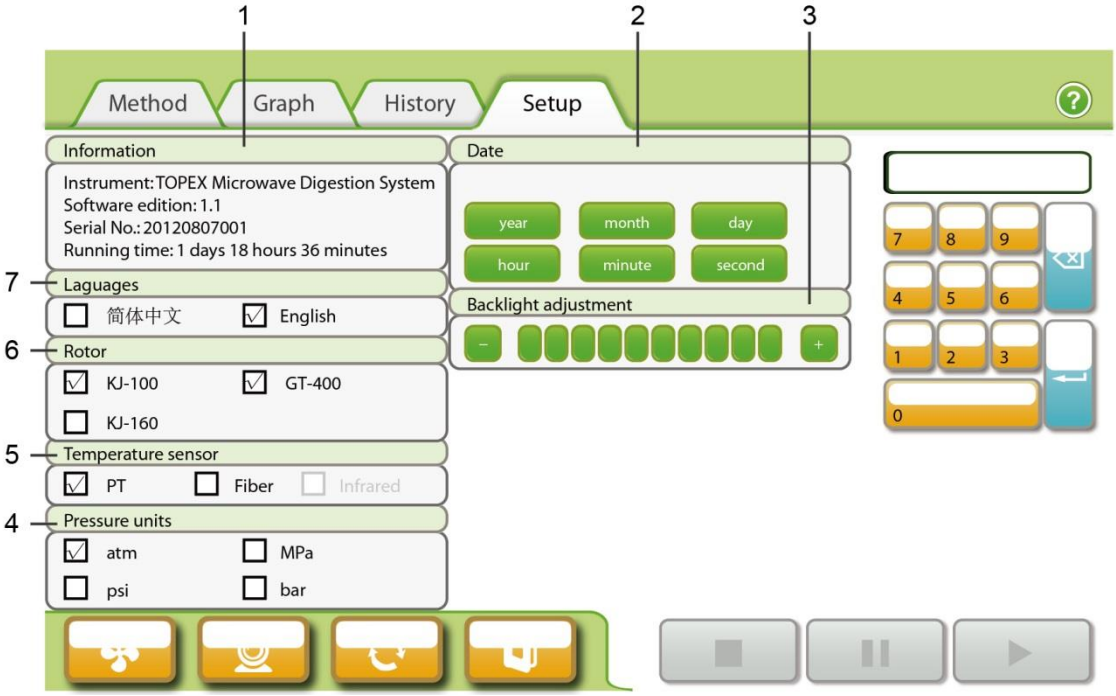


Figure 3-45: Set-up Interface

Interface set-up, displays the basic information and function, which including the current time, language, pressure unit, the temperature sensor and the rotor system. The instrument set-up should be strictly obeying the operating manual. PreeKem will be not responsible for any disoperation.

1. Instrument information, including, instrument name, software edition number.

2. Shows the current time, you can do set-up here, click the related key to input the numbers.

3. Backlight regulation, press the add &reduce menu to control the light.

4. The available pressure unit, 4 units is available: MPa, atm, psi, bar.

5. The available temperature sensor: platinum resistor, fiber-optical or IR temperature sensor.

6. Available rotor, KJ-100, KJ-160 and GT-400.

7. Available language, Chinese and English.

Operating method:

Enter into set-up

Press the set-up menu

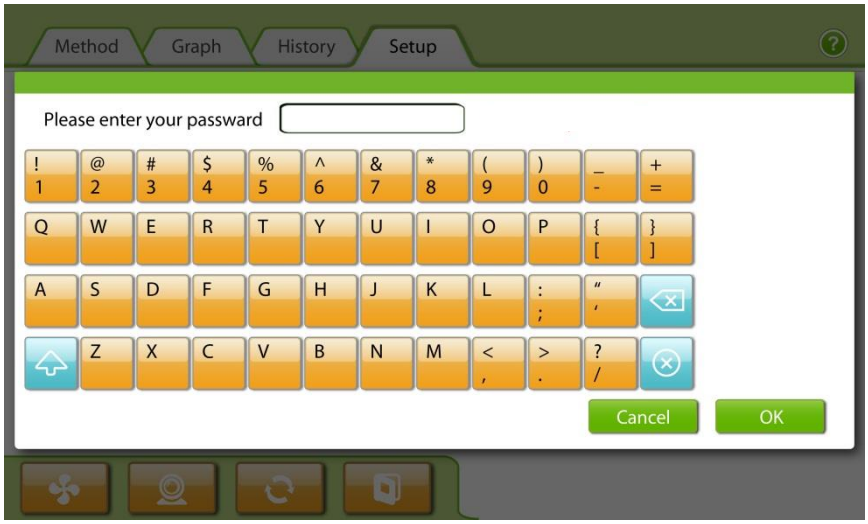


Figure 3-46: Enter the password

Press the password “preekem” in small letter and press the enter key to confirm.

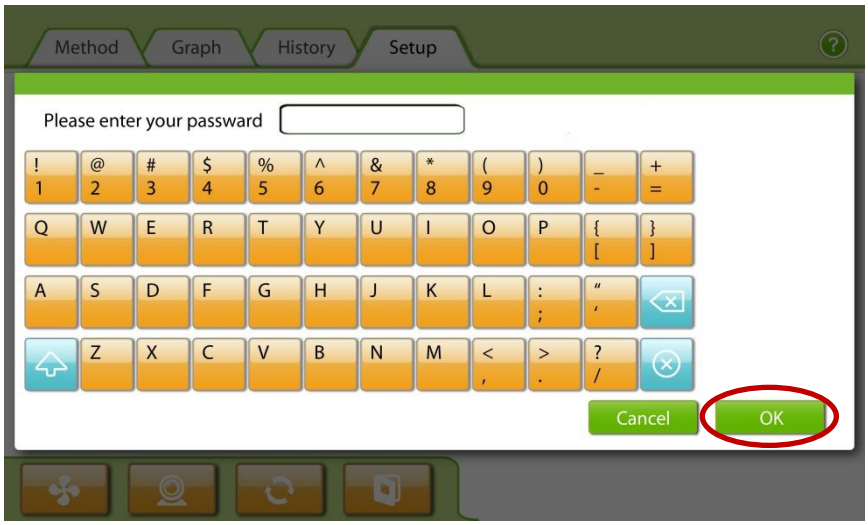


Figure 3-47: Confirm the password

Enter into the set-up interface.

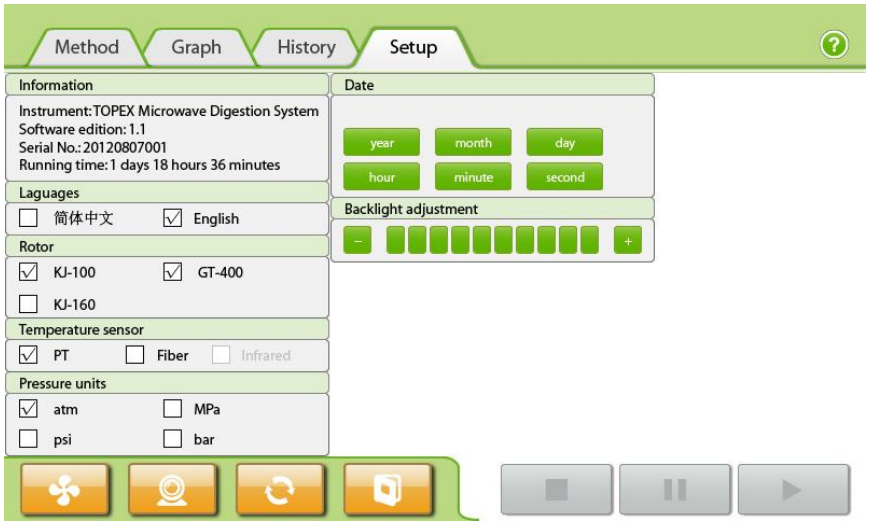


Figure 3-48: Interface set-up

Edit language

Directly click the language box to choose the desired language.

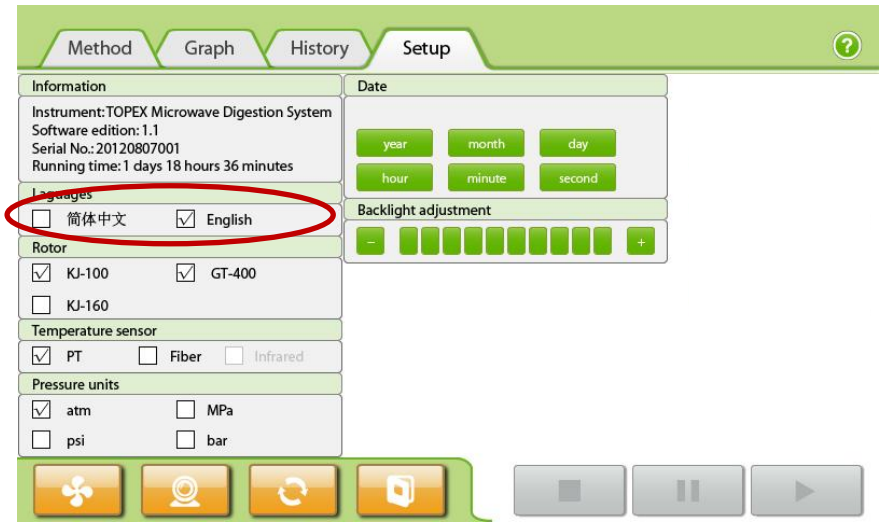


Figure 3-49: Choosing the language

Choose the rotor

Choose the rotor according to digestions vessel type.

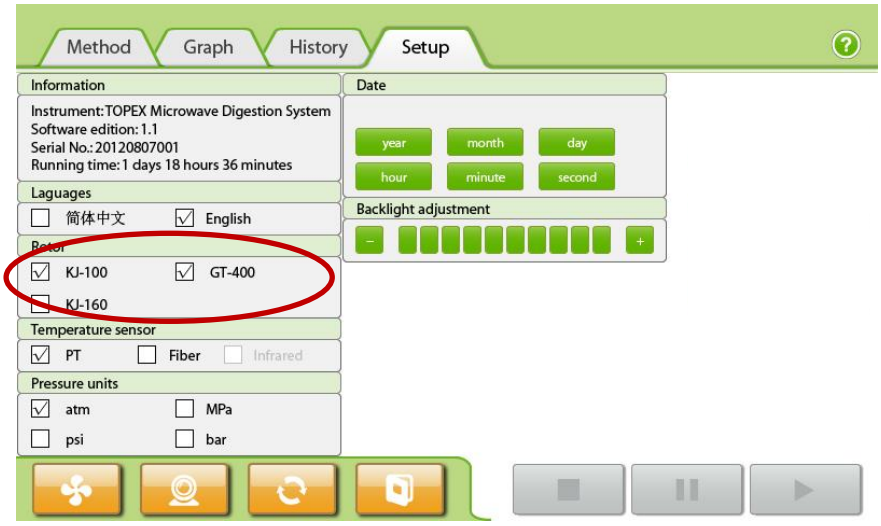


Figure 3-50: Choosing the rotor

ATTENTION **When applying the GT-400 digestion vessel, the display will show its using frequency, please strictly obey the using frequency and replace it if exceeding the using frequency.**

Choose the temperature sensor

Choose the suitable temperature sensor, for KJ-100 and KJ-160, the platinum resistor or the fibre-optical sensor can be chosen, if for the GT-400 digestion vessel, only IR temperature sensor can be chosen.

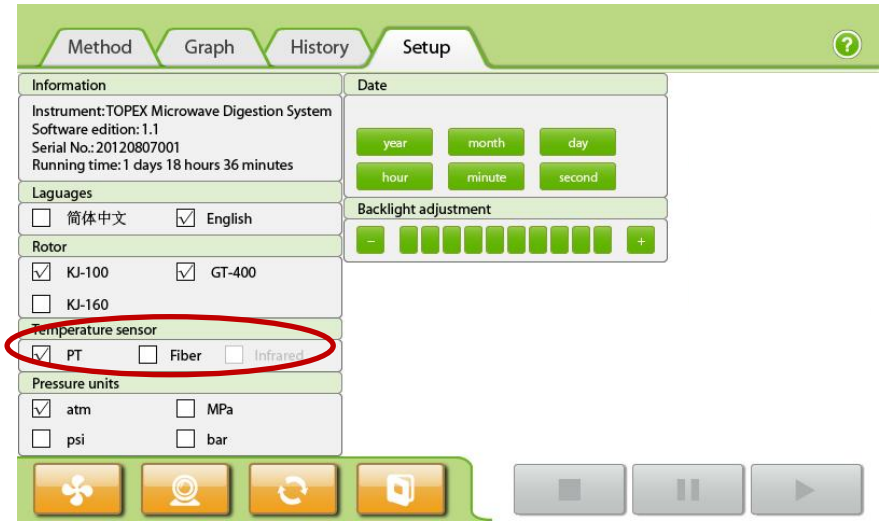


Figure 3-51: Choose the temperature sensor

ATTENTION **Before apply the IR temperature sensor, please reinstall the temperature sensor assembly and air-guide tube assembly and restart the instrument.**

Set the pressure unit

There are atm, MPa, psi and bar total four units available.

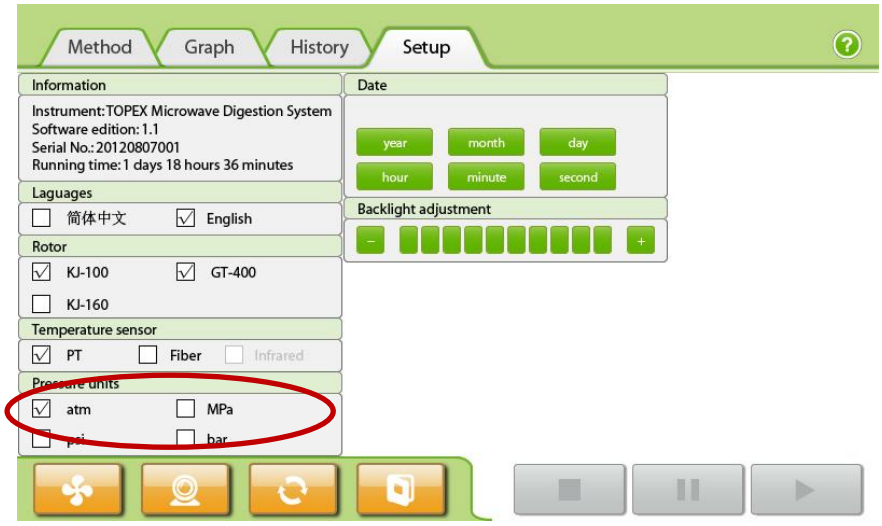


Figure 3-52: Set the pressure unit

Time set-up

Click to edit the time unit in the time bar.

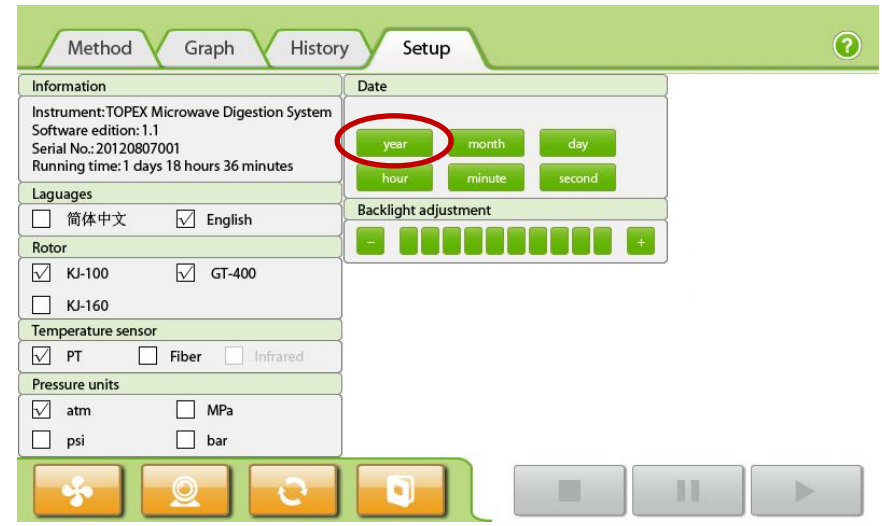


Figure 3-53: Choose time

Input the timer in the numerical keyboard on the right side.

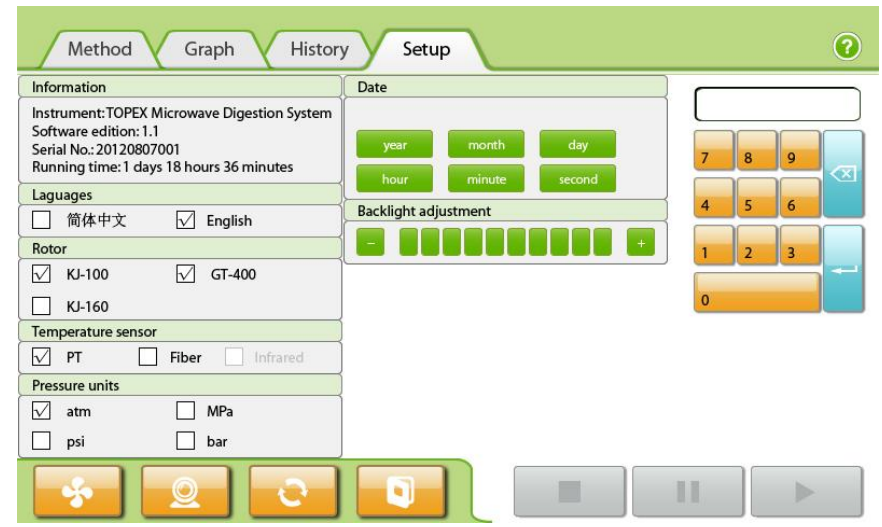


Figure 3-54: Input time

ATTENTION **Only two numbers can be input every time, if exceeding the limit, there will be no response.**

ATTENTION **If want to change the year, just type in the last two numbers, for example , the year of 2015, just type 15 and press the enter key.**

Press the enter key to finish the time setting.

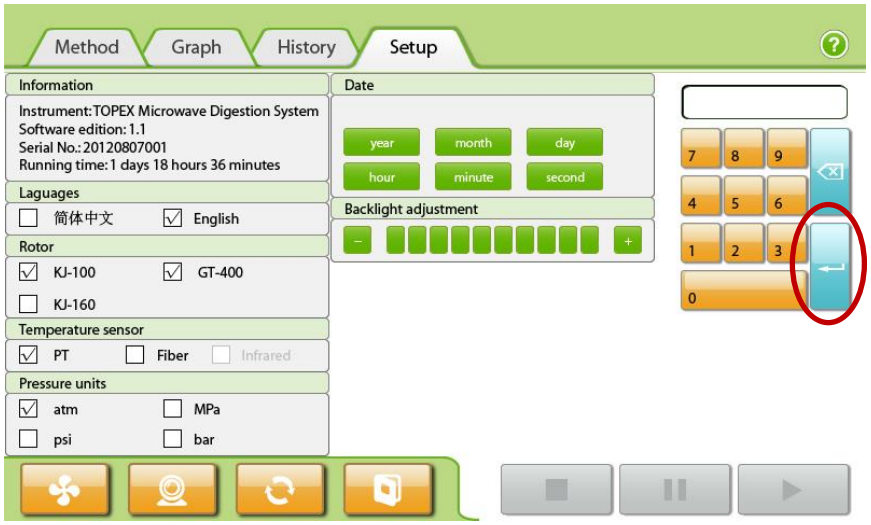


Figure 3-55: Finish setting

When the setting finished, the keyboard will be automatically hided, the edited time can be viewed on the time bar.

Regulate the lightness of the screen

To press the key of “+” “-“ in the backlight regulating bar to regulate the lightness.

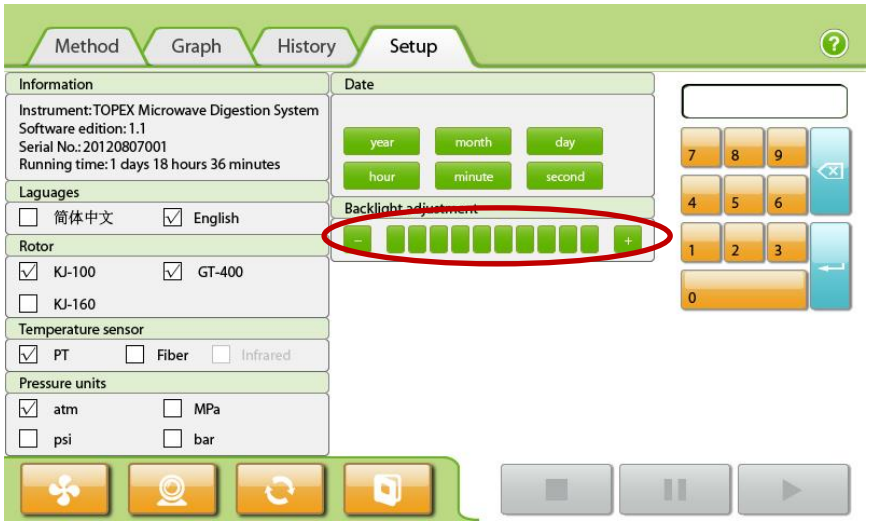


Figure 3-56: Set the backlight

4 Trouble shooting

If you have problems with using instrument, check the instrument refer to following table. If you can't solve the problem, please contact us.

Fault	Probable cause	Solutions
No reaction while turning on	No power supply	Check the fuse, replace it when needed. Check the input power
Indicator is on	Fuse is blown	Replace the fuse
Temperature reading is not stable	Shielded wire is not grounded well so that the signal is affected by microwave Temperature sensor failure	Re-install the temperature sensor Ensure the reliable grounding of temperature sensor assembly and shielded wire Reseat temperature sensor connector Replace the temperature sensor
Temperature reading error	Temperature sensor failure	Replace the temperature sensor
The pressure reading (not zero) is displayed on the screen after turning on the machine	Air retained in the pressure sensor Pressure sensor failure	Re-install the pressure sensor Replace the pressure sensor
Turntable does not rotate	Rolling balls are stuck in turntable	Check the rolling balls at the bottom of the turntable
No display on LCD screen	No access to power	Check the third route fuse
Fuse burnt during experiment	Heating time is too long	Replace the fuse
Instrument crash	Program runs into an infinite loop The temperature retaining time is set to 0	Restart the instrument Re-set the temperature retaining time
Sparking appears in cavity	Sample may contain metal Temperature sensor probe is too close to the oven chamber	Perform acid treatment before experiment Adjust the direction of temperature sensor
Screen prompts "door open"	Oven door is not closed	Close the door and then start the testing

Screen prompts “time-out”	Temperature setting range spans too widely Poor microwave absorbility of sample No sample is placed in control tank	Shorten the experiment temperature span Add the steps in experiment Verify if control tank sample conforms with standard tank Check the control tank for leakage
Screen prompts “abnormal”	The limits of temperature and pressure are achieved at the same time during the testing	Increase the safety limits setting of temperature and pressure
Sample digestion is not completed	Leakage phenomenon Target temperature is set too low to reach the sample digestion temperature Inappropriate acid proportion	Expand the skirt size Increase the digestion temperature Confirm the proportion of acid
Only the sample in control tank is digested completely	Temperature sensor is poorly grounded. Sample dissolution cup of control tank is used too frequently, resulting in its stronger microwave absorbility	Confirm ground condition of the temperature sensor, replace the temperature sensor according to the actual demands Select a newer dissolution sample cup as digestion inner tank
leakage in digestion tank	Not yet expand the skirt size The air-bleed screw is not tightened Safety film rupture	Expand the skirt size Tighten the air-bleed screw Replace the safety film
After the experiment, the screen displays a larger	"suffocating" in pressure sensor protection sleeve	Reinstall the pressure sensor to release the residual air

Appendix 1 Principle of Microwave Heating

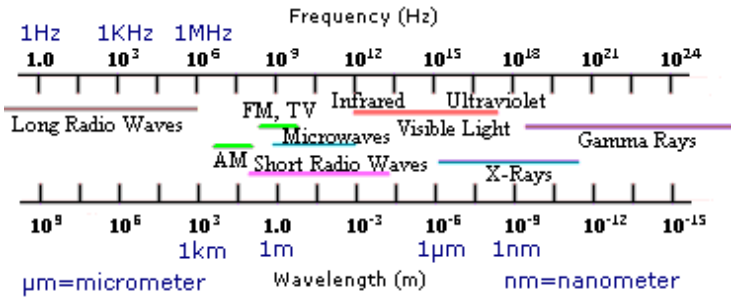


Figure f1-1: Electromagnetic Spectrum

Microwaves are electromagnetic waves with frequencies ranging from infrared to radio, or equivalently, between 300 MHz and 300,000 MHz.

In the process of microwave heating, there are two kinds of mechanism, which are dipole rotation and ionic conduction when microwave energy transfers to heat.

Dipole rotation mechanism is friction between the molecules internal objects caused by microwave radiation to generate heat. All medium in nature are composed of positively charged and negatively charged molecules (or dipole) component. Normally, the dipole in medium is chaotic motion and order. When the medium is in an electric field, its internal re-arranged to a certain orientation and regular arranged polarized molecules. When electric field direction changes as a certain frequency, the polarization orientation of the dipole in medium is changed as the same frequency. In the transformation process, friction and collisions between molecules produce heat. faster electric field changes, faster rotation of the dipole, and stronger thermal effects produced. Electromagnetic frequency of microwave is up to 10^8 orders of magnitude. So the heat generated by dipole rotation is considerable by microwave radiation. The system reaches high temperature in a very short period. The thermal efficiency generated by the dipole rotation depends on the relaxation time, temperature and viscosity of the medium.

The principle ionic conduction is the dissociation of ions in the electric field can be generated in conducting mobile, due to the medium on the ion barrier to generate thermal effects. The Thermal efficiency depends on the size, concentration, charge and conductivity of ion.

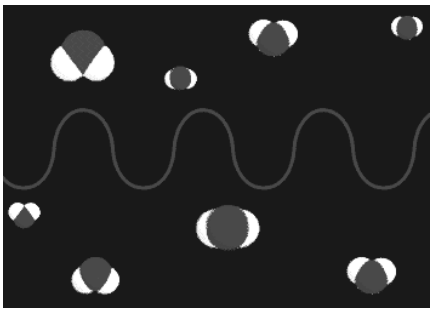


Figure f1-2: Molecular dipole rotating

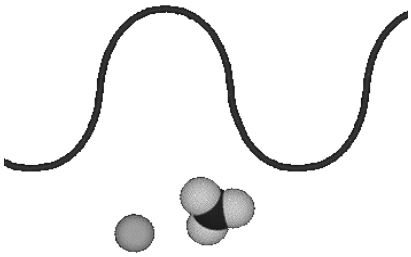


Figure f1-3:Ionic conduction

Traditional heating is by conduction and convection. First heating container, the container will conduct heat to the surface, and then passed to the inner of the object to obtain thermal equilibrium. Therefore, the heating takes a long time. Heating environment in general can not be strictly adiabatic closed. It could

disseminate a lot of heat to the environment for a long time heating. The microwave heating is usually carried out in the fully closed environment, Microwave power goes into the object at light speed, and transformed into heat energy in time, which avoid heat loss in the long time heating process. And microwave can "overall" heat inside and outside of the object. Microwave heating is high efficiency, fast, low energy compared to conventional method.

Appendix 2 Sample Preparation in Airtight Condition

The factors which can affect the result of this kind of digestion method are as the following: sample class and its quantity, acid solvent class and its quantity, pressure, temperature, time and the sample number inside the oven and so on. For some kinds of the sample, the digestion plan have been found, you can take it as a reference, but you should avoid by all means to repeat that the plan says. Meanwhile, for most of the samples, the digestion test have not been made and the digestion equipment are different with other, so the customer should test by himself for trying to found the digestion plan and data suitable for himself. Here, we list the principles of selecting the digestion factors before test as the following for discussing.

1 Sample Weight

Too much the sample will cause the reaction too strongly, and the pressure increasing speed may be over the responding speed of the controlling system. When the pressure inside the vessel is over the maximum standing pressure of the vessel, there will be the danger of explosion. So, at the beginning of the test, the sample weight must be controlled strictly. First, use small weight sample to set up the digestion process. After understanding the complete reaction process deeply and holding a certain assurance of the reaction, then the weight can be increased properly according to property of the sample, thus to be helpful of digestion the sample with bigger weight quickly and safely within short time.

1-1 Organic Sample

Large amount of gas will be created during the digestion period, so the weight of the sample should be restricted within the range of 0.1-0.2g, and the detailed weight is determined by the organic content of the sample. Like the petroleum, it's belonging to the class of heavy organic substance, and the safe limitation weight of the sample is no more than 0.2g. For the un-familiar sample, the test can be started from the sample weight of 0.1g and increase the sample weight step by step later on. Or the sample can be pre-treated in an opening container.

1-2 Inorganic Sample

The weight range of the sample is 0.1-0.5 g. But some kinds of the inorganic substances can absorb the microwave energy directly, like the charcoal black, carborundum and so on. This direct heating can improve the digestion speed of this kind of substances. But should pay attention that this kind of heating has its shortcoming of heating locally, this may cause the heating temperature being over the melting temperature of the container and so damage the container.

If the diameter of sample pellets is less than 100 meshes, then it will be digested easily. If the diameter of sample pellets is bigger, or the sample is one piece, then the digestion speed will be low. This is due to the small reaction surface of the sample.

For the inorganic sample, 10mL of mixed acid solution is always to be added. The types of the mixed acid solution are HNO3:HF, aqua fortis or aqua fortis plus HF. How to choose mixed acid solution is determined by the matrix and elements of the sample. It must be very careful when using the HF, because it will create large

amount of foams when adding it into the sample. So the HF should be added in small quantity steeply to avoid the foams created by the reaction between the acid solution and the sample being escaped out of the vessel.

1-3 Mixed Sample with Organic and Inorganic

It should be treated as the organic substances. If the sample contains 5% (calculated in weight) organic substances, the organic substances should be decomposed in first priority. Normally, use the stick nitric acid to decompose the sample in the beginning stage until most of the organic substances being digested. In some cases, the mixed acid solution made by nitric acid and vitriol may be used to digest the organic substances. After all the organic substances being digested completely, then the other kinds of acid can be added to digest the other inorganic substances.

The sample weight of the inorganic substances which can be decomposed is basically restricted by the vessel volume and organic substances content of the sample. Before making the further step decomposition in vessel, it's recommended to make a pre-decomposition for the sample by heating it in an opening vessel. In this way, the organic substances which can create gas during digestion process can be cleared. But in this step, some elements with high volatility (e.g.: boron (B), phosphor (P), Selenium (Se)) may also be lost. So in such cases, the digestion plan should be modified to keep the necessary elements for analysis. For the soil or deposit samples which have been polluted by organic substances, they should be treated as the organic but not the inorganic sample. Especially, if the brown smoke created by the oxide of nitrogen was observed during the digestion process, it shows that there is considerable amount of organic substances in the sample.

2 Acid and Mixed Acid

2-1 Nitric Acid

The nitric acid is most popular used in sample digestion. It's a strong oxidizer. It's widely used in releasing the trace elements from the bio-sample, and making the trace elements into the soluble nitrate. The boiling point (120°C, 68%) of the nitric acid is relatively low. For destroying the complicated basic organism, always over than 120°C is necessary. or other kind of strong oxidizers, like the H2O2 or HClO4 and so on, are to be added. The nitric acid will have ideal reacting ability when excited by the microwave energy, and can create the russet gases formed by the nitrous oxide (NO, NO2, N4O4) quickly. Over a certain temperature, food sample can be decomposed quickly. In vessel, the boiling point of the nitric acid can reach 176°C at 0.5MPa pressure. At this higher temperature, the oxidizing electric potential can be increased remarkably. This can make reaction more quickly. If necessary, the H2O2 will be added after the decomposition finished for achieving completely pretreatment.

The nitric acid is mainly used for digestion the organic sample, such as fat, protein, carbohydrate, floristic material, waste water, some kinds of pigments & polymers and so on.

2-2 Hydrofluoric Acid (HF, boiling point: 106°C, 49%)

This is a kind of effective solvent for resolving the silicon-based materials. The silicate will be changed into the volatile SiF4 and the other interested elements will be remained.

When the hydrofluoric acid mixed with the nitric acid in 5:3 or 1:1, the temperature and pressure increasing curve of the mixed acid solution based on the heating time is similar to the temperature and pressure curve for not only the hydrofluoric acid, but also the nitric acid when they are sole used. That means that these two kinds of acid didn't react with each other and create any decomposed product. The mixed acid with these two kinds of acid can be used for digestion of silicate, Ni-Cr alloy, lead-tin solder, SiO2 titanium-oxide and so on, and the mixed acid solution of boracic acid together with the hydrofluoric acid and nitric acid can used for the digestion of slag.

The hydrofluoric acid can not be used with glass- and quartz-made containers.

2-3 Hydrochloric Acid (Boiling point, 110°C, 36%)

This acid is one of the ideal solvents for some kinds of metal oxides and the metals which can be oxidated easier than hydrogen. At high pressure and high temperature, many kinds of silicates, un-soluble oxides, sulfate and fluorid can react with the hydrochloric acid to create the soluble hydro-ates. This acid is also an effective solvent for the alkaline compounds like alkaloid and amic compounds in the water solution, and for the organic metal compounds. But the application of hydrochloric acid solely is not popular. Materials which can be decomposed by the HNO3+HCl+HF solution are as followings: coal cinder, paper ash, boiler scale of the cooling water tower, fiberglass, kaolin, zeolite and so on. After the decomposition, add the boracic acid to counteract the dissociative fluoric ion.

2-4 Sulfuric Acid

It's the effective solvent for many kinds of organic tissues, inorganic oxides, hydrates, alloys, metals, ores and so on. Sulfuric acid l can destroy the tissue of almost all kinds of organic substances and dehydrate them completely. The boiling point (339°C, 98%) of Sulfuric acid is quite high, so when CFC containers are used, the temperature of the container must be monitored. The mixed acid solution of nitric acid and Sulfuric acid has a fairly low boiling point. The alumina sample can be decomposed successfully by the mixed acid of phosphoric acid and Sulfuric acid in the ratio of 1:1.

The mixed acid of nitric acid and Sulfuric acid can be used to decompose the following samples: syrup, confiture, polyethylene, polypropylene, PVC, acrylate.

2-5 Perchloric Acid

This is a strong oxidizer, can react with the metals which other acid can not react with. It also can decompose the organic substances completely. But when the hot and thick HClO4 solution contacts with the inorganic substance, there will be a possibility of bombing. In the process of airtight microwave digestion, the HClO4 solution is not allowed to use, because the temperature and pressure rises so quickly that it's obviously a potential danger.

2-6 Phosphoric Acid (Boiling point, 261°C, 85%)

This acid has been used successfully for the digestion of iron-based alloys, which when being decomposed by the hydrochloric acid, some special trace elements will be volatilized to lost. This acid can also be used to decompose the sample of aluminous slag, iron ore, chrome and alkali metals.

2-7 Aqua Fortis

This is a mixture of nitric acid and hydrochloric acid. The aqua fortis can be more effective for oxidizing many substances than hydrochloric acid or nitric acid solely. This can be used for digestion of slag and geologic sample, floristic tissue and waste water.

The aqua fortis should be prepared in the working site, because it's not stable and difficult to store. Normally, the ratio is 1:3 (HCl : HNO_3) in volume, and the boiling point of aqua fortis is 112°C.

2-8 Peroxide (boiling point, 107°C, 30%)

This is a strong oxidizer, and should be used carefully. It can be used with nitric acid as 5:1, but not be allowed to use it solely for digestion.

DANGER **For the acid with high boiling point like Sulfuric acid, phosphoric acid and so on, they should be used in thin density or mixed with some other acid.**

DANGER **Don't use peroxide solely.**

DANGER **Don't use perchloric acid.**

DANGER **The total volume of sample and solvent should not be more than 20 mL.**

3 Reaction Condition of Temperature, Pressure, Time

The digestion reaction can be increased on the condition of high pressure and high temperature. For digestion sample complete, it's necessary to know the type of acid and the proper temperature and pressure suitable for the analysis of this matrix and elements. A proper designed digestion method is to digest all elements of the sample quickly at the lowest temperature and pressure as much as possible.

3-1 Temperature/Pressure for the Reaction of Organics

When digesting organic samples in vessel, by-products such as CO2 and NO2, may create very high pressure. Saccharide, protein and fat are three basic components of bio-sample. Protein can be digested quickly at 150°C, and the saccharide and fat at 160°C. When the temperature is over 175°C, only π-bond among all the organic bonds in the phenyl structure will be remained unbroken. This is the amino acid of aromatic family. The organic substances can not be digested completely when using the nitric acid. For digestion them completely, the special reagent like HClO4 solution must be used. But to use such kinds of strong

oxidizers should be avoided. When digesting over 50 mg sample or samples which can produce gas, the digestion should be made in steps to reach the final pressure. Only in this way, the digestion process can be controlled. The digestion temperature of the organic molecules is different from ranged 140°C to 180°C based on the difference of molecule weight. The critical temperatures of the common foods being decomposed with nitric acid are as followings:

Sample	Digestion Temperature with Nitric Acid (°C)
Amyloid type carbohydrates	140
Proteins	145-150
Saccharide	150
Lipoid, Fat	>160-165
Heavy oil, petroleum, bitumen	>180-185

3-2 Temperature/Pressure for the Reaction of Inorganics

For the digestion of the inorganic samples, more strong causticity mixed acid solution and high temperature should be used. Such kind of samples can be digested in opening quartz-made containers, because during the digestion process, there will be only very few gas produced and the pressure will not increase too much. The samples with strong oxidbillity such as mineral, ore, ceramics or some alloys should be digested in vessel. The pressure produced by digesting inorganics with acid will be lower than that for organics. Most of the inorganic samples can be digested at lower than 185°C and lower than 1MPa, and it's unnecessary to increasing the pressure and temperature in steps. Normally, high temperature and the pressure will not increase the digestion speed, but will reduce the digestion vessel life.

3-3 Effect of Vessel Numbers on Temperature/Pressure/Time

The temperature/pressure/time will be varied with the difference of sample numbers (vessel numbers) in the cavity. Normally, after the condition of a certain sample being completely digested in two control vessels being tested, the number of samples in the cavity can be increased one by one, and the temperature and pressure should be increased and the time should be prolonged in a mitigated way, so to find out the proper temperature/pressure/time condition for multi samples (vessels)

4 Forbidden Samples for Microwave Digestion

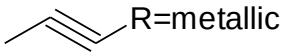
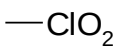
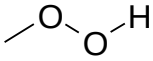
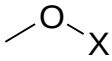
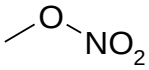
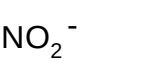
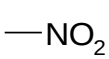
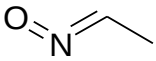
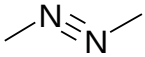
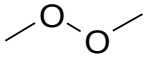
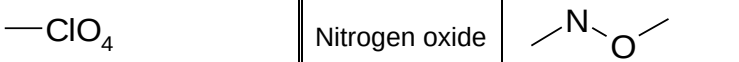
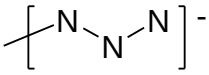
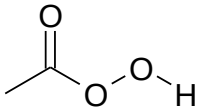
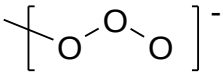
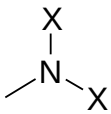
DANGER

When the following dangerous samples and the atom radical are introduced, much more attention should be paid to such kinds of chemical reactions. Before inspecting the controlling ability of the microwave system and taking measures to reduce the sample weight and the violence level of the reaction, EXCEL should not be misused. We will not take the responsibility of equipment damage and personal injury due to digestion the following samples by microwave:

- Explosive (TNT, nitrocellulose and so on)
- Kindling Chemical Articles
- Aeronautic Fuel (Jp-1 and so on)

- Ethyne Compounds
- Acraldehyde
- Paint
- Dual-element mixture (nitric acid and phenol, nitric acid and ethylamine, nitric acid and acetone, and so on)
- Propellant (hydrazine, ammoniac chlorate and so on)
- Binary Alcohol (glycol, propylcol)
- Chlorate (ammoniac chlorate, potassium chlorate and so on)
- Aether (Fibre-resolving agent-glycol propyl aether)
- Ketone (acetone, formyl- and ethylic- acetone and so on)
- Hydrocarbon-alkyl (butane, hexane and so on)
- Fat of the animal (nitroglyceride, nitroglycerin of other kinds of organic nitration)

Please pay attention to the products may be produced in the reaction between special sample and solvent, the features of the volatile radicel are as the following:

Name	Structure	Name	Structure
Ethyne Compounds		Chlorate	
H_2O_2 Compounds		sub-rock salt	
Nitrate		nitrite	
nitryl		fulminate	
diazo		peroxide	
chlorate		Nitrogen oxide	
diazo compound		peroxide-acid	
ozone compound		N-haloid-amine	

PreeKem Scientific Instruments Co., Ltd.

Address: No.100-101 Building, No.2338 Duhui Road, Shanghai, China

Postal code: 201108

Tel: 86(21) 5442 7057, 18916066078(maintenance line), 18918169066(complaint line)

Fax: 86(21) 5442 7063

Web: www.preekem.com

E-mail: service@preekem.com

Technical Maintenance File

Operation Manual

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