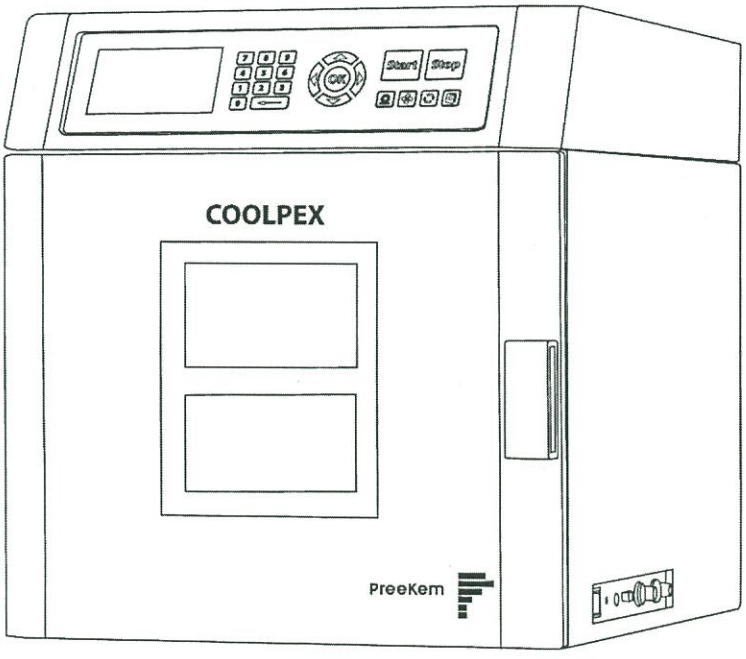
PreeKem

Operation Manual



COOLPEX

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 designed safe standard and correct usage of instrument, the result of wastage 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

Must connect an earth terminal to the ground

Content

COOLPEX Smart Microwave Reaction System Operation Manual

1 Safety Guidelines

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 utilizes high voltages and microwave radiation. Instrument service and repair should be performed only by those trained in repair and maintenance of high voltage and microwave power systems.

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 used, and the electrical power capacity of the tri-core plug should not be lower than 220 V/8 A.

ATTENTION

1.5 In normal working conditions, this instrument will not arise 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 operating without load and large amount of microwave being reflected.

ATTENTION

1.8 The highest working pressure is 3.5 MPa, highest working temperature 235°C. Never operate the instrument over that pressure or temperature to avoid damaging the vessels or other accessories.

DANGER

1.9 Before starting the instrument, close the door. After starting, the operator should not leave and should observe the running the instrument for knowing abnormal conditions immediately. If abnormal conditions happened, press "Stop", and cut off power.

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 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 are trained by our company, 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 without Certification of Instrument Operation.

2 Instrument Introduction

2-1 Overview

COOLPEX Smart Microwave Reaction System is originally created specialized microwave chemistry instrument by PreeKem which can meet the application demands of microwave digestion, extraction and synthesis. This instrument combines the industrial microwave resonant cavity with original microwave focus technique and PID technique together to control the chemical reactions more accurately. The instrument has a fashionable and practical exterior with electric locking door system, and also owns monitor system to view the working status in the cavity in real time. This instrument will help your lab work more convenient and safe.

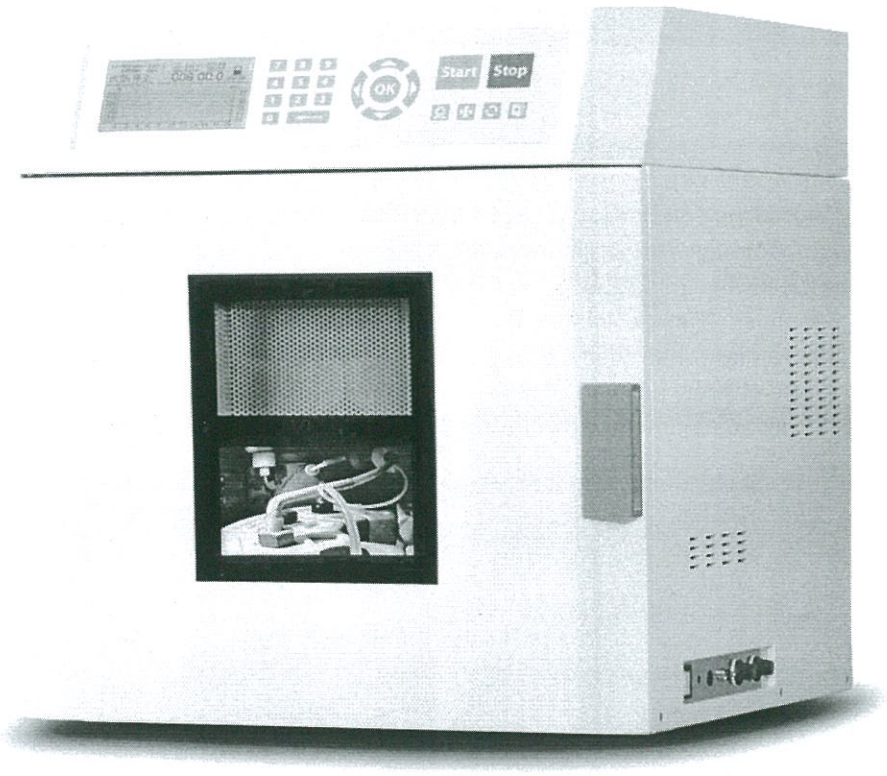


Figure 2-1: COOLPEX Smart Microwave Reaction System

2-1-1 Application

This instrument is used for: accurate procedure control of extremely-high voltage microwave digestion, air-tight organic solution extraction, 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 is covered the synthesis 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 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

- Original design of special microwave resonant cavity in the country for microwave chemistry use
- Volume of cavity is 34L
- Cavity coated with as many as 5 layers modified Teflon PFA (modified FTFE)
- Composite structure auto-locking safety door
- Genuine non-pulse continuous microwave output
- High-frequency closed-loop feedback control
- Platinum resistor temperature measure and control system
- High precision high temperature melt pressure measure and control system
- Strong convective fast-cooling system
- High air-flow capacity anti-corrosion centrifugal blower
- Electric locking, one-touch open the door
- Large LCD screen and comfortable touch panel for operation and view
- Built-in camera and HD LCD monitor system

2-2 Hardware Introduction

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

2-2-1 Core component

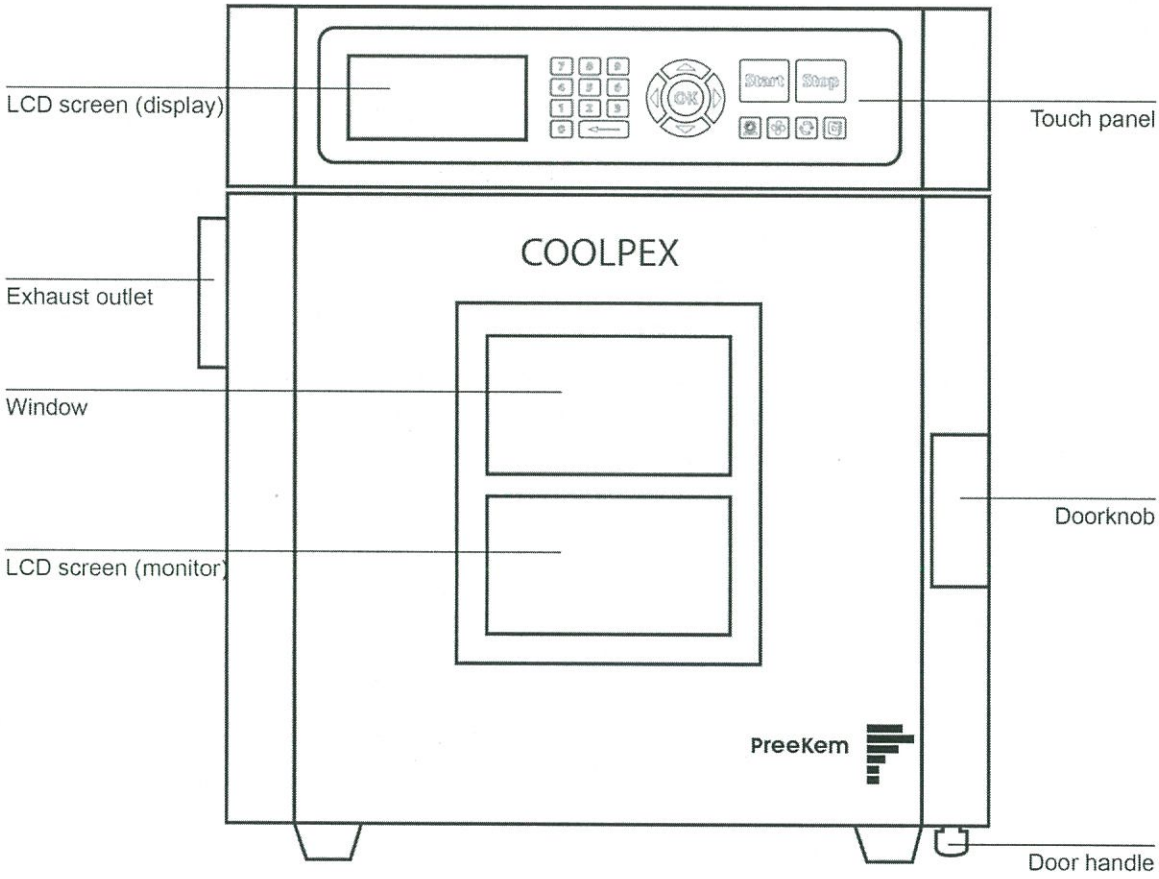


Figure 2-2: COOLPEX Smart Microwave Reaction System front

2-2-2 Right bottom

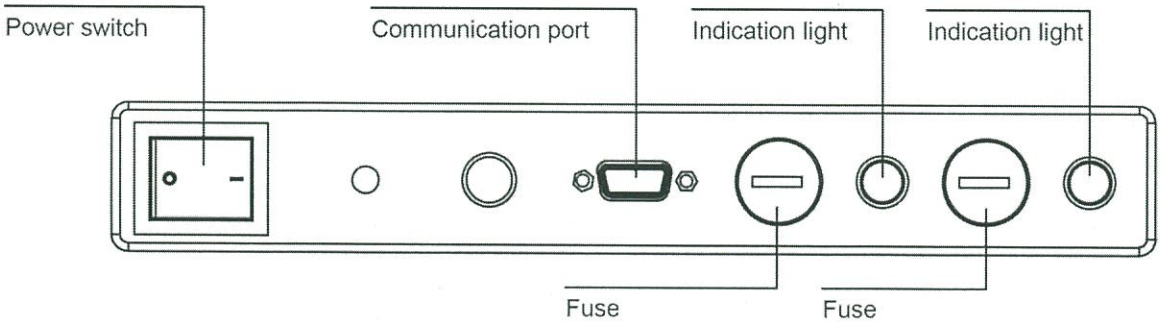


Figure 2-3: Switch and fuse schematic

2-2-3 Back

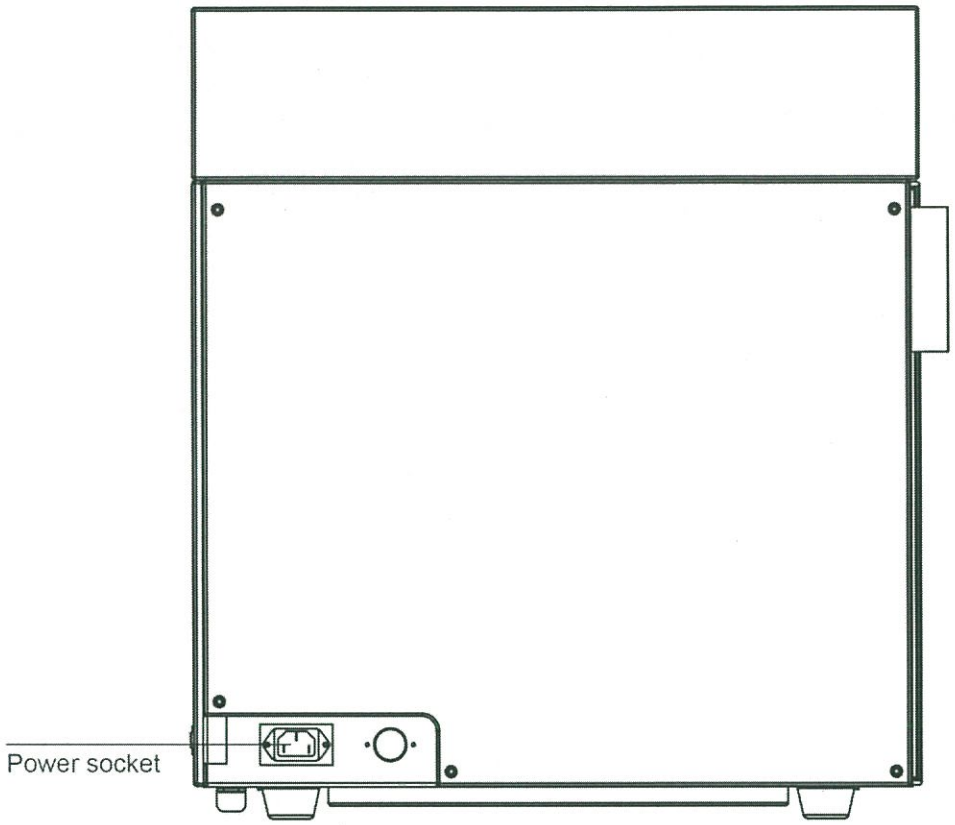


Figure 2-4: Power socket schematic

2-3 Software Introduction

The following paragraphs will introduce you about functions of software.

2-3-1 Screen

The content in LCD screen is composed by a group of different function operational screens, include welcome information, function selection, parameters setup, method management and microwave starting.

2-3-2 Control Panel

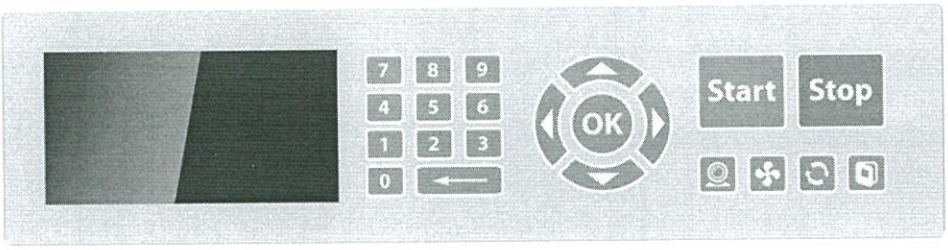


Figure 2-5: Control panel

Button	Illustration
Start	For starting the microwave heating. After all the data settings being finished, push this button to make the whole machine work.
Stop	For pausing or stopping the microwave heating.
	Move the cursor (up, down, left, right), browsing the previous page or following page of "temperature-pressure-time" curve.
OK	For confirming the inputted setting parameter and entering into next interface.
0	Setting the value of temperature, pressure, time, power, vessels and steps.
	Control button for monitor system, turn on the screen on the oven door to observe the situation in the cavity.
	Control button for exhaust system, turn on or off the exhaust fan.
	Control button for rotor, start or stop the rotor.
	Control button for door, open or close the door.

	Adopt the current choice, modification or input and back to the previous interface.

2-3-3 Software Flow Chart

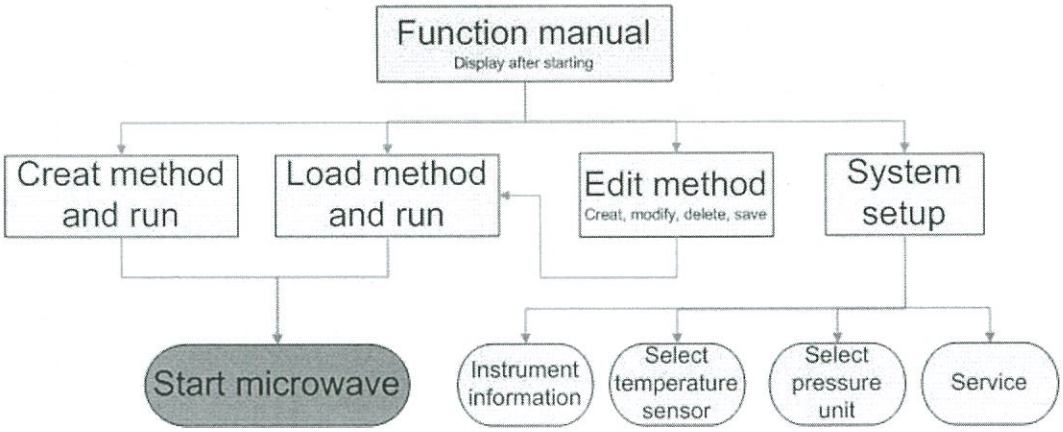


Figure 2-6: Software flow chart

2-3-4 Function Interface

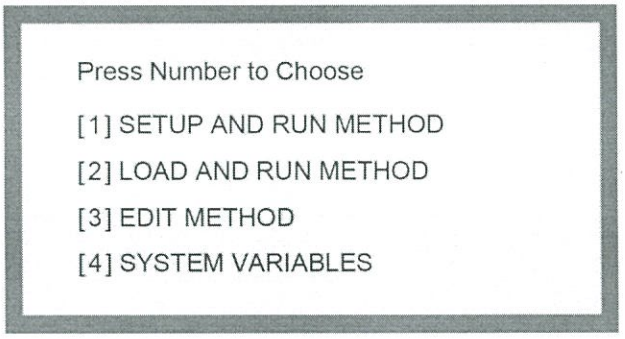


Figure 2-7: Function selection interface

[1] SETUP AND RUN METHOD: Setup a new method, and then start microwave heating after setting directly.

[2] LOAD AND RUN METHOD: Load an existed method and start microwave heating with this method.

[3] EDIT METHOD: Enter in method edit menu to modify, delete and save method.

[4] SYSTEM VARIABLES: For instrument maintenance and adjusting, customer was not allowed to use it. We will not take any responsibility for any accidents caused by incorrect use of system settings.

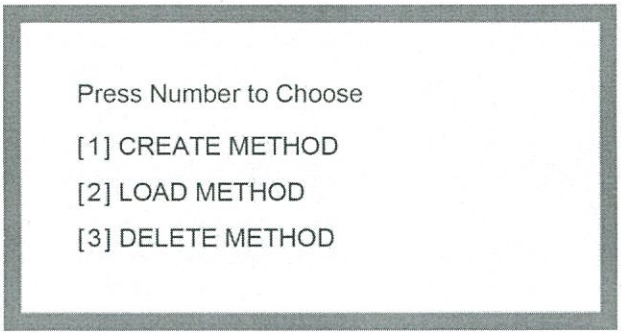


Figure 2-8: Method function selection interface

[1] CREAT METHOD: Edit method.

[2] LOAD METHOD: Edit selected method.

[3] DELETE METHOD: Remove the method has been established.

2-3-5 Edit Parameters Interface

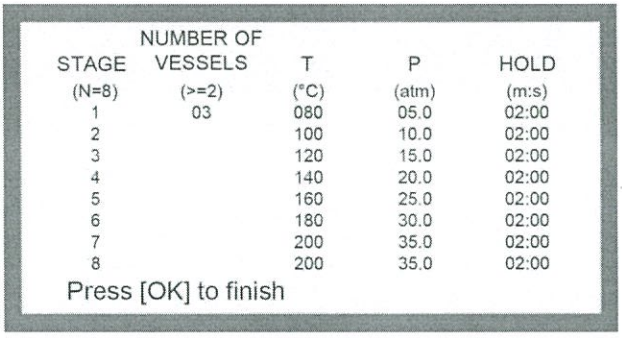


Figure 2-9: Edit parameters interface

STAGE: The total reaction steps, maximum 8 steps;

NUMBER OF VESSELS: The numbers of vessels in the cavity, range 2-10;

T: Reaction temperature, maximum 235°C;

P: Safety limit pressure caused by reaction, maximum 3.5MPa;

Hold: Holding time, maximum 59 min 59 s.

2-3-6 Running Interface

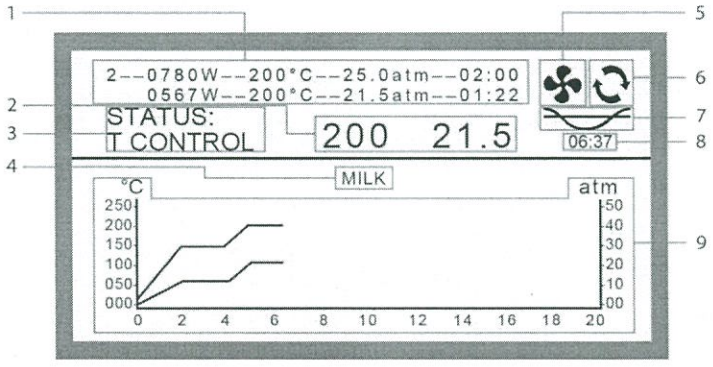


Figure 2-10: Running interface

1: The setting values of first step or current step number, power, temperature, pressure, and hold time are displayed in the first row. The current power, temperature, pressure and hold time are displayed in the second row.

2: Current temperature and pressure in real time. The left number is temperature and the unit is centigrade. The right number is pressure and the unit is atmosphere.

3: Current Working Condition.

4: Method Name.

5: Fan icon: icon is static means fan is turned off, icon is rotating means fan is turned on.

6: Rotor icon: click the button of rotor on the control panel, the icon is rotating and rotor is also rotating.

7: Microwave icon: indication of microwave working conditions, dynamic icon indicates microwave heating, while static icon indicates microwave stop.

8: Reaction timer, record total reaction time from beginning to end.

9: Real time Temperature-Time, Pressure-Time curves from the microwave starting to end.

2-3-7 System Setup Interface

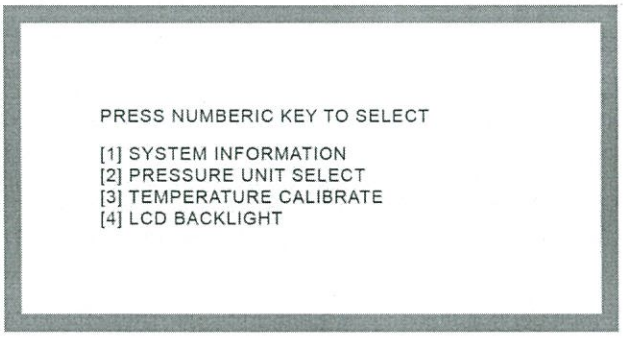


Figure 2-11: System setup interface

This interface consists of temperature calibration, pressure calibration, software information. It is used for instrument maintenance and adjusting. The operator is not allowed to use it. We will not take any responsibility caused by your incorrect use of system settings.

2-4 Specification

Input AC power: AC220-240 V, 50 Hz, 8 A

Microwave power output: maximum 1200 W

Total power input: 1600 W

Microwave power adjust: frequency conversion adjustment

Microwave frequency: 2450 MHz

Microwave leak power density: $\leqslant$0.5 mW/cm^2

Cavity volume: 34L

Electric motors: speed 5 r/min

Power setup: setting number of vessels by control panel, and the heating power will be adjusted automatically by the program

Timer: Setting time by control panel, the timer calculates the time.

Timing range: Single step 0-59 min 59 s

Pressure measure and control system

- Measuring range: 0-5 MPa
- Special pressure sensor, measuring precision $\pm$0.01 MPa
- Pressure control stability ±0.05 MPa, display pressure in vessels on screen in real time

Temperature measure and control system

- Measuring range: 0-300°C
- Measuring precision: $\pm$1°C
- Temperature control stability: $\pm$2°C

Safety measures

- The temperature sensor and pressure sensor equipped with the instrument are used for real time monitoring the variation of temperature and pressure in vessels.
- The safety diaphragm or skirt in sealing cover will be broken for pressure release if the pressure can't be stop. 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 be a certain distance to the outside to reduce the impact of the buffer if vessels explode when microwave heating improper digestion of a sudden or explosive chemical reaction samples. The door, made of high strength stainless steel and explosion-proof glass visible window, can stop fragments effective and avoid personal injury.

Oven chamber ventilation system: centrifugal fan, maximum airflow capacity 2.5 m^3/min

Working environmental temperature: 5-40°C

Working environmental relative humidity: 15-80%

Size of mainframe: 502 width×600 depth×562 height mm

Weight: 58kg

3 Use of Instrument

3-1 Preparation

The following paragraphs will help you understand requirements about how to place and install the instrument.

3-1-1 Placement of Instrument

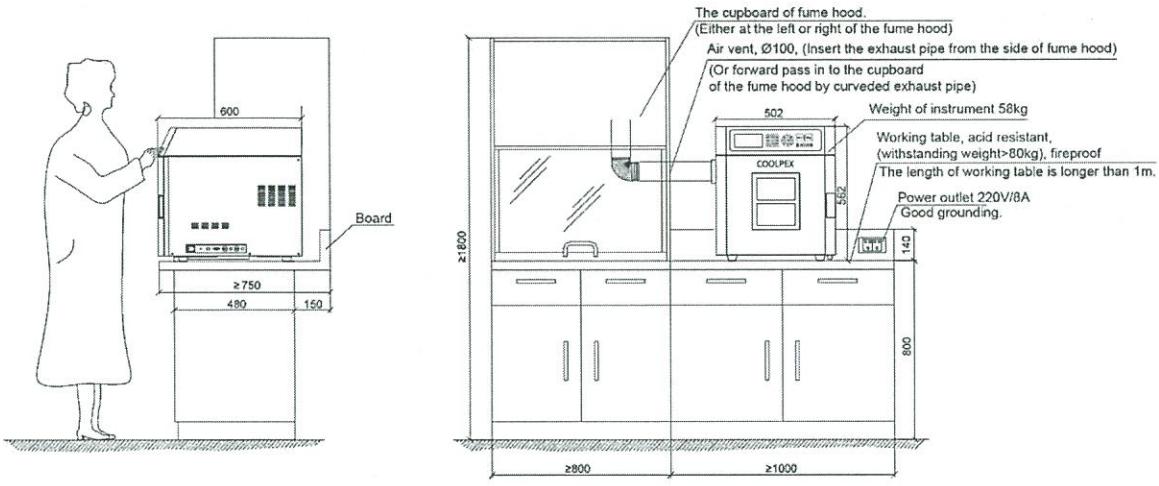


Figure 3-1: Installation of instrument schematic

Put the main instrument on the working table, which is able to withstand at least 80 kg, in the laboratory nearing the fume hood. The distance between the main instrument and the fume hood should be about 1 m. The guideline is that the end of the vent-hose can be put into the fume hood. The back cover of the main instrument should be 10 cm away from the wall for fume hood. On the wall nearing to the main instrument, there should be a tri-core electrical power socket with ground end away from the main instrument about 1 m. The power voltage should be AC 220-240 V. The socket should have the capability to output 8 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 Vent-pipe

Parts:

- Main machine
- Vent-pipe (09fa001)
- Hoop (00dy002)

Flow:

Connect the vent-pipe to the exhaust outlet which located at the back of the main instrument. A special-purpose screw driver was employed to connect the flank. Screw tightly to fix the tube securely at the outlet.

ATTENTION **Ensure the vent-pipe would not fall off, and also there should be no leak or gap after the installation in order to avoid leakage of acid mist.**

3-1-3 Power Connection

Parts:

- Main machine
- Power line (00cd301)

Flow:

Connect the power supply. Make sure it is earthed. Earth resistance should be equal to or smaller than 4 Omega. Also the electric capacity should not lower than 220 V/8 A.

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

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

3-1-4 Install temperature sensor assembly and gas-guide tube assembly

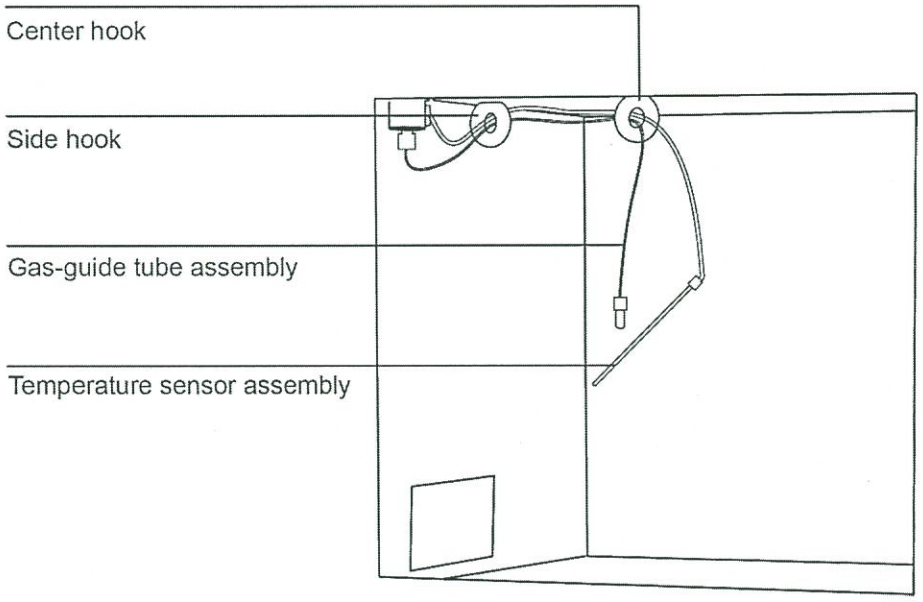


Figure 3-2: Installation of sensors schematic

Parts:

- Temperature sensor assembly (23tj204)
- Gas-guide tube assembly (01tj203)

Flow:

1. Insert the joint of gas-guide tube assembly into the socket at the top of cavity and spin it tightly.
2. The other end of gas-guide tube assembly passes through the side hook and center hook.
3. The probe of gas-guide tube assembly passes through the side hook and center hook.
4. Make sure the red point of joint on temperature sensor assembly and red point on socket are on the same line, push the joint into the socket.
5. Install the cooper bush onto the cooper seat.

ATTENTION **The temperature sensor assembly and gas-guide tube assembly are forbidden winding together.**

3-2 Use of Hardware

The following paragraphs will help you understand how to operate instrument.

3-2-1 Start/Stop Instrument

Press the boat-like power switch to start the instrument. Press the boat-like power switch once again to stop the instrument after the experiment finished. There is an indicator light in the switch. It lights with power on.

3-2-2 Open/Close Safety Door

Press the button on the control panel to open the safety door automatically, and push the safety door to close it.

ATTENTION **Make sure the door hook is in the lock, and can't be pull out after it closed.**

Emergency handle is at the bottom right side of instrument where is near the door. If the door button on the control panel is disabled or the instrument is cut off with power supply you can pull the emergency handle to the direction of door and then safety door will be open.

ATTENTION **Please open the door after microwave stopped and temperature was under 80°C.**

3-2-3 Turn on/off Monitor Screen

You can press the monitor control button on the control panel to turn on the monitor screen and observe the situation in the cavity through it. If you need to turn off the screen just press the button again.

3-2-4 Rotor Control

You can press the rotor control button on the panel directly, the rotor will turn forth and back, if you need to stop the turning of rotor, please press the rotor control button again.

3-2-5 Top Fan Control

You can press the fan control button on the control panel to make the fan work, if you want to stop the fan just press the button again.

3-3 Use of Software

The following paragraphs will introduce you how to operate the software. It consists of setting, editing, creating and starting etc.

3-3-1 Function Selection

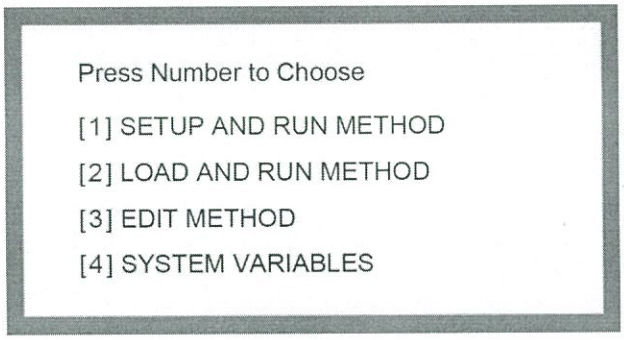


Figure 3-3: Function selection interface

Press the number key (1-4) in the panel to select functions.

3-3-2 Edit Method

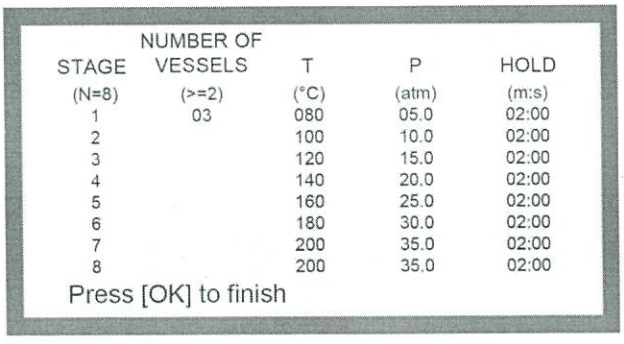


Figure 3-4: Edit parameters interface

In Edit method mode:

a. Set method and run.

b. Edit method→Customer's method→New method.

c. Edit method→Customer's method→Edit method→Name.

You can setup 5 parameters, Stage, Number of vessels, Temperature, Pressure and Hold for this instrument.

A blinking cursor is in the edit method mode. Move the cursor by direction key and input parameters by number keys.

At most 8 steps, maximum working temperature 235°C, maximum working pressure 3.5MPa, 59 minutes and 59 seconds can be set for this instrument.

The NUMBER OF VESSELS is the numbers of vessels in cavity actually.

ATTENTION **At least 2 vessels are used, and one of them must be control vessel.**

3-3-3 Microwave Heating

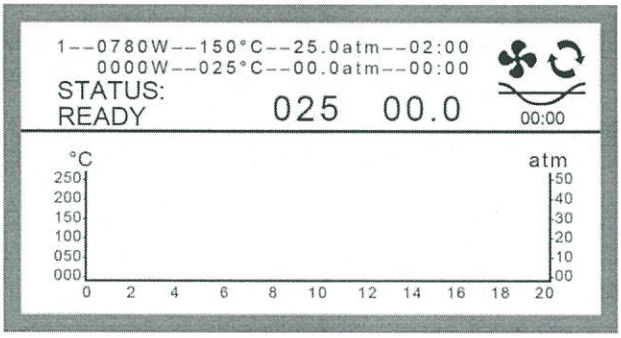


Figure 3-5: Ready interface

Press "OK" and enter into running interface.

Press "Start" to heat.

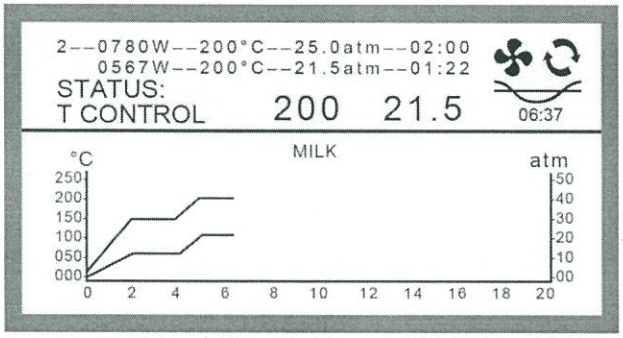


Figure 3-6: Microwave heating interface

Temperature-Time and Pressure-Time curves are illustrated on the screen, at meantime timer starts work until reaction is finished.

You can pause or stop the instrument during reaction. Press "Stop" to pause, then press "Start" to heat again. Press "Stop" once again at pause status to quit from this method. You have to go back to menu manual to find out this method if you would like to continue this experiment.

3-3-4 Manage Method

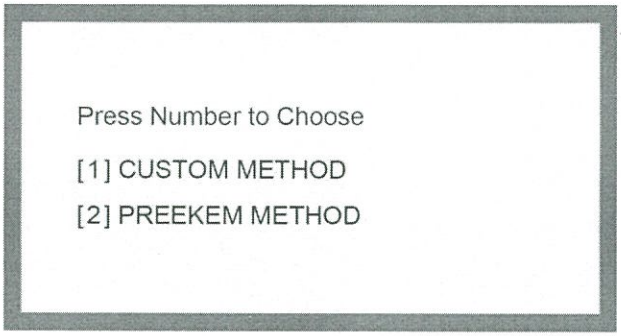


Figure 3-7: Select method library interface

The methods are consisted of CUSTOMER METHOD and PREEKEM METHOD.

You can use CUSTOMER METHOD.

PREEKEM METHOD is built-in method, and customer is not allowed to load it.

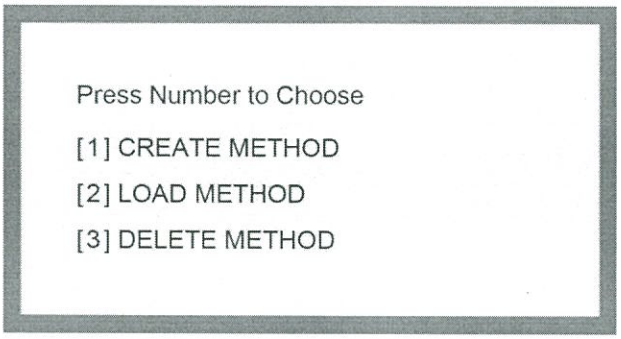


Figure 3-8: Method function selection interface

In CUSTOMER METHOD, you can create method, edit method or delete method. Press the ccorresponding number keys on panel to carry on their functions.

In EDIT METHOD, you can move cursors by direction keys to the method that you want to modify in the set method list, press OK button to enter into the method you have chosen to edit it once again.

In DELETE METHOD, you can move cursors to the method that you want to delete in the set method list, press OK button and confirm it to delete this method.

Figure 3-9: Method list interface

After complete new method, system enters into interface of Input Method Name.

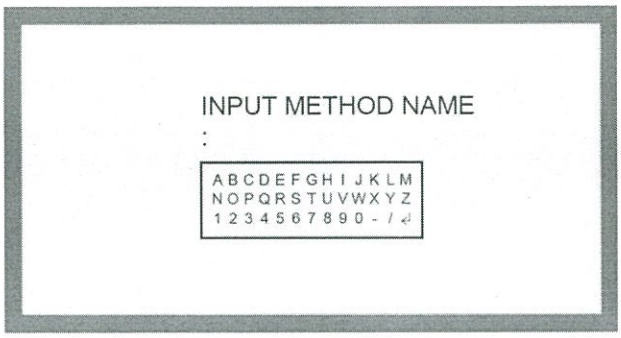


Figure 3-10: Input method name interface

Move cursors by direction keys to the letters, numbers or "-", press OK button to input, when input is over move cursor to the sign of enter and press OK to complete the setting and turn back to main interface.

If the method name is already existed a confirm interface will jump out and then you can choose corresponding number key to cover the existed method or quit this setting.

After finishing input, the method is saved automatically. It can be selected in load method to start microwave heating.

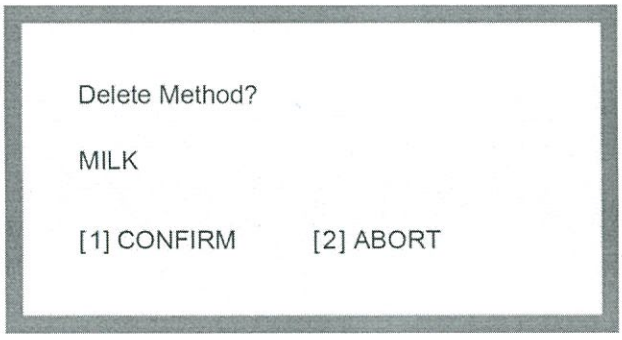


Figure 3-11: Delete method interface

Select the method you want to delete, a dialogue will be displayed, press 1 button to delete permanently, or press 2 button to keep this method.

3-3-5 System Setup

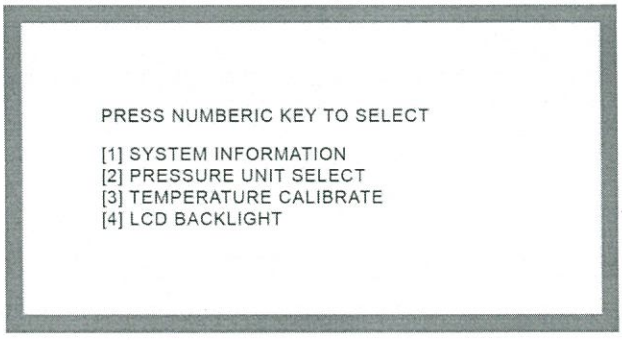


Figure 3-12: System setup interface

Press number key "1-4" on the control panel to choose corresponding functions.

ATTENTION **The function of temperature calibration affects the accuracy of temperature measurement directly, so it's only open to engineer from PreeKem, users are forbidden to operate this function without permission.**

Figure 3-13: Information interface

In system setup interface you can press "1" button to enter into system information interface, you can check version of software, type of temperature sensor and running time of system in this interface.

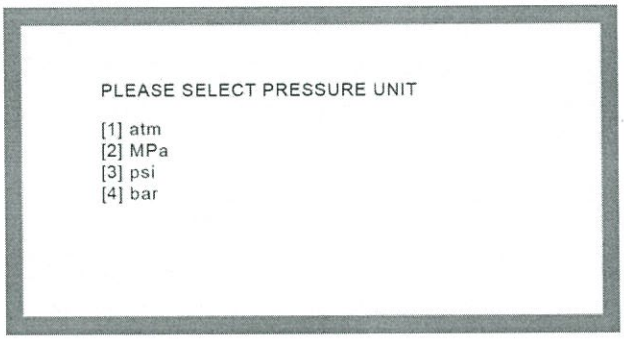


Figure 3-14: Select pressure unit interface

In system setup interface you can press "2" button to enter into pressure unit interface. There are four units of pressure for you to choose, they are atm, MPa, psi and bar, you can press the corresponding number key "1-4" to choose the pressure unit you want, the default setting of unit is atm.

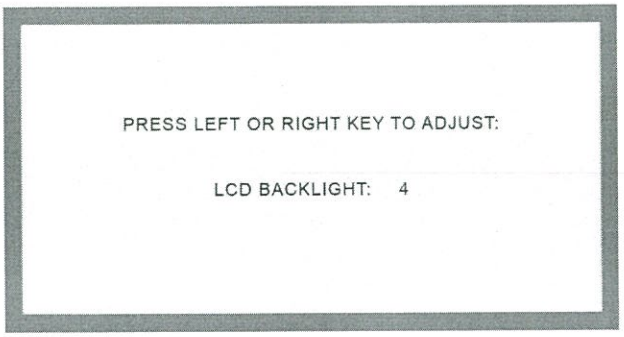


Figure 3-15: Brightness interface

In system setup interface you can press "4" button to enter into brightness interface. You can press the right and left direction button to adjust the brightness of screen, right direction button is used to increase the brightness and left direction button is used to decrease the brightness.

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 display on control screen when power is on	No power supply Connection between power plug and socket is not well Fuse is broken	Make sure there is power supply Make sure power plug is connected well with socket Check the fuse, replace a new one when needed.
Microwave cannot be started, screen displays door is not closed	Oven door is not closed	Make sure oven door is closed well
Temperature reading is abnormal	Temperature sensor is not installed Temperature sensor has a failure	Install the temperature sensor correctly Replace the temperature sensor with a new one
Screen displays microwave is working, but temperature is not rising actually	Temperature sensor has a failure	Replace the temperature sensor with a new one
Turntable does not rotate	Rolling balls are stuck in turntable or coupling half is worn down	Check the rolling balls at the bottom of the turntable Replace a new coupling half
Screen is lighten, but no content is on it	Brightness of screen is too high or too low	Adjust the brightness of screen
Screen displays "door is open"	Oven door is not closed	Close the door and then start the testing
Sparking appears in cavity	There is residual or reagent in the liner for temperature sensor probe	Clean and dry the liner
Screen displays "time-out"	Temperature setting range spans too widely Poor microwave absorbency of sample No sample is placed in control vessel	Shorten the experiment temperature span Add the steps in experiment Make sure sample in control vessel is the same with which in standard vessel

Sample digestion is not completed	Leakage phenomenon Target temperature is set too low to reach the sample digestion temperature Inappropriate acid proportion Inappropriate acid types Digestion time is not long enough	Expand the skirt size Increase the digestion temperature Confirm the proportion of acid Choose the right types of acid Increase the digestion time
Only the sample in control vessel is digested completely	Sample dissolution cup of control vessel is used too frequently resulting in its stronger microwave absorbency	Select a newer sample cup as inner cup of control vessel
leakage in digestion vessel	Not yet expand the skirt size The deflation screw is not tightened Safety film ruptures	Expand the skirt size Tighten the deflation screw Replace a new safety film

Appendix 1 Principle of Microwave Heating

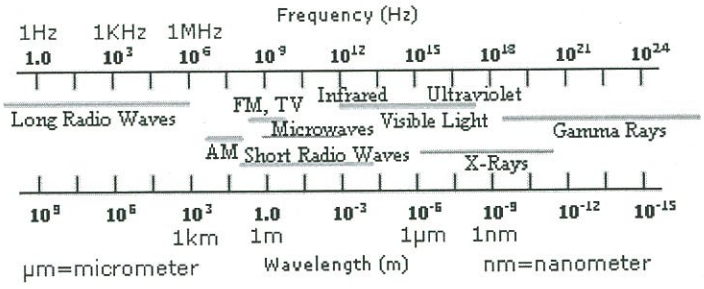


Figure 22: Electromagnetic Spectrum

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.

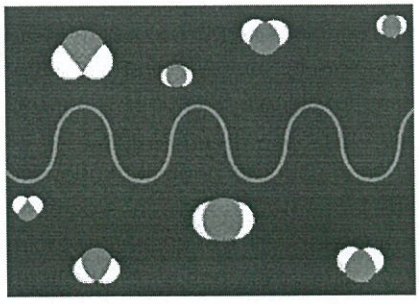


Figure 23: Molecular dipole rotating

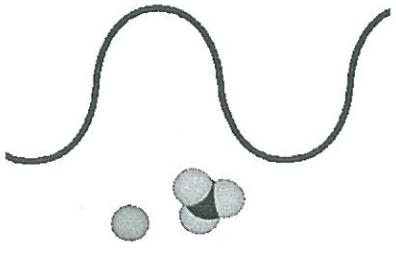


Figure 24:Ionic conduction

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 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 I 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)

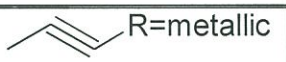
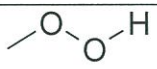
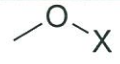
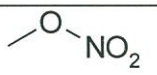
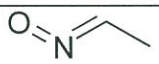
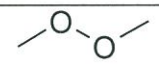
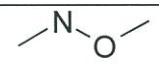
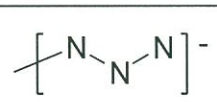
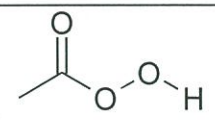
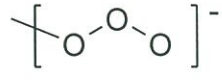
4 Forbidden Samples for Microwave Digestion

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