



Microwave Digestion System

Operation Manual



AE-MD-610

A & E LAB (UK) Co.Ltd.

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A & E Lab (UK) Co., Ltd.

Tel: +44 20 3627 1763

Fax: +44 20 3627 1763

www.aelabuk.com

Packing list

| No. | Item Name | Pictures | Qty. | Unit | |
|-----|-------------|---------------------------------|--|-----------|-----|
| 1 | Instrument | Main Engine |  | 1 | set |
| 2 | Accessories | Power Cord |  | 1 | pcs |
| 3 | | Manual |  | 1 | pcs |
| 4 | | Digestion Vessels |  | 6/8/10/12 | set |
| 5 | | Sample Rotor |  | 1 | set |
| 6 | | Auxiliary Platform |  | 1 | set |
| 7 | | Vessel Holder |  | 1/2 | set |
| 8 | | Unload Tool |  | 1 | pcs |
| 9 | | Pressure Calibration Ring (PCR) |  | 2 | set |
| 10 | | Exhaust Pipe |  | 1 | set |
| 11 | | Fuse(8A) |  | 2 | pcs |

Safety Guidelines

1. DO NOT install the instrument in the fume hood to avoid any damage or problem caused by acid, alkali or corrosive gas etc.
2. Make sure there is enough space for instrument ventilation and user operation.
3. DO NOT do empty run. There must be one or more digestion vessels filled with sample solution or acid on the rotor.
4. Make sure all parts of the digestion vessel, inner vessel, outer vessel, sample rotor, auxiliary platform and inner surface of furnace chamber should be clean and dry.
5. Keep the signal monitoring surface flat and smooth without any marking and scretch on the surface.
6. Wear acid proof and heat insulation gloves during operation.
7. Reduce the sample volume appropriately according to the difficulty of the sample digestion and the viscosity of the sample.
8. Samples containing fat, oil, nitroglycerine, aromatic compounds, nitro aromatic compounds, and volatile samples must do pre-digestion before microwave digestion.
9. During pre-digestion procedure, the sealing cover of the inner vessel should be taken off to make sure the digestion procedure is under normal pressure.
10. The temperature and pressure measuring probes must be cleaned with alcohol cotton swabs.

Introduction

Microwave Digestion System AE-MD-610 is a unit could well satisfy the requirements of modern analytical instrument for sample preparation, with the advantages of fast and uniform heating, less reagent dosage, low blank value, not easily polluted, energy efficient, etc. Especially in the analysis of volatile element, it's better to maintain sample integrity and has a higher sample rate.

Its stable performance, easy operation and simple panel and human-centered module design make it outstanding during all competitive products. It is the best choice for sample preparation in AAS, UV-VIS, ICP-AES, AFS, GFAA, XFS, ICP-MS, HPLC analysis which reduces sample preparation time and improves efficiency.

Now the system is widely used in food, textile, Geology, Metallurgy, Coal, Biological, Cosmetics, Petrochemicals, Environment, waste water treatment, battery manufacturing fields, etc.

Features

1. Closed vessel microwave digestion
2. Simultaneous digestion of 1-12 samples (alternative)
3. 10 digestion steps method with storage up to 255 programme
4. Self vent-reseal digestion vessel
5. Multiple layers of TEFLON coating for the cavity, high-power exhaust system
6. Alarms or sensors for detection of abnormal pressure, temperature, sound
7. Real time monitoring of changes in digestion
8. Contactless IR temperature sensor to measure individual sample temperature
9. Contactless optical fiber pressure sensor to measure individual sample pressure
10. Automatic calibration and troubleshooting
11. Vertical pressure release for safe operation
12. Multiple safety protection function

Specifications

| | |
|---------------------------------|------------------------------------|
| Model no | AE-MD-610 |
| Sample quantity | 6/8/10/12 per batch |
| vessel volume | 100ml |
| Microwave power | 1000W |
| Microwave frequency | 2450MHz |
| Temperature control range | 50-250°C |
| Temperature limit | 300°C |
| Pressure control range | 0-60Bar |
| Microwave leakage | <5mW/cm ² |
| Display Controller | 7 inches touch screen |
| Inner vessel material | Inert polymer TFM |
| Outer vessel material | Composites of PEEK and Glass Fiber |
| Pressure control and testing | Non-contact full scanning |
| Temperature control and testing | Non-contact full scanning |
| Power | AC220V, 50/60Hz,10A |
| Dimension | 490mm×560mm×630mm |
| Host Net Weight | 47kg |

Instrument Components Introduction

1. Host Engine

Main components of the instrument.

2. Display Controller

It's the liaison between operator and instrument, using "man-machine conversation" control model.

3. Pressure Control System

It monitors and controls the pressure changes in real time during the digestion process.

4. Temperature Control System

It monitors and controls the temperature changes in real time during the digestion process.

5. Digestion Vessel

It's combined by a inner vessel and a outer vessel. The inner vessel used to contain the samples and acids, it provides closed environment for high temperature and high pressure digesting. The outer vessel provides a protective environment for high temperature and high pressure digesting.

6. Rotor System

It is combined by a drive motor, a sample rotor and an auxiliary platform. The sample rotor provides space for loading the digestion vessel. The auxiliary platform provides a stable and smooth platform which also reduces rotary resistance. The drive motor keeps the sample rotor rotating uniformly and continuously on the auxiliary platform toward the same direction.

7. Exhaust System

It contains a high power exhaust fan and a exhaust pipe. The system out the spilling acids from the digestion vessel in time to avoid erosion and also cool down the digestion vessel when digestion finished. The collected waste gas is discharged by the exhaust pipe.

8. Programming

Ensures intelligent and efficient running of the instrument.

9. Vessel Holder

Users can place the inner vessel into the vessel holder.

10. Repair Tool

Users can repair the sealing cover that not sealed very well with it. Method of use: press the repair tool head into the sealing cover, press and turn the repair tool 1 to 2 minutes then pull out.

11. Unload Tool

Users can take out the inner vessel that too tight to remove out from the outer vessel after digestion especially when acid spilled. When it happens, please use unload tool to insert into outer vessel bottom and remove inner vessel.

12. Pressure Calibration Ring (PCR)

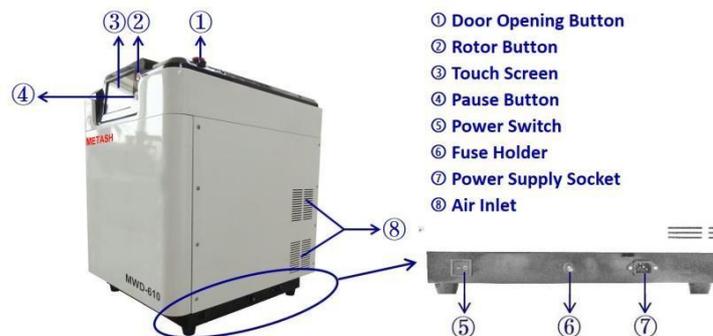
After a long period of running, the original output parameters of the instrument may need to be calibrated. Users can calibrate instrument pressure signal in Calibration Interface with the PCR. Each set of PCR consists of two components, the thick one is PCR-5 that is used to calibrate P5, and the thin one is PCR-2.5 that is used to calibrate P2.5.

13. Fuse

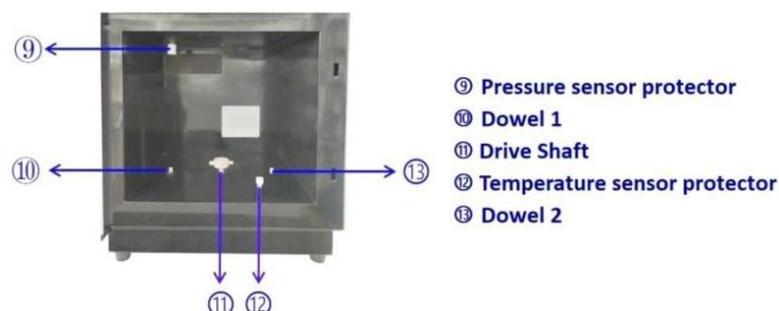
Spare part.

Host Engine Structure

A. External Structure



B. Internal Structure

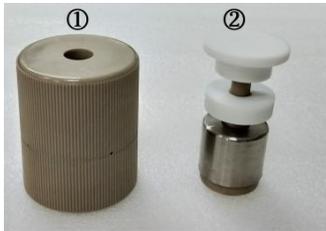
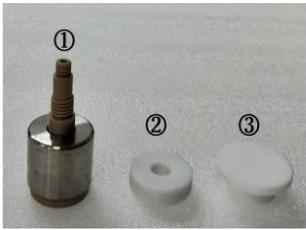
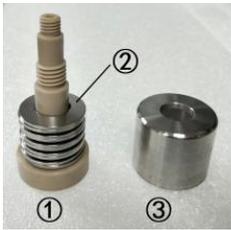


Important Accessories Introduction

A. Digestion Vessel

1. Structure

- (1) The Digestion Vessel consists of an Inner Vessel and an Outer Vessel.
- (2) The Inner Vessel consists of an inner vessel body (cup body) and a sealing cover.
- (3) The Outer Vessel consists of a vessel body and a vessel cover assembly.
- (4) The vessel cover assembly is assembled with a vessel cover and a pressure sensing module.
- (5) The pressure sensing module consists of a pressure component, a lock nut, and a signal board.
- (6) The pressure component consists of a guiding rod, a shield, and a set of springs.

| Digestion Vessel | |
|---|---|
|  <p>Digestion Vessel:</p> <ul style="list-style-type: none"> ① Outer Vessel ② Inner Vessel |  <ul style="list-style-type: none"> ① Outer Vessel Body ② Cover Assembly ③ Inner Vessel Body ④ Sealing Cover |
| Cover Assembly | |
|  <ul style="list-style-type: none"> ① Cover ② Pressure Sensing Module | |
| Pressure Sensing Module | Pressure component |
|  <ul style="list-style-type: none"> ① Signal Board ② Lock Nut ③ Pressure component |  <ul style="list-style-type: none"> ① Guiding Rod ② Spring ③ Shield |

2. Assembly process of Cover Assembly

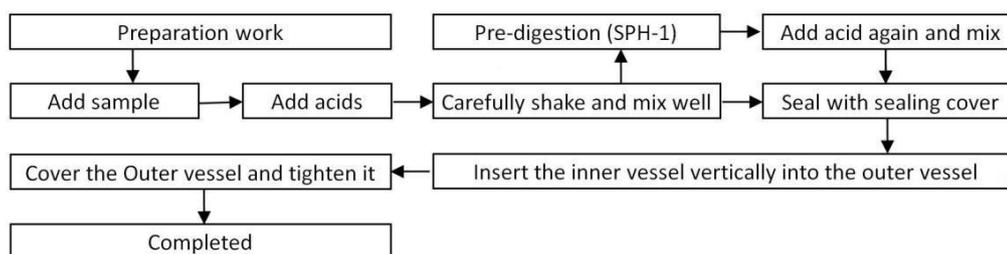
- (1) Put the springs in order into the guiding rod, and also put the shield into the guiding rod.
- (2) Insert the guiding rod into the vessel cover.
- (3) Gently screw the lock nut into the guiding rod.
- (4) Screw the signal board into the guiding rod, and then screw more with a little force to avoid the signal board loose.

(5) Then the assembly of Cover Assembly is finished.



3. Assembly process of Digestion Vessel

(1) Assembly procedure of the Digestion Vessel flowchart:



(2) Preparation work:

➤ **Check sealing of the sample digestion vessels:** When slowly pressing the sealing cover into the cup body there is a friction; when quick pull out the sealing cover from the cup body there should be a sealed sound. Before using the sample digestion vessel, please match the cup body and the sealing cover first, so as to ensure the good sealing performance of the combined sample digestion vessel. Not well sealed sample digestion vessel can be improved by using the repair tool to grind the seal cover. Method of use: press the repair tool head into the sealing cover, press and turn the repair tool concentric 1 to 2 minutes then pull out.

➤ Check the Signal board: It must be clean, dry without loose and any scratch.

(3) **Loading sample:** Load the weighed sample to a clean inner vessel, and make sure there is no sample suspended on the wall.

(4) **Add acids:** Choose suitable acids. If there is suspension, please use acid to wash sample off into the vessel when adding acid.

(5) **Mixing:** Carefully shake the inner vessel to make the sample and the acids mix well and remove the gas off the vessel as much as possible. Make sure to operate gently to avoid acid spilling out to burn other people. Caution: Some samples need to be pre-digestion before microwave digestion (Please choose SPH-1 for pre-digestion).

(6) **Sealing:** Seal the inner vessel with the sealing cover. **Note: For unknown samples and samples that will generate much gas, or contain much oil, or with complex components, pre-digestion is required, and then add acids again before sealing .(Please choose SPH-1 for pre-digestion). After sealing, pay attention to the finger gesture: one finger pressing the sealing cover; the remaining four fingers holding the inner vessel body.**



- (7) **Tighten:** Insert the sealed inner vessel gently and vertically into the outer vessel carefully. Use one finger to insert from the bottom against the inner vessel, then cover the outer vessel and tighten it, and then screw more with a little force to avoid loose. **Note: Put the inner vessel carefully into the outer vessel; Pay attention to finger gesture: One finger pressing the sealing cover, the remaining four fingers holding the inner vessel body to avoid liquid spill out to burn other people. Please do not tighten tightly, it's better to rotate the vessel cover until it meets resistance, and then tighten it for about 15°**

4. The disassembly process of Digestion Vessel



- (1) When digestion completes and vessels cool down, open the door and hold the cover of the outer vessel with acid proof and heat insulation gloves and take it out. Put the digestion vessels into the fume hood for 10 minutes.
- (2) One hand hold the outer vessel cover and the other hand hold the vessel body, loosen and then stop.
- (3) Gently and slowly open the outer vessel cover, stop when there is a sound of air blow out or brown air blow out, wait for the air blown sound or the brown air disappeared. Repeat this operation until the pressure released.
- (4) While removing the outer vessel cover, **pay attention to the finger gesture: one finger pressing the sealing cover, the remaining four fingers holding the outer vessel body to prevent the sealing cover pop up.**
- (5) Use a finger to upward the inner vessel through the hole of bottom of outer vessel. Please use the unload tool if use finger cannot remove the inner vessel: Put the unload tool on the fume hood table, put the bottom of inner vessel on the unload tool through the hole of bottom of outer vessel, pull the outer vessel body downward with force, then take out the inner vessel. **Pay attention to the finger gesture: one finger pressing the sealing cover, the remaining four fingers holding the inner vessel body.**
- (6) Cover the sealing cover with one hand, fingers pressing on the peripheral surface of the sealing cover, the other hand hold the inner vessel body; spin up and pull out the sealing cover. **Note: Be sure to spin up and pull out, only spin up not pull out cannot open the inner vessel. only pull out not spin up may cause the reagent in the inner vessel shake and spill out to cause loss of elements or burn other people.**
- (7) While removing the sealing cover, quickly put the sealing cover upward to avoid reagent loss which suspends on the sealing cover. Gently shake the inner vessel body to accelerate the gas flow out.
- (8) Insert the inner vessel body into the SPH-1 instrument to heat and drive the acid until the reagent left about 1-2ml.
- (9) Transfer the reagent to the volumetric flask, rinse the inner vessel and the sealing cover 2-3 times and transfer the rinse-reagent into the volumetric flask.

5. Important Notes of using Digestion Vessel



- (1) Make sure the sealing parts of the inner vessel are in good and without any scratches or damage.
- (2) Make sure each part of digestion vessel is in a dry and clean condition, except the inner wall of the inner vessel directly contacting the sample and acids.
- (3) Clean suspended acid on vessel wall in time. **Note: The inner vessel shall be soaked in diluted nitric acid for 12 hours and then cleaned; It is not allowed to use any other acid or organic reagent to clean outer vessel.**
- (4) Please make signal board on the outer vessel clean, dry and smooth without any scratch.
- (5) Please place the outer vessel cover with signal board upward to keep the signal board flat, smooth, dry and clear.
- (6) **Note: During the assembly, make sure the signal board of pressure sensing module is no loose.**
- (7) **Note: Pay attention to the tightness when tightening outer vessel, it's better to rotate the vessel cover until it meets resistance, and then tighten it for about 15 °.**

B. Rotor System

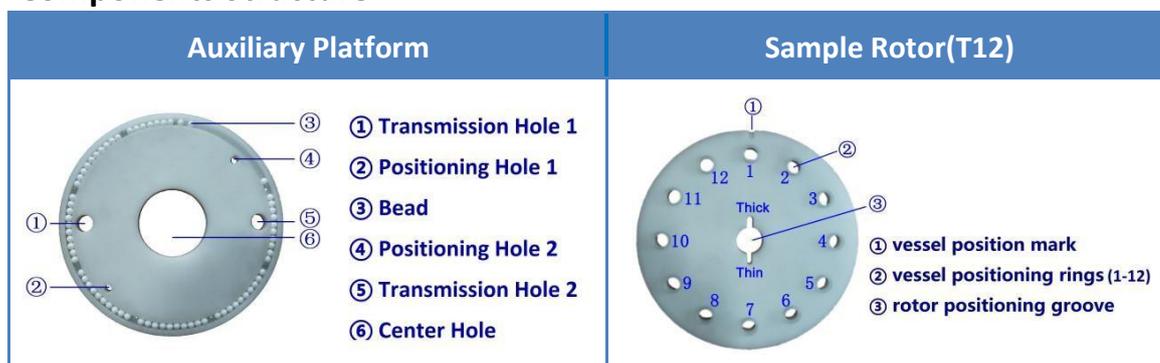
1. Components

The rotor system is combined by a drive motor, a sample rotor and an auxiliary platform.

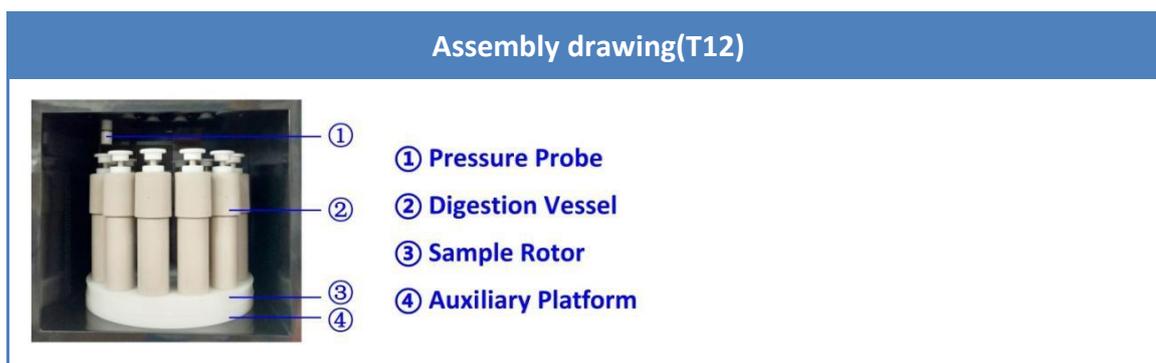
2. Types of Sample Rotor

There are four types of Sample Rotor, and the number of digestion vessel is 6, 8, 10 and 12 respectively.

3. Components Structure



4. Roter and Digestion vessels



Instrument Installation

A. Installation Conditions

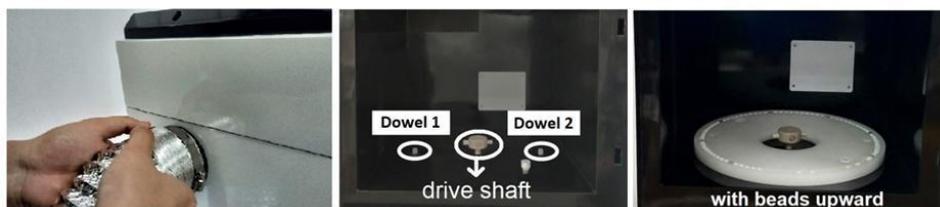
1. The instrument should be installed on a horizontal and stable worktable over 600mm long and over 650mm wide which could bear over 60kgs.
2. A bigger worktable is required if assist device is also installed on the same worktable.
3. For correct operation, the instrument should have sufficient air flow. Allow with 200mm of space at the back and both sides of the instrument.
4. The instrument is around 630mm tall, please choose suitable worktable for easy operation.
5. It is allowed to place the worktable next to the fume hood, but it is strictly prohibited to put the instrument into the fume hood.

B. Installation Environment

Working environment affects a lot of instrument performance and lifetime. Please check below environment before installation.

1. Temperature: 5~40°C.
2. Humidity: 45~70%RH.
3. Good ventilation, no external corrosive gas, no external strong magnetic interference, and avoid direct sunlight.

C. Installation Procedure



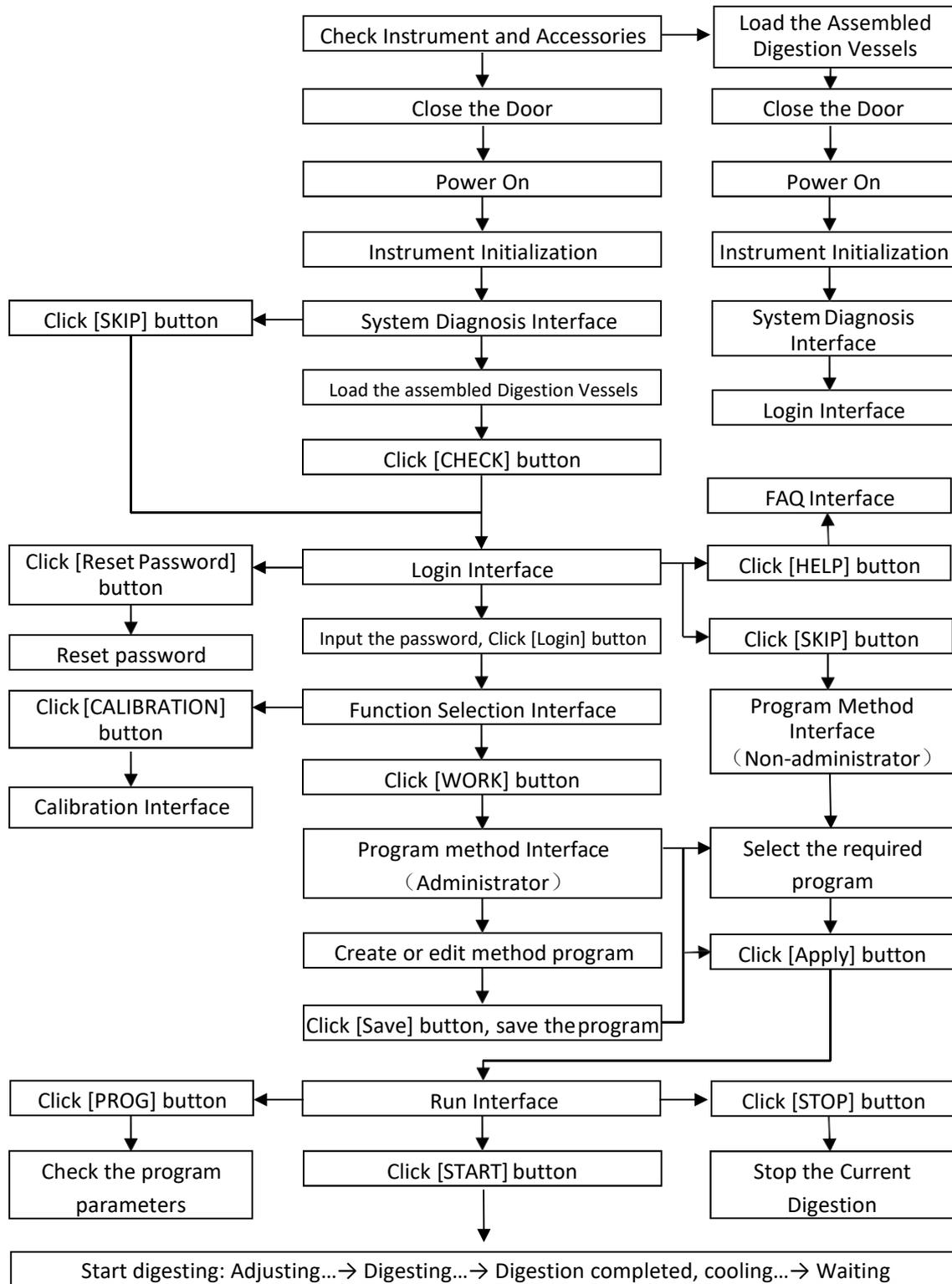
1. Take out the exhaust pipe from the accessory box, and tighten it in the air outlet of the instrument, and put another end into fume hood
2. Take out the auxiliary platform from the accessory box and fix it into the two dowels in the furnace chamber with the beads upward.
3. Take out the sample rotor from the accessory box and fix it into the drive shaft in the furnace chamber. Pay attention to the thick card slot in line with the drive shaft thickness direction.
4. Put the well assembled digestion vessel into the vessel positioning ring. For multiple vessels digestion, make the digestion vessels evenly arranged in the sample rotor.



5. Connect the power cord and ensure good grounding .
6. Turn on power switch and the instrument will automatically do system diagnosis. If user does not load any digestion vessel, the system will prompt “Undetected Digestion vessel”. Load one or several digestion vessels according to requirements, close the door and press [CHECK] button to continue and then enter into login interface. If user needs admin authorization, please enter password in login interface and press [Login] to enter function selection interface. If not, please press [SKIP] to jump to the program method interface.

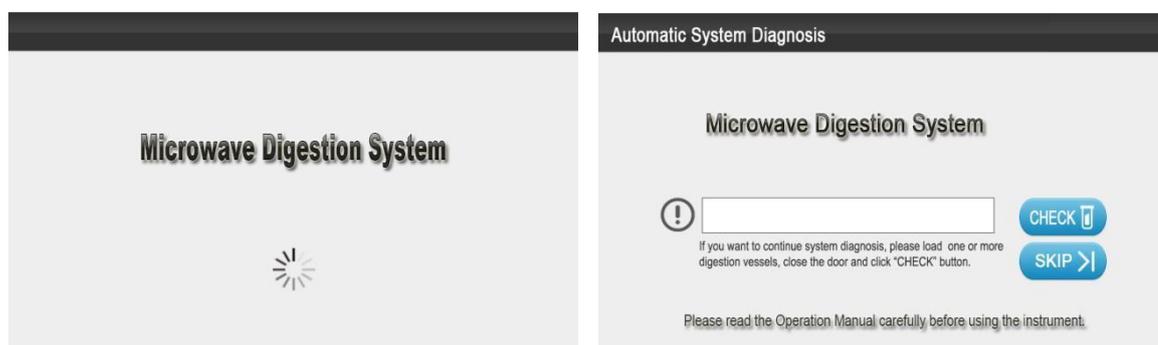
Instrument Operation

A. Operation Flowchart



B. Operation Interfaces

1. Instrument Initialization and Automatic System Diagnosis Interface

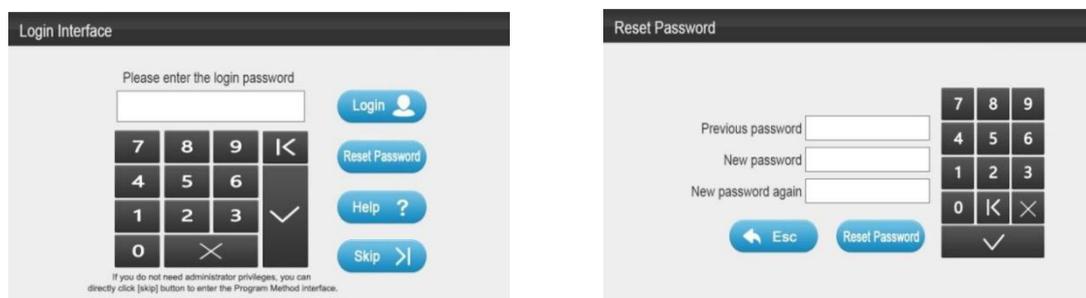


- (1) Before turning on the power, please check all wires to ensure all wires have been connected properly.
- (2) Check the chamber, and make sure the auxiliary platform and the sample rotor are normally, then close the door.
- (3) Turn on the power, the instrument starts to initialize.
- (4) When initialization finished, system will go into the Automatic System Diagnosis Interface.

2. Automatic System Diagnosis Interface

- (1) When first use the instrument or doing periodic diagnosis, please load one assembled digestion vessel according to the requirements and click the button of [CHECK] to continue system diagnosis.
- (2) The problems found during the Automatic System Diagnosis will be prompted to display in the long text frame of the interface. Click [SKIP] button to enter the Login Interface. In the Login Interface, click [Help] button to enter the Help Interface and user can refer to related questions and analysis (FAQ).
- (3) After System Diagnosis, system will enter directly Login Interface.

3. Login Interface



- (1) Click the password display frame, enter the login password (Initial password: 1234), click [v] to confirm. Then click [login] button to enter the Function Selection Interface (administrator).
- (2) Click [Reset Password] button and enter Reset Password Interface. The administrator

can modify the login password (four digits) on this interface.

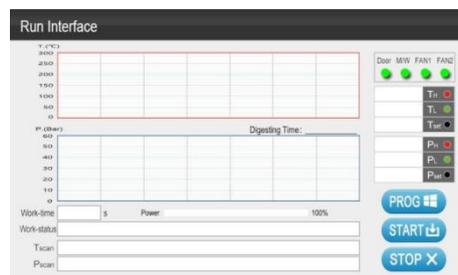
- (3) Click [Help] button to enter the Help Interface and users can refer to related questions and analysis (FAQ).
- (4) Click [Skip] button to enter the Program Method Interface (non-administrator).

4. Program Method Interface(non-administrator)



- (1) Enter the Program Method Interface, the system will load digestion system that used last time by default.
- (2) The system can set 1-255 kinds of program methods. There are 8 preset program methods in the Program Method Interface, which help users to enter the digestion process conveniently and quickly.
- (3) Click the name of left program method can load the corresponding program. If user needs to load 9-255 program methods, please input the corresponding number into the text box of the program No. and click the [Load] button.
- (4) Under normal permissions, user cannot modify the related parameters in the program or create a new digestion program. Please enter the administrator's Program Method Interface, and then create or edit the method program. Please refer to the Chapter 6.4.7.
- (5) Click [Esc] button to enter the [Login Interface].
- (6) Click [Apply] button to enter the [Run Interface].

5. Run Interface

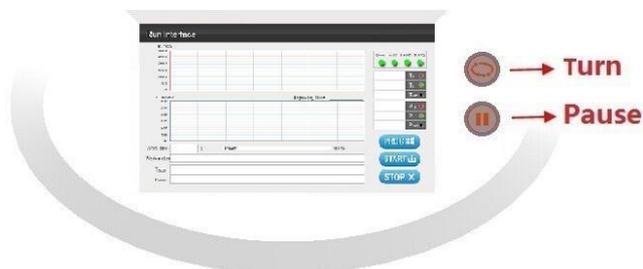


- (1) Working parameters are displayed in real time on the right side of the Run Interface.
 - **Door:** It means the door closed safely when the door status light is green. User can click [START] to start the application program for sample digestion. It means

- the door closed unsafely when the door status light is grey, and then the button of [START] is invalid. Please check whether the door is closed safely or not.
- **M/W:** When microwave status light is green, it means the instrument in working status. Do not lean on the instrument to avoid microwave irradiation.
 - **FAN1 and FAN2:** The status lights are green when the fan1 and fan2 work normally.
 - **T display frame:** Display in real time the highest temperature value (T_H), the lowest temperature value (T_L) of the reagent in all digestion vessels and the program setting temperature value (T_{Set}).
 - **P display frame:** Display in real time the highest pressure value (P_H), the lowest pressure value (P_L) of the reagent in all digestion vessels and the program setting pressure value (P_{Set}).
- (2) On the left side of the Run Interface:
- **Diagram of curves:** The red frame is the temperature curve frame, which shows the change of the temperature in the digestion vessel in real time. The blue frame is the pressure curve frame, which shows the change of the pressure in the digestion vessel in real time. The digestion time between the red curve frame and the blue curve frame is the total time of the effective steps. The combination of charts can more easily show the change of sample digestion.
 - **Microwave power progressing bar:** Display the output state of microwave power in real time.
 - **Working-time display:** Shows the time used in each stage of run in real-time (s).
 - **Working-status display:** Display or prompt the current condition or the fault hints in the operation of the instrument.
 - **Tscan display and Pscan display:** Display the temperature and pressure changes of each digestion vessel in real-time.
- (3) Click [START] button to enter the digestion process, and the Working-status display frame will in turn display: **Adjusting→ Digesting→ Digesting completed, cooling (buzzer sound) → waiting (buzzer sound)**, until then the digestion process completes.
- For the first time to enter the Run Interface, the Working-status display frame displays with "Waiting".
 - After clicking the button of [START], the Working-status display frame displaying with "Adjusting".
 - After "Adjusting", the rotor positions that do not load the digestion vessels, the values of temperature and pressure in " T_{scan} " and " P_{scan} " will display 0, and the Working-status display frame displaying with "Digesting".
 - When the working-status enters "Digesting", check whether the exhaust fan is ventilated or not, if not, please check it.
 - If abnormal situation occurs, the Working-status display frame will display prompts such as "Zero Warning", "Door Unlocked", and so on. Please refer to the corresponding common problems in the Help Interface.
 - After "Digesting", working-status display frame will show "Digesting completed, cooling" with the buzzer sound (**Cooling stop conditions: all vessels temperature $\leq 100^\circ\text{C}$ and pressure $\leq 0.3\text{MPa}$, or cooling time reached 3000s.**) If digestion

vessel start cooling, please don't open the door until the working-status shows "waiting", and then take out the vessels to the fume hood. This can avoid accident due to higher temperature of the outer vessels without cooling. **NOTE: The temperature of the outer vessel will be very high after digestion, please hold the cover of the outer vessel with acid proof and heat insulation gloves while taking out the digestion vessels to avoid getting burnt.**

- (4) Click [PROG] button to return to the Program Method Interface (non-administrator). User can search the parameters values.
- (5) Click [STOP] button to stop the current application procedure. **Note: Please operate carefully,** because after clicking [STOP], user can't click [START] to start digestion again until the pressure in all loaded digestion vessels changes to zero. If user needs to use the pause function, please press the mechanical button [] which is next to the touch-screen.

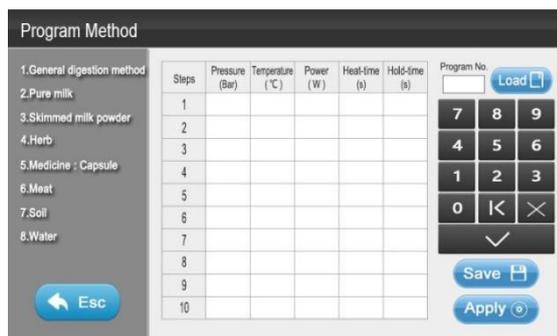


6. Function Selection Interface(Administrator)



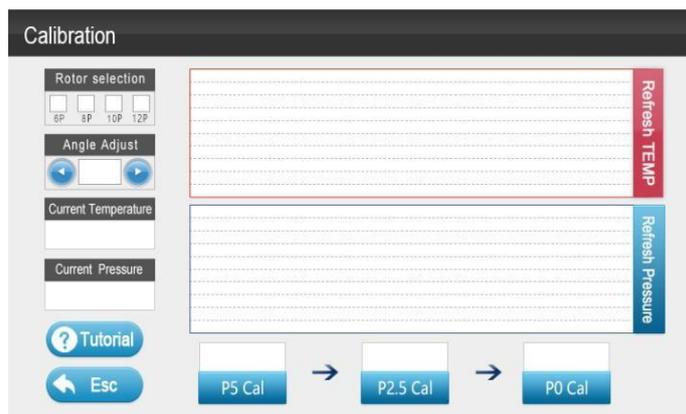
- (1) Click the button of [WORK] to enter the Program Method Interface.
- (2) Click the button of [CALIBRATION] to enter the Calibration Interface.
- (3) Click the button of [] to return the Login Interface.

7. Program Method Interface(Administrator)



- (1) Administrator can edit program steps and parameters in Program Method Interface. For examples: Pressure, Temperature, Power, Heat-time and Hold-time.
- (2) Click the button of [Save] to save the current modification, Click the button of [Apply] to invoke the current program and enter the Run Interface, click the button of [Esc] to return the previous interface that is Function Selection Interface.
- (3) Users can input 1-255 into “Program No.” display frame and click the button of [Load] to load the corresponding program.
- (4) Each digestion program consists of 10 working steps; each working step consists of a heat-time step and a hold-time step. That means, each digestion program consists of 20 digestion steps, which are enough to meet all kinds requirements of complicated samples in the control process of digestion.
- (5) In the process of digestion, steps that did not set parameters or steps parameter value of heat-time and hold-time are all “0”, the system will automatically start diagnosis and regard all the following steps invalid. The instrument system runs only effective steps and will stop in case of the invalid steps.
- (6) Click the parameter in the display frame, edit or modify it by using the “editing keyboard”. For example, click the program No. display frame, the background color become blue. When clicked, the background recover as before and there is a black cursor on the right of the display frame. Input the new program No. value using the “editing keyboard”, the new program No. value will show blue and on the right side of the display frame, click [←] to delete the recently input value, click [×] to cancel the modified editor, click [✓] to replace the original black value with the blue value, and the blue value will be saved as black.
- (7) Pressure setting range is 0~60Bar; Temperature setting range is 50-250 °C . Time setting range is 0~9999s; Power setting range is 0~1000W. All parameters should not exceed the setting range. Otherwise, the button of [✓] is invalid and all the values cannot be saved and user has to edit input value again.

8. Calibration Interface (Administrator)



- (1) Click [CALIBRATION] button on the Function Selection Interface to enter the instrument Calibration Interface.
- (2) **Note: The Calibration Interface is only for internal parameters calibration. Personnel without training are not allowed to operate in [CALIBRATION] interface.**
- (3) Click [Tutorial] to enter the interface for detail introduction of the instrument calibration procedures.
- (4) Click [Esc] to return to the Function Selection Interface.
- (5) **Instrument calibration Tutorial:**
 - **Note: Calibration period generally is one week, but shall be adjusted according to instrument condition and using frequency. Please calibrate instrument if user restart instrument after long time.**
 - **Calibration Principle and Sequence: Angle Adjust → Pressure Calibration → Temperature and Pressure Verification.** User must follow above calibration sequence and cannot skip any calibration steps.
 - **Calibration Procedure:**
 - a) Assemble one set of vessels (no sample or liquid inside) and put on No.2 position on the rotor, close door and press [].
 - b) Calibrate instrument angle according to Angle Adjust instructions.
 - c) Calibrate pressure in sequence according to Pressure Calibration (P-Calibration) instructions.
 - d) After finishing step b) and step c), press [ESC] to Function Selection Interface, and press [Work] to enter Program Method Interface.
 - e) Set temperature and pressure verification parameters: Pressure 20Bar, Temperature 180°C, Power 1000W, Heat-Time 300s and Hold-Time 600s.
 - f) Put 10ml purified water into a clean set of vessels (TK-V100) and seal it. Run digestion and after 180°C hold-time, pressure inside vessel shall be around 10Bar.
 - g) Instrument calibration finished.
 - h) **Note: Calibration period generally is one week, but shall be adjusted according to instrument condition and using frequency. Please calibrate instrument if user restart instrument after long time.**

(6) Angle Adjust instructions:

- a) Press [Refresh Pressure] on the right side of pressure monitoring frame until a smooth signal peak appears.
- b) Check if the vertical line in the frame is in the middle of the signal peak. If yes, please go directly to Pressure Calibration. If not, please follow below steps to do Angle Adjust.
- c) Press [] or [] on the left upper corner to adjust angle value. Press [Refresh Pressure] again to see if the vertical line is in the middle of the signal peak or not.
- d) Repeat step c) until vertical line is in the middle of the signal peak.
- e) Angle Adjust finished.

(7) P-Calibration instructions:

- **Calibration Principle: Place the same digestion vessel at the same vessel position and cannot be interchanged; and make sure calibration sequence: P5 → P2.5 → P0.**
- **P5 calibration**

Select a well assembled digestion vessel as vessel A and a fixed vessel position from the Sample Rotor as position A. First, put the PCR-5 into the position A and load the vessel A into the position A, then close the door. The Sample Rotor starts to rotate and the calibration begins after clicking [P5 Cal] button, the signal values in the "Pressure" frame are detected. P5 Cal is completed after the Sample Rotor stops moving, and the signal values will be updated and saved to [P5 Cal] frame automatically.

The signal value range of P5 is 200±20. If the calibrated signal value is not in the range, the alarm will be off. In this case, please re-calibrate the pressure signals according to the calibration sequence: P5 → P2.5 → P0, or refer to the Help Interface of P5 Warning.
- **P2.5 calibration**

Open the door and take out of the vessel A and the PCR-5, put the PCR-2.5 into the position A and load the vessel A into the position A, close the door. The Sample Rotor starts to rotate and the calibration starts after clicking [P2.5 Cal] button, the signal values in the "Pressure" frame are detected. The P2.5 Cal is completed after the Sample Rotor stops moving, and the signal values will be updated and saved to [P2.5 Cal] frame automatically.

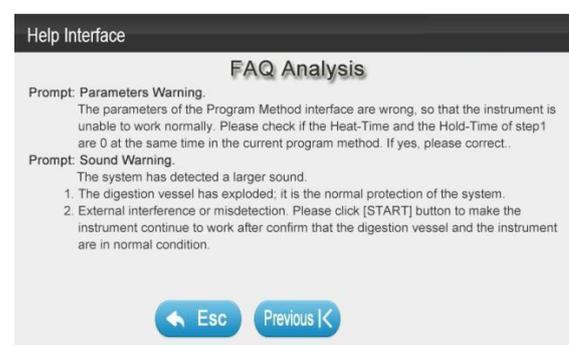
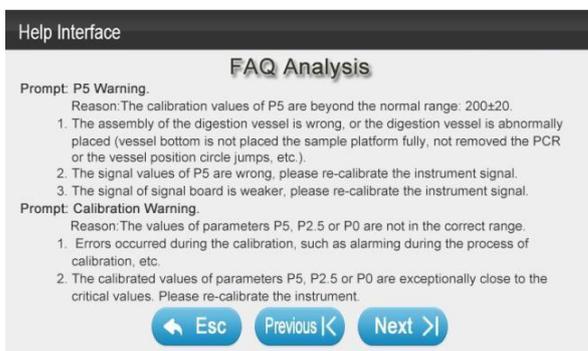
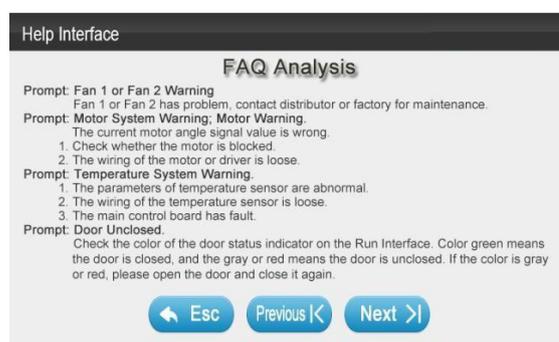
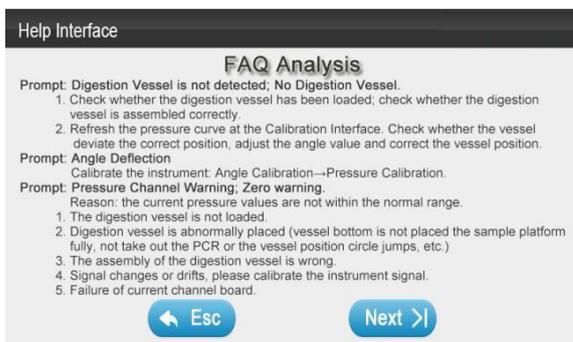
The difference between the corresponding values of P5 and P2.5 must be more than 20. If the difference is smaller than 20, the alarm will be off. In this case, please re-calibrate the pressure signals according to the calibration sequence: P5 → P2.5 → P0, or referring to the Help Interface of Calibration Warning.
- **P0 calibration**

Open the door and take out of the vessel A and the PCR-2.5, load the vessel A directly into the position A, and then close the door. The Sample Rotor starts to rotate and the calibration starts after clicking [P0 Cal] button. The signal values in the "Pressure" frame are detected. The P0 Cal is completed after the Sample Rotor stops moving, the new signal values will be updated and saved to "P0 Cal" frame automatically.

The difference between the corresponding value of P2.5 and P0 must be more than 20. If not, the alarm will be off. In this case, please re-calibrate the pressure signals according to the calibration sequence: P5→P2.5→P0, or referring to the Help Interface of Calibration Warning.

- Pressure Calibration finished.

9. Help Interface



- (1) On the Login Interface, click [Help] button to enter the Help Interface. User can check problems that might happen during self-diagnosis and digestion, or check operation manual Chapter 8.
- (2) Click the [Previous] and [Next] button to view the whole listing of FAQ.
- (3) Click the [Esc] button and return the Login Interface.

Discussion on Microwave Digestion and Methods

A. Discussion on Microwave Digestion

1. As a new sample pretreatment technology, microwave digestion has attracted more and more people's attention. The advantages of microwave digestion technology to digest samples include shorter digestion time, more simple operation, less use of solvent and less environment pollution, etc.
2. Digesting samples by microwave equipment:
 - (1) **Key factors:** the digestion temperature, digestion time and digestion reagent. Proper digestion reagent and proper digestion temperature ensure that the samples can react with chemicals, thus being digested. The Digestion time ensure that the samples can be digested completely.
 - (2) **Digestion Principle:** digest with small but enough power with multiple steps of temperature and pressure increasing.
 - (3) The ideal microwave digestion program is that completely digest the most important component in the sample matrix as far as possible at the minimum required temperature. It is necessary to know the components of the sample matrix for determining the most effective temperature of the digestion. Understanding the digestion characteristics and the interaction between the sample components and the different reagents can make it easier for the analyst to control the digestion process of the sample. For different organic or inorganic samples, the changes in the intensity and physical volume of the chemical reaction at each temperature point are different during microwave digesting.
3. Organic samples of carbohydrates, proteins and lipids are the three matrix components of the animal and plant samples. In the microwave digestion of the animal and plant samples, high pressure can be produced by the gas by-products such as CO₂ and NO₂. In general, the critical temperature of the organic sample is digesting by nitric acid(HNO₃) as follows:
 - (1) Carbohydrates, such as starch, >140°C;
 - (2) Proteins, >145-150°C;
 - (3) Sugars, >150°C;
 - (4) Lipids and fats,> 160-165°C;
 - (5) Heavy oil and petroleum bitumen, >180-185°C.
4. The digestion of inorganic samples needs to be reacted with the corrosive mixed acid at high temperature to achieve complete digestion. The pressure of the inorganic sample is lower than that of organic samples, most inorganic samples without CO₃²⁻ can be digested completely around 185°C, and can digest under the program only one step.
5. For microwave digestion, the selection of reagents is very important. The

digestion of organic samples needs to maintain the acid condition of strong oxidizing, mixed digestion reagents are commonly used, such as $\text{HNO}_3/\text{H}_2\text{O}_2$ 、 $\text{HNO}_3/\text{H}_2\text{O}_2/\text{HF}$ 、 HNO_3/HCl , etc.

6. HNO_3 , H_2SO_4 and HCl are commonly used in wet digestion method. This is due to sulfate and chloride of some metals are insoluble or slightly soluble, and boiling point for H_2SO_4 is high which will easily cause damage to digestion vessel because of too high temperature, so microwave digestion mostly use HNO_3 . Advantage for HNO_3 is that all nitrates are soluble to water and strong oxidants which makes organics easy to digest. Moreover, boiling point for HNO_3 (120°C) is low which is in safe temperature range ($\leq 250^\circ\text{C}$). HF makes soil and sediments easy to digest and has little effect on bio-samples. H_2O_2 , as weak acid oxidizer which resolve into high energy state reactive oxygen can degrade some organics like humic acid. If user use H_2O_2 and HNO_3 together can improve oxidizing ability and damage organics which results in easy digestion and little effect on reaction matrix.
7. It is common to use $\text{HNO}_3/\text{H}_2\text{O}_2$ for food and cosmetics samples; HNO_3/HF , $\text{HNO}_3/\text{HCl}/\text{HF}$ for environment samples; $\text{HNO}_3/\text{H}_2\text{O}_2$, HNO_3/HF , HNO_3/HCl for biological and pharmaceutical samples; HNO_3/HF , HNO_3/HCl , $\text{HNO}_3/\text{HCl}/\text{HF}$ for geological mineral samples or industrial samples.

B. Microwave Digestion Methods

Some of the following samples need to be predigested before microwave digestion. For example, paint.

NOTE 1: The methods in the table are only apply to microwave digestion under both control of temperature and pressure; please don't use them to the microwave equipment which only has pressure control system.

NOTE 2: The table only lists the temperature, pressure and hold-time of the last step of the program method. Please complete the previous procedure steps before using the corresponding methods.

| Sample name | | Sample Quantity | Reagents (mL) | P _H (Bar) | T _H (°C) | Holding Time(s) |
|-----------------------------|-------------------|-----------------|--|----------------------|---------------------|-----------------|
| Food Samples | Flour | 0.50g | 4HNO ₃ , 1H ₂ O ₂ | 30 | 185 | 600 |
| | Orange juice | 5.00ml | 6HNO ₃ , 1H ₂ O ₂ | 30 | 185 | 600 |
| | Edible oil | 0.50ml | 8HNO ₃ , 2H ₂ O ₂ | 40 | 190 | 900 |
| | Chocolates | 0.30g | 5HNO ₃ , 1H ₂ O ₂ | 30 | 195 | 900 |
| Cosmetics Samples | Toner (ml) | 3.00ml | 4HNO ₃ , 2H ₂ O ₂ | 30 | 180 | 600 |
| | Face ream | 0.50g | 3HNO ₃ , 2H ₂ O ₂ , 0.5HF | 30 | 185 | 600 |
| | Soap | 0.20g | 5HNO ₃ , 2HF | 35 | 190 | 900 |
| Environmental Samples | Soil 1 | 0.20g | 6HNO ₃ , 2HCl, 2HF | 40 | 195 | 900 |
| | Soil2 | 0.20g | 6HNO ₃ , 2HCl | 40 | 195 | 900 |
| | Domestic Sewage | 5.00ml | 6HNO ₃ , 2H ₂ O ₂ , 1HF | 40 | 190 | 600 |
| Biopharmaceut-icals Samples | Hair | 0.20g | 1HNO ₃ , 4H ₂ O ₂ | 25 | 185 | 600 |
| | Animal liver | 0.30g | 10HNO ₃ | 30 | 190 | 600 |
| | Capsule | 0.50g | 5HNO ₃ , 1HF | 40 | 190 | 900 |
| | Chinese herb | 0.30g | 5HNO ₃ , 1H ₂ O ₂ | 30 | 190 | 900 |
| Geological Mineral | Rutile | 0.10g | 2HNO ₃ , 6HF | 45 | 235 | 1200 |
| | Alumina | 0.10g | 4H ₂ SO ₄ , 4HCl, 2HF | 35 | 195 | 1200 |
| | Tantalum ore | 0.10g | 1HNO ₃ , 5HF | 40 | 210 | 1200 |
| | Boron Carbide | 0.10g | 5HNO ₃ , 5HF | 45 | 240 | 1800 |
| Industrial Materials | Crude Oil | 0.50g | 8HNO ₃ , 1H ₂ O ₂ | 50 | 210 | 600 |
| | Cement | 0.20g | 8HNO ₃ | 30 | 195 | 900 |
| | Textile Dyestuffs | 0.30g | 6HNO ₃ , 1H ₂ O ₂ | 40 | 195 | 1200 |
| | Paint | 0.10g | 6HNO ₃ , 3H ₂ SO ₄ | 35 | 205 | 1200 |

Note: Unit for solid sample is gram; Unit for liquid sample is ml.

Questions and Answers (FAQ)

FAQ Table 1:

| Prompt | Analysis |
|---|--|
| Digestion vessel is not detected | <ol style="list-style-type: none"> 1. Check whether the digestion vessel has been loaded; check whether the digestion vessel is assembled correctly. 2. Refresh the pressure curve at the Calibration Interface. Check whether the vessel deviate the correct position, adjust the angle value and correct the vessel position. |
| No Digestion vessel | |
| Angle Deflection | Please do instrument calibration (full calibration procedure, first angle Adjust, then pressure calibration. |
| Pressure Channel Warning | <p style="text-align: center;"><u>Reason: the current pressure values are not within the normal range.</u></p> <ol style="list-style-type: none"> 1. The digestion vessel is not loaded. 2. Digestion vessel is abnormally placed (vessel bottom is not placed the Sample Rotor fully, not take out the PCR or the vessel position circle jumps, etc.) 3. The assembly of the digestion vessel is wrong. 4. Signal changes or drifts, please calibrate the pressure signal. 5. Failure of current channel board. |
| Zero warning | |
| Fan 1 or Fan 2 Warning | Fan 1 or Fan 2 has problem, contact distributor or factory for maintenance. |
| Motor System Warning | <p style="text-align: center;"><u>The current motor angle signal value is wrong.</u></p> <ol style="list-style-type: none"> 1. Check whether the motor is blocked. 2. The wiring of the motor or driver is loose. |
| Motor Warning | |
| Temperature System Warning | <ol style="list-style-type: none"> 1. The parameters of temperature sensor are abnormal. 2. The wiring of the temperature sensor is loose. 3. The main control board has fault. |
| Door Unclosed | Check the color of the door status indicator on the Run Interface. Color green means the door is closed, and the gray means the door is unclosed. If the color is gray or red, please open the door and close it again. |
| P5 Warning | <p style="text-align: center;"><u>The calibration values of P5 are beyond the normal range: 200±20.</u></p> <ol style="list-style-type: none"> 1. The assembly of the digestion vessel is wrong, or the digestion vessel is abnormally placed (vessel bottom is not placed the Sample Rotor fully, not removed the PCR or the vessel position circle jumps, etc.). 2. The signal values of P5 are wrong, please re-calibrate the pressure signal. 3. The signal of signal board is weaker, please re-calibrate the pressure signal. |

FAQ Table 2:

| Prompt | Analysis |
|----------------------------|--|
| Calibration Warning | <p><u>The values of parameters P5, P2.5 or P0 are not in the correct range.</u></p> <ol style="list-style-type: none">1. Errors occurred during the calibration, such as alarming during the process of calibration, etc.2. The calibrated values of parameters P5, P2.5 or P0 are exceptionally close to the critical state. Please re-calibrate the instrument. |
| Parameters Warning | <p>The parameters of the Program Method interface are wrong, so that the instrument is unable to work normally. Please check if the Heat-Time and the Hold-Time of step1 are 0 at the same time in the current program method. If yes, please correct.</p> |
| Sound Warning | <p><u>The system has detected a larger sound.</u></p> <ol style="list-style-type: none">1. The digestion vessel has exploded; it is the normal protection of the system.2. External interference or misdetection. Please click [START] button to make the instrument continue to work after confirm that the digestion vessel and the instrument are in normal condition. |

Instrument Quality Guarantee

A. Warranty Range

1. Our company will be responsible for the instrument failure caused by manufacturing defects.
2. When repair, we may use the spare parts to replace the original parts, also may use the same component to replace the damaged parts.
3. Abandoned instruments or resold instruments are not in warranty range.

B. Warranty Period

Since the sales date:

1. One year: Main Engine (including the internal parts).
2. Instrument Accessories and consumables are not in warranty range.

C. Conditions with No Warranty

The following conditions even during the warranty period are not in warranty range.

1. Any damage or problem caused by operation environment that is not allowed.
2. Any damage or problem caused by operations that do not follow the voltage and power using instructions.
3. Any damage or problem caused by corrosive gases that have nothing to do with the instrument, which affect the spare parts, circuit elements and optical parts.
4. Any damage or problem caused by improper operation or maintenance.
5. Any damage or problem caused by improper repair of untrained technical persons.
6. Abandoned instrument or second-handed instrument.
7. Any damage or problem caused by disassembly without permission of our company.
8. Consumables and spare parts that not in warranty period.
9. Problems caused by spare parts which not in the Warranty Range Warranty Bill
Warranty Bill.
10. Any problem or damage caused by force majeure, such as earthquake, typhoon, flood, fire, lightning, riots, unrest, crime, terrorism, war, radioactive pollution, the pollution of harmful substances etc.
11. Problem caused by thunder or unexpected power outage.

D. The Warranty Limits

1. That out of the warranty are not in warranty range.
2. Commercial implied warranties as well as the appropriateness of a particular purpose cannot be warranted. The direct or indirect damage resulting from the express or implied guarantee cannot be compensated.

Instrument Maintenance

- A. Users should pay great attention to routine maintenance of the instrument which not only keeps it in stable and high efficient conditions, but also extend its service life.
- B. Please do maintenance regularly depending on instrument condition. It is suggested to do once a week. **Note:** Please calibrate instrument If user restart instrument after long time.

C. Routine Maintenance

1. For the first use of the Sample Digestion Vessels:

Disassemble the Sample Digestion Vessels and wash the sealing covers and inner vessel bodies with flowing water first, and then soak them in **25%** of the dilute nitric acid (HNO_3) for more than 12 hours, clean them three times with distilled water or deionized water, then dry them naturally or at 50°C in the special drying oven.

2. After daily use of digestion vessels:

- (1) **Sample Digestion Vessels:** Disassemble the Sample Digestion Vessels and wash the sealing covers and inner vessel bodies with flowing water first, and then soak them in **20%-25%** of the dilute nitric acid (HNO_3). After more than 12 hours, clean them three times with distilled water or deionized water, then dry them naturally or at 50°C in the special drying oven.
- (2) **Vessel Cover assembly and the Outer Protective Vessel:** Disassemble the Vessel Cover assembly, clean the cover, signal board, lock nut, guiding rod and the outer vessel body with flowing tap water first, and then wipe them with clean cloth or dry them naturally. **Note: The pressure component in the vessel cover assembly is a multi-structure component, it can not be directly washed or immersed in running water.**
- (3) **The pressure component:**
 - After daily use, **please clean it with a clean wet cloth.**
 - When do maintenance regularly (about 1 week) or when there is a leak removal situation, **please disassemble it by trained operators and clean it with running water.**
- (4) **Repair Tool:** Please clean it according to the way of daily cleaning the sample digestion vessel (refer to the 10.2.2(1)).
- (5) **Signal Board:** Please clean it in time, and keep the signal monitoring surface dry and clean.
- (6) Make sure the digestion vessel (sample digestion vessel, outer protective vessel), sample rotor, auxiliary platform and inner surface of chamber are clean and dry.

(7) Please use alcohol cotton swab to clean pressure and temperature monitoring sensors slightly after each use.

D. Not using in long time

1. Pull out the power cord.
2. Do not place the instrument in the place with vibrating or temperature higher than 40°C or lower than -20°C or humidity over 85%.
3. Do not place the instrument in the place with acidic and alkaline gas.
4. Do not place the instrument in the place with much dust.
5. Do not place the instrument in a place under direct sunshine.