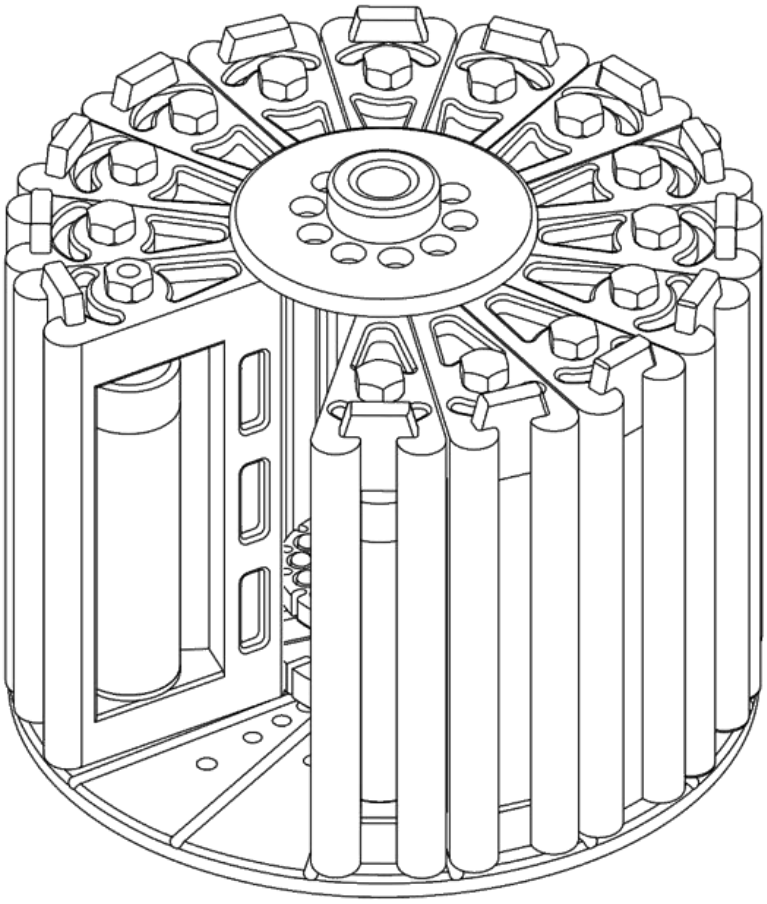
PreeKem

Operation Manual



KJ-160

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.

Table of contents

KJ-160 Vessels operation guide

1 Safety instruction

ATTENTION

1.1 All vessel and frame components must be dry and free of particulate matter. Drops of liquid or particles will absorb microwave energy, causing localized heating which may char and damage vessel components, leading to possible vessel failure.

ATTENTION

1.2 DO NOT heat concentrated alkali or salt solutions inside the vessel. PreeKem suggests that the concentration of alkali or salt be less than 10%. Otherwise, heating such solutions with microwave will result in the formation of crystal deposits that sticks on the vessel wall. And they will absorb microwave energy and cause localized heating, leading to possible vessel components failure.

DANGER

1.3 The use of perchloric acid is prohibited to avoid explosion. DO NOT solely heat high boiling point acids (concentrated sulfuric or phosphoric acid) inside the vessels. The temperature may beyond vessel melting point and damage vessels.

DANGER

1.4 Need follow the related regulation to weigh the samples. In terms of organic samples, large amount of gases will be generated during the digestion process. Excessive samples may cause reaction so fierce that the rate of pressure and temperature rise will exceed the responding rate of the pressure and temperature control system of the machine and there will be a risk of explosion when the internal vessel pressure exceeds the limitation. The weight should start from 0.1g for unknown samples, and increase the weight according to the actual pressure and the discrepancy from instrument withstand pressure.

ATTENTION

1.5 In terms of organic samples: they should always be predigested in unsealed inner vessel. It is up to the sample itself.

(1) Place the sample in the fume hood for several minutes or hours, and gently shake it, then install the vessel in instrument after small amount of bubbles or gas is released.

(2) Put it on a specialized hot block (temperature < 150°C) for a while, and then install the vessel in instrument after some gas and heat is released. After that, set digestion procedure with low temperature (100-120°C) and pressure (0.2-0.5 MPa) first, then middle temperature and pressure, and high temperature and pressure in the end. This method is safe and good for digestion.

DANGER

1.6 Control vessel with temperature and pressure control should be mounted in the instrument when the digestion is conducted in sealed vessels, and the control vessel must not be empty.

DANGER

1.7 In a single digestion, the type, weigh, size and original temperature of sample and solvent in each vessel (including the control vessel) should be same.

ATTENTION

1.8 The safety diaphragm should be replaced with a new one before each experiment.

ATTENTION

1.9 Never install more than 1 safety diaphragm (with specific thinness) in the sealed lid of every standard vessel.

DANGER

1.10 Using specialized torque wrench to tighten the top plug. Do not use the common wrench to over tighten the top plug. Otherwise, the excessive pre-tighten force will result in frame broken or crack.

DANGER

1.11 Standard vessels and control vessel should be used at the same time. Using standard vessels only is prohibited. The control vessel must not be empty.

ATTENTION

1.12 The inside of temperature probe tube on control vessel should keep dry and clean. Otherwise, localized heating will lead to temperature probe and sleeve damage.

DANGER

1.13 Make sure it is tighten and no leak between the connector of air-guide tube and control vessel. Otherwise, the leakage of acid solution (or gas) might lead to sparking and components burning. The instrument must be shut down if it happens.

ATTENTION

1.14 Before starting heating, check the rotation of turntable to make sure it can rotate 360° back and forth. Otherwise, the localized overheat and overpressure might lead to explosion. Make sure the temperature probe can rotate freely after being inserted into the vessel without blockage; otherwise the connection wire may be broken after twisting.

DANGER

1.15 For all the vessels, only after temperature drops below 80°C, the deflation screw can be loosen slowly in the fume hood. Only after finishing releasing the air, the top plug can be removed to take out the vessel. Otherwise, the operation of a vessel with pressure will cause the acid to be splashed onto the operator and result in injury.

DANGER

1.16 Before opening the vessel, must loosen the deflation screw in the fume hood first. Only after finishing releasing the air, the top plug can be removed to take out the vessel. Otherwise, the operation of a vessel with pressure will cause the acid to be splashed onto the operator and result in injury.

ATTENTION

1.17 Vessels are consumables. Repeated expansion and contraction by heating and cooling, along with acid corrosion, will lead to aging and crack. If cracks are found, replace it as soon as possible to avoid explosion, especially for sleeve and frame.

ATTENTION

1.18 The material of vessel is modified PTFE (TFM), whose melting point at normal pressure is 300°C. If heat it by heater, the heater must has overheating insurance.

ATTENTION

1.19 No matter which sample is digested, the digestion procedure must be carried on as soon as possible, as long as the sample and solvent have been in inner vessels and have been loaded in outer vessel. Open the vessels in time after digestion finished. The digestion solvent is not allowed to stay in the vessels for too long to avoid sleeve and frame damage caused by acid leak.

ATTENTION

1.20 Suggest the working temperature to be no more than 240°C for extending the lifetime of the vessels.

DANGER

1.21 Digestion samples that are not suitable for airtight microwave digestion is prohibited; please refer to “Forbidden samples for microwave digestion”.

2 Rotor introduction

2-1 Overview

KJ-160 framework Digestion vessel is a airtight reaction vessel, designed to handle various types of sample reactions, including synthesis, extraction and digestion. It includes inner digestion vessel with corrosion resistance capability and an enhanced frame. It can work with the different assembly of turntable, where you can install a maximum of 15 KJ-160 framework digestion vessels for reaction at a time.

KJ-160 framework digestion vessels include two different types of digestion vessels; they are control digestion vessel and standard digestion vessel respectively. The former one has a special structure, and it can be connected with temperature sensor components and the pressure sensors, so as to real-time measure the temperature and pressure values inside the vessel, and there is only one control vessel among the entire sets of digestion vessels. The latter one is a standard digestion vessel, which be loaded into oven chamber with control vessel at the same time to complete the reaction. The temperature and pressure inside the standard vessel can refer to the values reading from control vessel. Their operating temperature range is 0-240°C and operating pressure range is 0-5 MPa.

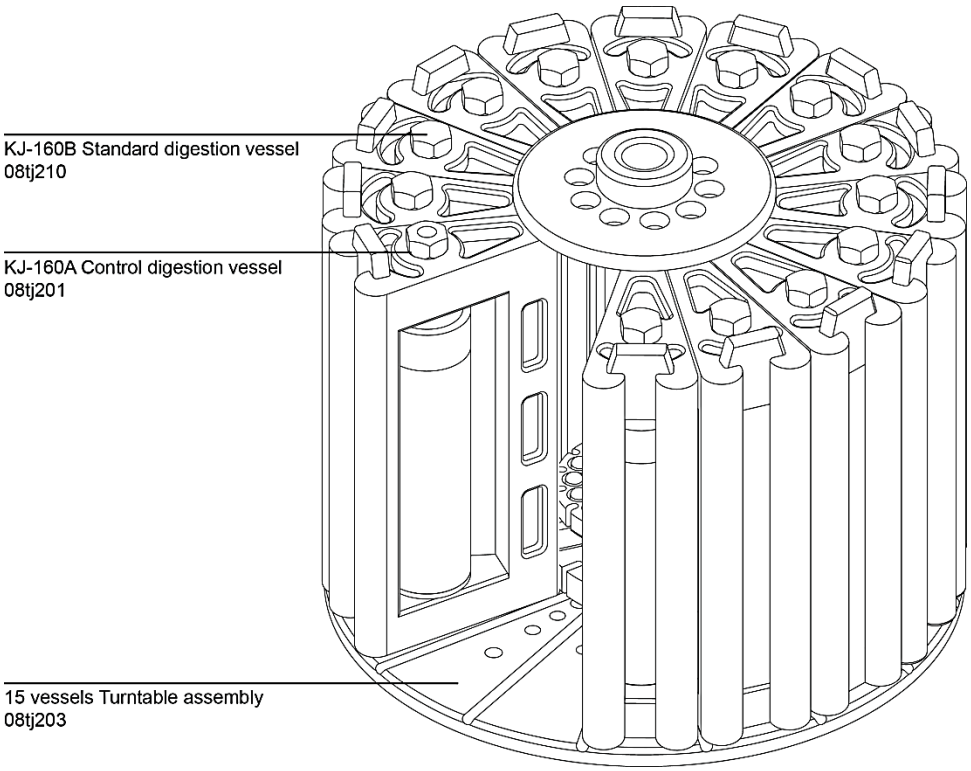


Figure 2-1: KJ-160 Vessels

2-2 Technical Performance

Inner vessel material: TFM

Protection sleeve material: PEEK

Digestion vessel volume: 100 mL

Maximum operating pressure: 5 MPa

Maximum operating temperature: 240°C

3 Rotor operation

3-1 Turntable installation

Related component:

- Main instrument
- Half shaft coupling (09ca027)
- 15 vessels turntable assembly (08tj203)

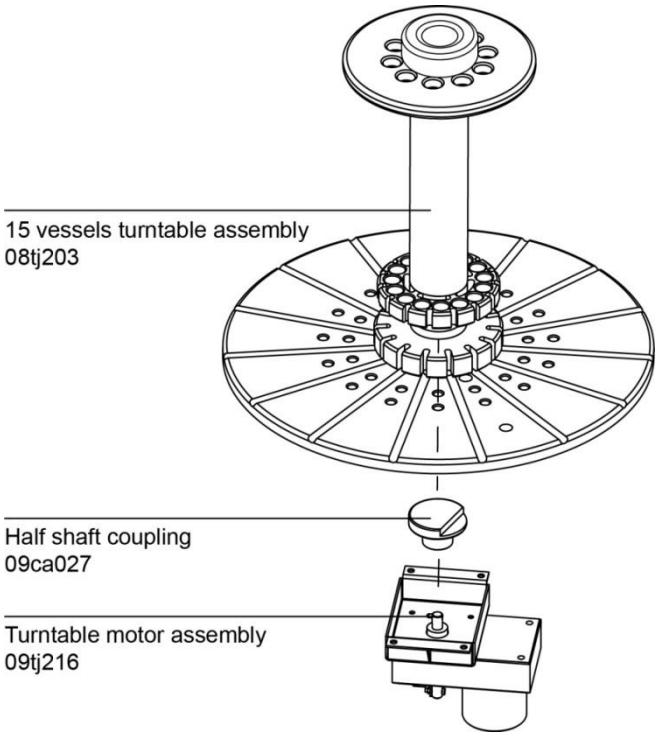


Figure 3-1: Turntable installation scheme

Procedure:

1. Before installing the turntable, please open the oven door, and insert the half shaft coupling into the center hole at the bottom of the oven chamber.

2. Load the entire turntable into the oven chamber. During loading, align the connection shaft with the notch on the center of the turntable.

ATTENTION **After installation, the turntable can be leaved in the oven chamber. It is not required to install it every time of operation.**

3-2 Installation of KJ-160A control digestion vessel

Related component:

- Control top cushion (09ca014)
- Control seal lid (17tj403)
- Control top plug (17ca107)
- Sample cup (17ca103)

- Sleeve inner pad (17ca108)
- Frame (17ca106)
- Sleeve (17ca104)
- Bottom cushion (17ca105)
- Torque wrench (00fb704)
- Location pin assembly (01tj307)
- Opening expander (08ca104)

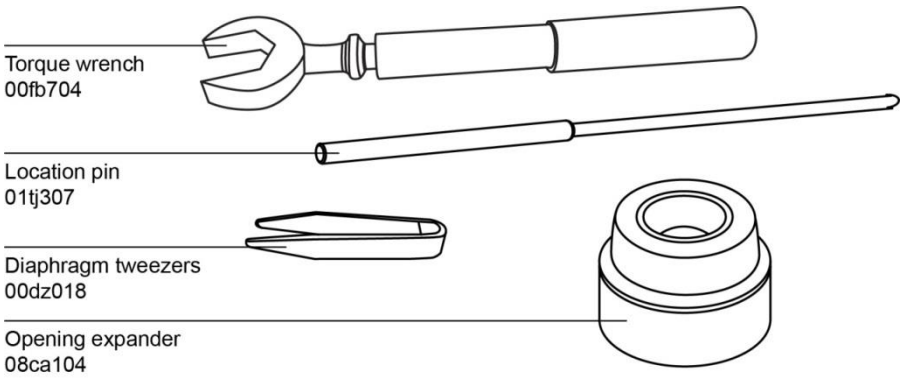


Figure 3-2: Tools scheme

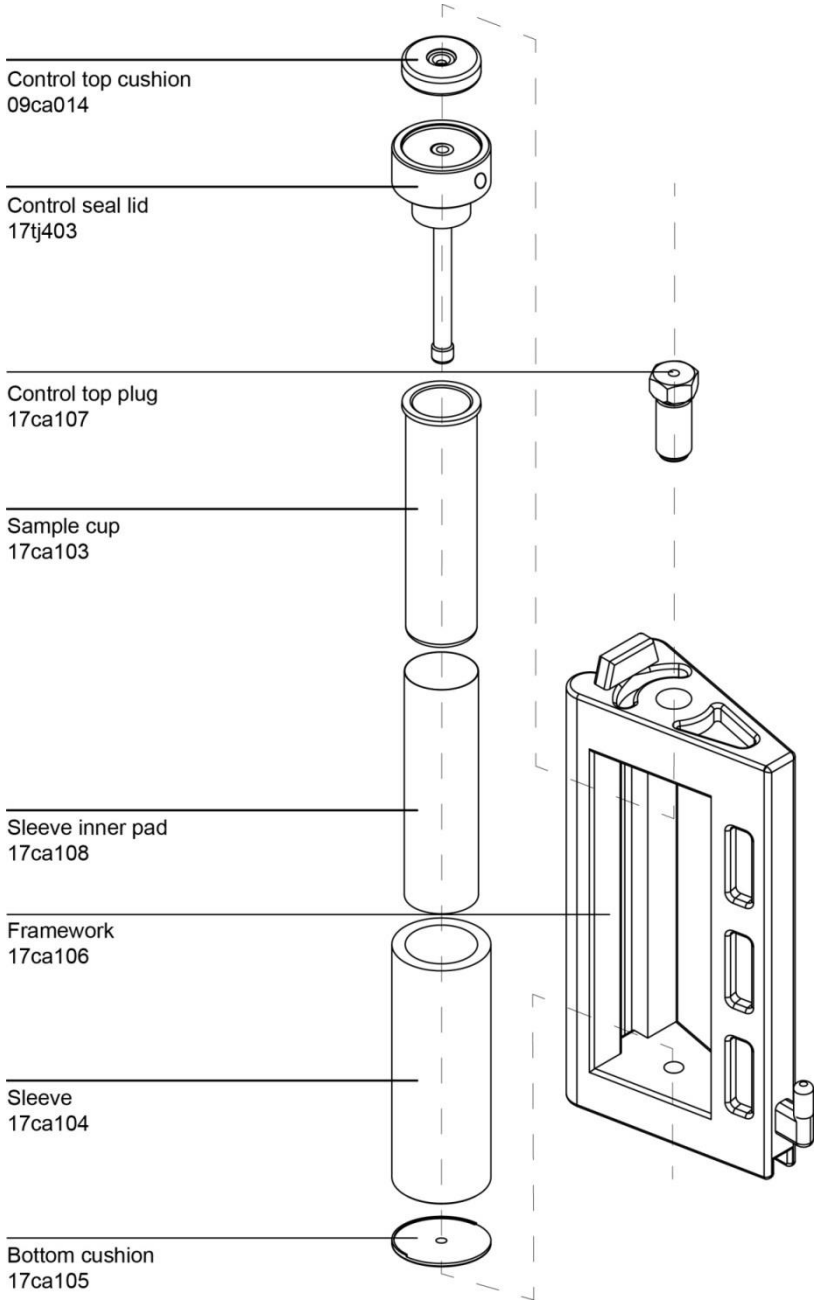


Figure 3-3: KJ-160A control digestion vessel

Procedure:

1. Weigh the sample with scale, and put it into sample cup.

ATTENTION **The sample must be placed at the bottom of the sample cup, to avoid sticking onto the wall of sample cup.**

DANGER **The weight of the unknown sample must be less than 0.1 g to avoid the risk of vessel explosion caused by too violent reaction.**

2. Move the sample cup loaded with sample to fume hood. Weigh and put proper amount of acid solvent into the sample cup, to immerse the sample completely. Shake the sample cup gently or stir it, to disperse the sample evenly in solvent. If

needed, put the sample cup that is loaded with acid solvent and sample onto the electrical heater for pretreating for a certain time (15-30min).

ATTENTION **Observe if violent a reaction occurs after sample is dispersed in the solvent. If the reaction is too violent, please DO NOT close the seal lid until the reaction almost completes.**

ATTENTION **The amount of solvent added should be in the range of 3-15 mL. The amount of solvent added for other microwave chemical reaction should be in the range of 3-25 mL.**

3. Insert the opening expander into the skirt of the seal lid to the bottom and then expand the opening of the skirt of seal lid by rotating the expander for 2-3 circles. The seal quality of the digestion vessel can be improved after expanding the opening, and so as to effectively avoid the loss of the sample or elements during the process of the reaction.

ATTENTION **DO NOT tilt the axis of opening expander during expanding in order not to crack or deform the skirt due to uneven expanding.**

ATTENTION **Cover the seal lid onto the sample cup as soon as the expanding is done, otherwise the skirt will shrink and recover from the expanding effect. Expand the skirt again if so.**

ATTENTION **There is a well on the control seal lid, which is used for the installation of temperature sensor. Pay attention not to touch the well wall when inserting and removing the opening expander to avoid the possible damage.**

4. Close the seal lid (the opening of sample cup need be placed into the sealed slot completely). Then place the sample cup into the sleeve from the top to bottom. Place the top cushion in the round slot at the top of the seal lid, where the indentation faces up.

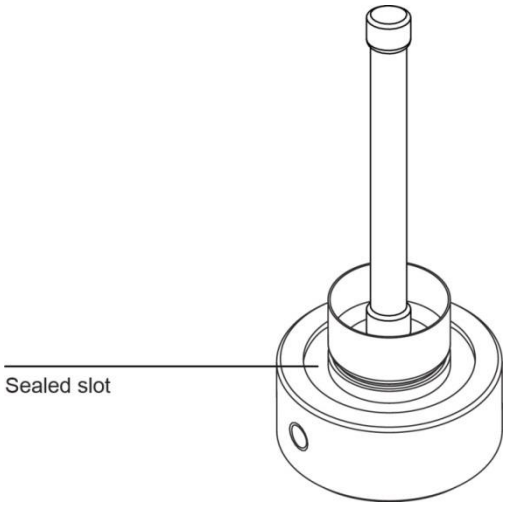


Figure 3-4: Sealed slot scheme

5. Put the bottom cushion at the bottom of the framework, aligning its opening with the opening of the framework. Push the digestion vessel into the framework from the opening side, and touch the raised area of the bottom pad to locate its position.

6. Insert the location pin into the thermal probe jack to check if the channel is free to ensure that the installation of components is in accurate position. Use the torque wrench to tighten the top plug. When the torque reaches the pre-set value, the mechanism will be activated. There will be an angle formed between the handle and the end of the wrench and you can hear a click, which indicates that the top plug is tightened and the location pin can be removed.

ATTENTION

If there is a block during inserting the location pin, please check if the digestion vessel is centered on the bottom cushion. If there is misalignment, please loosen the top plug, readjust the position and insert the location pin again.

DANGER

ONLY torque wrench is allowed to tighten the top plug. Keep in mind! DO NOT use the common wrench to tighten the top plug, to avoid the excessive force and damage the framework.

3-3 Installation of KJ-160B standard digestion vessel

Related components:

- Common top cushion (08ca107)
- Standard seal lid (08tj402)
- Standard top plug (17ca111)
- Sample cup (17ca103)
- Sleeve inner pad (17ca108)
- Framework (17ca106)
- Sleeve (17ca104)
- Bottom cushion (17ca105)
- Deflation screw (09ca052)
- Safety diaphragm (09ca050)
- Torque wrench (00fb704)
- Diaphragm tweezers (00dz018)
- Opening expander (08ca104)

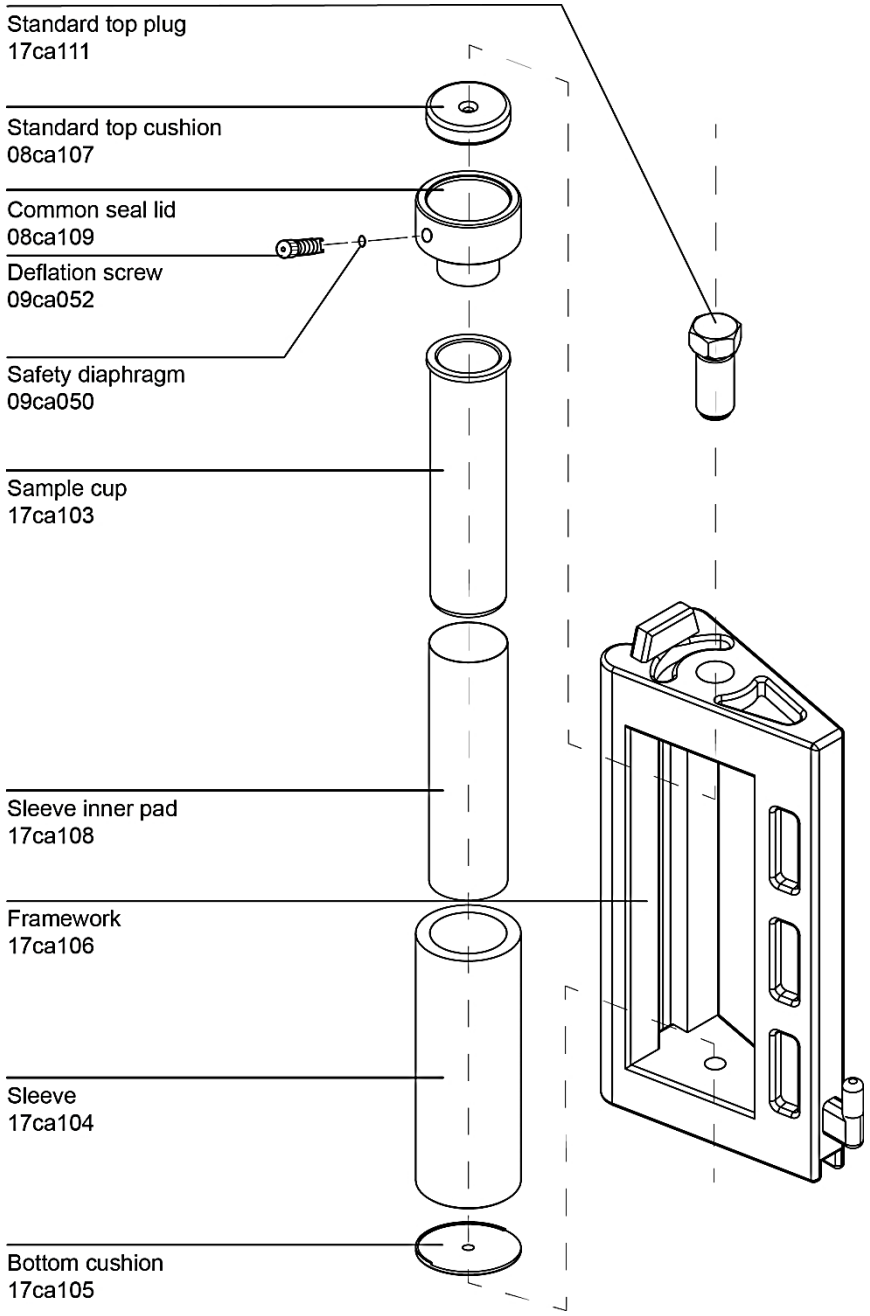


Figure 3-5: KJ-160B standard digestion vessel

Procedure:

1. Weigh the sample with scale, and put it into sample cup.

DANGER **The amount of sample added in the standard vessel should be the same as the amount added in the control vessel.**

ATTENTION **The sample must be placed at the bottom of the sample cup, to avoid sticking onto the wall of sample cup.**

DANGER **The weight of the unknown sample must be less than 0.1 g to avoid the risk of vessel explosion caused by too violent reaction.**

2. Move the sample cup loaded with sample to fume hood. Weigh and put proper amount of acid solvent into the sample cup, to immerse the sample completely. Shake the sample cup gently or stir it, to disperse the sample in solvent evenly. If needed, put the sample cup that is loaded with acid and sample onto the electrical heater for pretreating for a certain period (15-30 min).

DANGER

The amount of solvent added in the standard vessel should be same as the amount added in the control vessel.

ATTENTION

Observe if there is a violent reaction occurs after sample is dispersed in the solvent. If the reaction is too violent, please DO NOT close the seal lid until the reaction almost completes.

ATTENTION

The amount of solvent added should be in the range of 3-15 mL. The amount of solvent added for other microwave chemical reaction should be in the range of 3-25 mL.

3. Remove the deflation screw from the standard seal lid. Check the deflation hole to ensure the passage is free. Use the diaphragm tweezers to put one safety diaphragm into the socket (recess) of the deflation screw (Only one specialized safety diaphragm is allowed). Make sure that the safety diaphragm is placed flat in the socket and tail node toward the gap. Then twist the deflation screw into the threaded hole of the seal lid. During twisting the screw, rotate it to the end until the screw is tightened to compress the safety diaphragm to prevent gas leakage. However, do not overtighten the screw to avoid thread stripping. The seal of the safety diaphragm is very important since the leakage caused by diaphragm seal failure will lead to the inconsistent pressure and temperature between standard vessels and control vessel, which will result in experiment failure.

ATTENTION

In the process of digestion, when the pressure is less than 3 MPa, if there is gas leak or pressure dropping in proximity to the safety diaphragm, please check the installation of safety diaphragm to ensure that the tail node toward the gap of deflation screw.

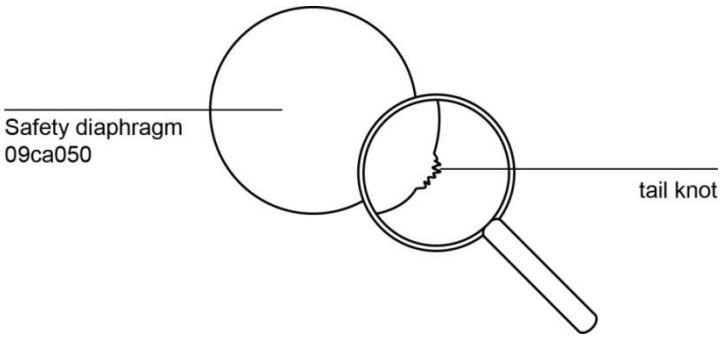


Figure 3-6: Safety diaphragm tail knot scheme

4. Insert the opening expander into the skirt of the seal lid to the bottom and then expand the opening of the skirt of seal lid by rotating the expander for 2-3 circles.

The seal quality of the digestion vessel can be improved after expanding the opening, and so as to effectively avoid the loss of the sample or elements during the process of the reaction.

ATTENTION

DO NOT tilt the axis of opening expander during expanding in order not to crack or deform the skirt due to uneven expanding.

ATTENTION

Cover the seal lid onto the sample cup as soon as the expanding is done, otherwise the skirt will shrink and recover from the expanding effect. Expand the skirt again if so.

5. Close the seal lid (the opening of sample cup need be placed into the sealed slot completely). Then place the sample cup into the sleeve from the top to bottom. Place the top cushion into the round slot at the top of the seal lid, where the indentation faces up.

6. Put the bottom cushion at the bottom of the framework, aligning its opening with the opening of the framework. Push the digestion vessel into the framework from the opening side, and touch the raised area of the bottom pad to locate its position.

7. Use the torque wrench to tighten the top plug. When the torque reaches the pre-set value, the mechanism will be activated. There will be an angle formed between the handle and the end of the wrench and you can hear a click, which indicates that the top plug is tightened and the location pin can be removed.

ATTENTION

ONLY torque wrench is allowed to tighten the top plug. Keep in mind! DO NOT use the common wrench to tighten the top plug, to avoid the excessive force and damage the framework.

3-4 Load digestion vessel into oven chamber

Related components:

- KJ-160A Control digestion vessel (08tj201)
- KJ-160B Standard digestion vessel (08tj210)
- Turntable assembly (08tj203)
- Temperature sensor assembly (17tj104)
- Air-guide tube assembly (08tj204)

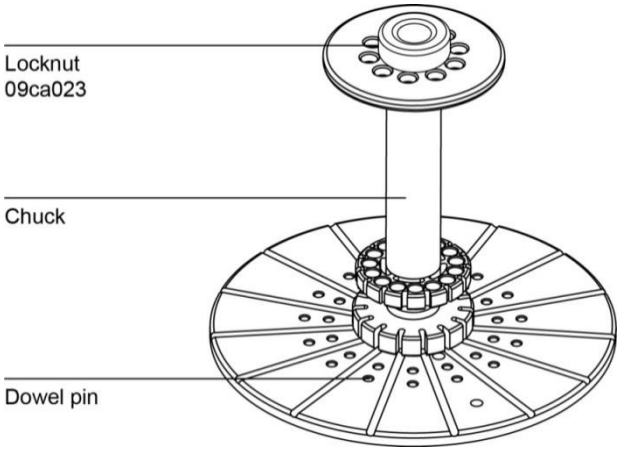


Figure 3-7: Scheme of part of components in turntable

Procedure:

1. Check the temperature sensor assembly and air-guide tube in the cavity, the temperature sensor assembly and air-guide tube compatible with KJ-160 vessels must be installed correctly.

ATTENTION **Please refer to the related chapter in Product Operation Manual for how to install KJ-160 temperature sensor assembly and air-guide tube.**

DANGER **The other types of temperature sensor assembly and air-guide tube are not compatible with the KJ-160 vessels, which will cause instrument failure, and thus they must be replaced.**

2. Insert temperature probe into the center opening of assembled KJ-160A control digestion vessel, and place the KJ-160A control digestion vessel into the oven chamber.

3. Screw the fittings of air guile tube in the control seal lid hole of the sealed vessel. Make sure it is tight.

4. Place the assembled framework digestion vessels respectively on the proper position of the turntable in the oven chamber. The digestion vessels should be placed symmetrically and uniformly so as to enable the turntable rotating smoothly.

ATTENTION **Before shipping, the set of pressure guide system has been installed and adjusted in factory and passed the seal performance inspection. It is not necessary to uninstall it during operation. If it has to be installed or replaced, please consult PreeKem and perform the operation under the guidance of our service engineer.**

3-5 Uninstallation of digestion vessel

The framework type standard digestion vessel can be removed from oven after the temperature reading drops below 80°C and the pressure reading drops below 0.05

MPa. The operator must be particularly careful to open the vessel when the temperature drops below 80°C and the pressure in vessel is still higher than 0.05 MPa, to prevent pressured air injury.

DANGER **Before opening the vessel, please put on acid resistant glove, safeguard eyewear, respirator and coveralls. Be careful of the danger of corrosion and burns.**

DANGER **DO NOT disassemble digestion vessels when the temperature is above 80°C.**

DANGER **DO NOT open the safety door and remove digestion vessel from the oven chamber when the temperature is above 120°C.**

ATTENTION **After uninstallation, please store all the components into cabinet to prevent loss.**

Related components:

- KJ-160A control digestion vessel (08tj201)
- KJ-160B standard digestion vessel (08tj210)
- Turntable assembly (08tj203)
- Temperature sensor assembly (17tj104)
- Air-guide tube assembly (08tj204)

Procedure:

1. Open the safety door of the instrument, screw off the locknut at the top of the column, lift the column, and remove the standard digestion vessel from turntable and move it to fume hood.

2. Loosen the deflation screw of the standard vessel in the fume hood, to release the gas inside the vessel.

ATTENTION **Before loosening the deflation screw, please make sure the fume hood is on, so that the acid gas can be exhausted in time.**

DANGER **After loosening the deflation screw, the gas inside the vessel will be released from the opening of the deflation screw, watch out for corrosion and burns from acid gas.**

3. After temperature drops below 80°C, slowly loosen the pressure fittings that connects with the control digestion vessel and then remove the air-guide tube after the gas release is finished.

ATTENTION **If necessary, place the control digestion vessel to the back of oven chamber so as to exhaust the released acid gas quickly by the rear exhaust system.**

4. Remove the control digestion vessel from the turntable, unplug the temperature sensor assembly and place the digestion vessel to fume hood as soon as possible.

ATTENTION **Lower the control digestion vessel or tilt it by 45° after removing it from the turntable in order to leave enough space to unplug the temperature sensor.**

5. Loosen the top plug with torque wrench. Push out the digestion vessel from the framework, including the sample cup, sleeve inner pad, and the sleeve. During pushing, please press the vessel lid with hand gently in case the remaining gas push out the seal lid causing the seal lid drop and result in sample loss or seal lid deforming or damage.

ATTENTION **The deflation screw must be loosened in the fume hood in advance before opening the standard vessel. Loosen the top plug to remove the digestion vessel only after the gas inside the vessel has been released completely. This is to prevent the acid liquid in the pressurized vessel spraying on operator.**

ATTENTION **After the gas release is completed, loosen the top plug, disconnect the vessel with framework and a gap between the top cushion and the framework can be observed; if the gas release is not completed, the vessel lid will push the framework upwards. If this happens, please keep on loosening the deflation screw until the gas release completes.**

6. Remove the top cushion from the vessel lid, hold the sleeve with hand and push out the inner vessel. After pushing out part of inner vessel, hold the sample cup firmly with hand, and place the digestion inner vessel on the rack.

7. Hold the sample cup on the rack with hand and open the digestion vessel lid upwards. After opening the vessel move the digestion liquid to other container.

DANGER **DO NOT open the vessel lid without any support in case the vessel is unstable during opening, and result in spraying the acid liquid and injury.**

ATTENTION **DO NOT reuse these components of digestion vessel until they are cleaned and dried.**

3-6 Rinsing

After finishing, you must rinse the sample cup and vessel lid, specific method is as follows:

Procedure

1. Rinsing sample cup at least three times with deionized water.

2. Standard seal lid and sample cup should be soaked in 10%-15% nitrate cylinder through the whole night.

3. Taking one sample cup, pouring 10%-15% nitric acid to nearly full, then putting on seal lid and soaking. You should make the vessel cup straight all the time.

4. After soaking, rinsing sample cup at least three times with deionized water and ultrapure water.

Hot rinsing

The effect of hot rinsing is similar to the common rinsing, its operation is more complicated than common rinsing, but it has higher speed. So it can finish rinsing quickly.

Procedure:

1. Rinsing sample cup at least three times with deionized water.

2. Pouring 5mL nitric acid and 5mL ultrapure water into every sample cup.

3. Doing the experiment in the following table:

Step	Temperature (°C)	Time (min)	Pressure (atm)
1	120	2	10
2	180	10	20

4. After opening vessel, rinsing sample cup and seal lid at least three times with deionized water and ultrapure water.

Complete rinsing

When common rinsing can’t accomplish the requirement such as it is polluted seriously or it has a high require of blank, you must use complete rinsing method.

Procedure

1. Rinsing sample cup and seal lid at least three times with deionized water

2. Pouring 5mL hydrochloric acid and 5mL ultrapure water into every sample cup.

3. Doing the experiment in the following table:

Step	Temperature (°C)	Time (min)	Pressure (atm)
1	120	2	10
2	180	10	20

4. After opening vessel, rinsing sample cup and seal lid at least three times with deionized water.

5. Pouring 5mL nitric acid and 5mL ultrapure water into every sample cup.

6. Do the experiment as the procedure 3.

7. Rinsing sample cup and seal lid at least three times with ultrapure water.

ATTENTION

If user needs to do digestion, synthesis and extraction on one microwave system, we suggest use two rotors for the separate using. If you only have one rotor, you should clean the vessels strictly following the orders between two different using, and then put the vessels in the oven to dry them at 120°C for no less than 24 hours.

ATTENTION

The cleaning method for synthesis and extraction is as below: use ethanol or acetone to clean the inner vessels and wash with pure water two or three times before drying.

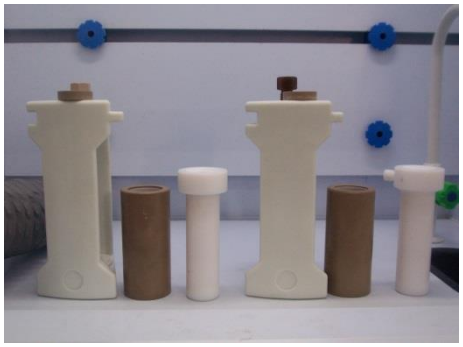
Appendix 1: Operating specification of KJ-160 vessels

Before installing the digestion vessel, please put on acid resistant glove, safeguard eyewear, respirator and coveralls.

1. Check framework, digestion vessel, make sure all components are clean, dry and without any tiny crack; The skirt on sample cup lid is round and intact; deflation screw has correct thread, no block in deflation passage. And control vessel (#0vessel) is different from standard vessel .there are round hole in the center of framework top plug, top cushion, seal lid.

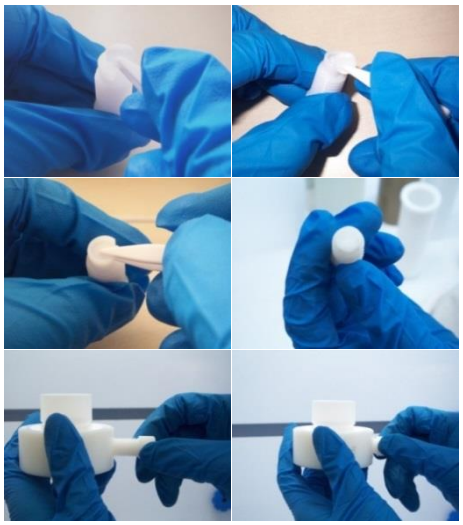
Note: you must check it strictly as the requirement and avoid over temperature and pressure while microwave heating, which results the destruction of digestion vessel and a failure experiment.

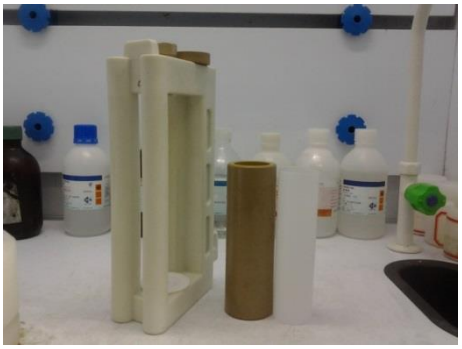
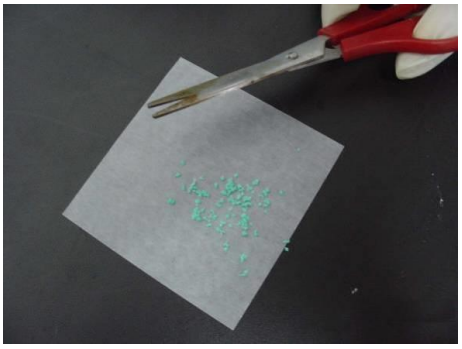
Components: (1) Framework (2) Sleeve (3) Sample cup (4) Sleeve inner pad (5) Control seal lid (6) Control top cushion (7) Control top plug (8) Common seal lid (9) Standard top cushion (10) Standard top plug

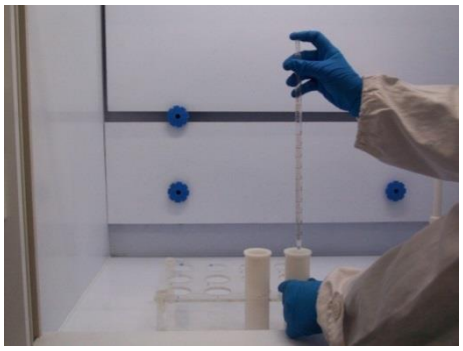
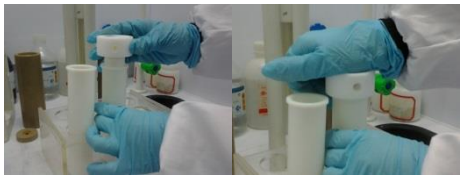


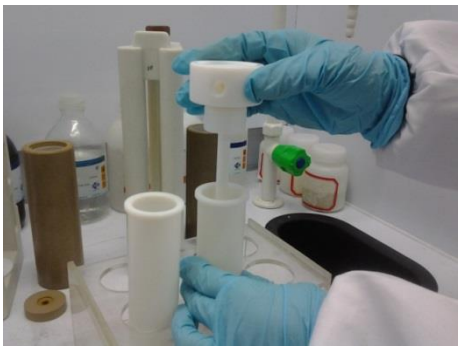
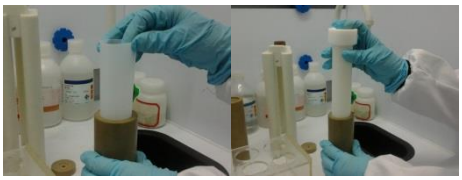
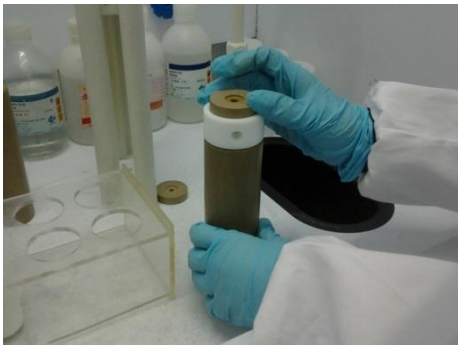
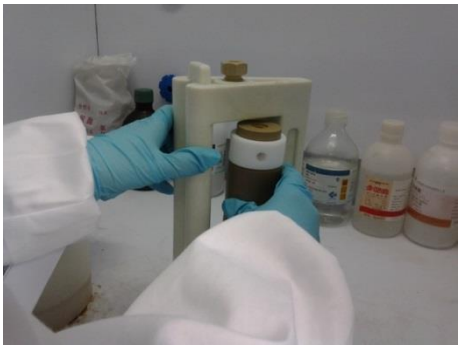
2. Remove the deflation screw from the seal lid of KJ-160B standard digestion vessel, remove the safety diaphragm inside of deflation screw with diaphragm tweezers, and check whether the diaphragm is flat of swells up. The diaphragm must be replaced if there is a swelling or hole. Place the deflation screw face up horizontally, confirm that the passage of deflation hole is free, and use the diaphragm tweezers to put one safety diaphragm into the socket (recess) of the deflation screw. Make sure that the safety diaphragm is placed flat in the socket and tail node toward the gap. Then twist the deflation screw into the threaded hole of the seal lid. During twisting the screw, rotate it to the end until the screw is tightened to compress the safety diaphragm to prevent gas leakage.

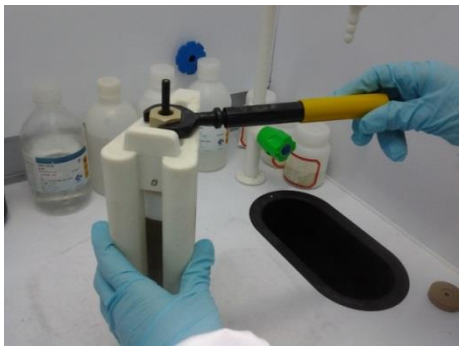
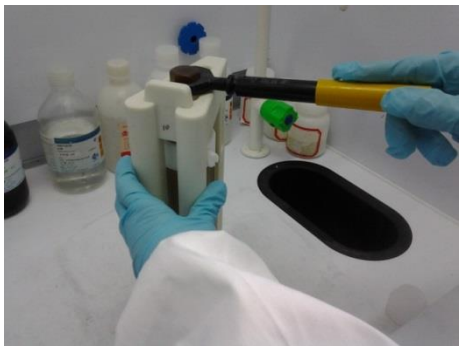
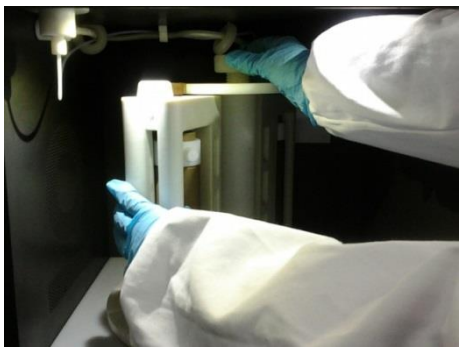
Note: The diaphragm of the standard vessel must be examined before heating the samples every time. Only one diaphragm is allowed to place. If put more than one diaphragm, the added force will disable its pressure releasing capability. If no diaphragm is installed, the reaction vessel won't be sealed, and therefore the reaction cannot be completed normally.

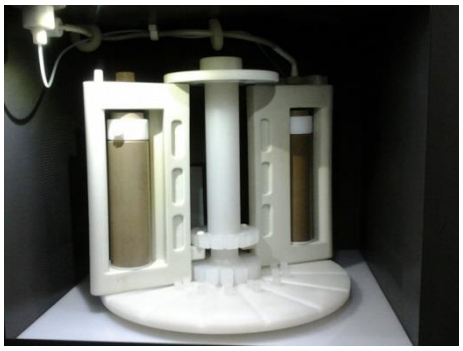
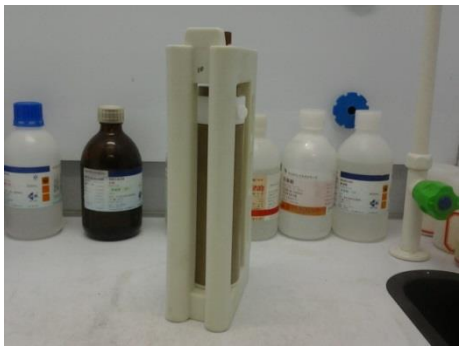
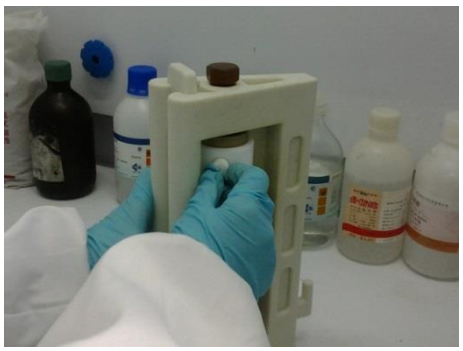
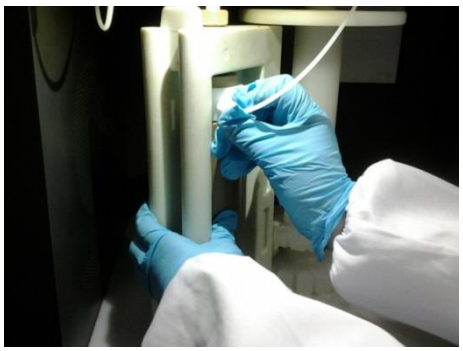
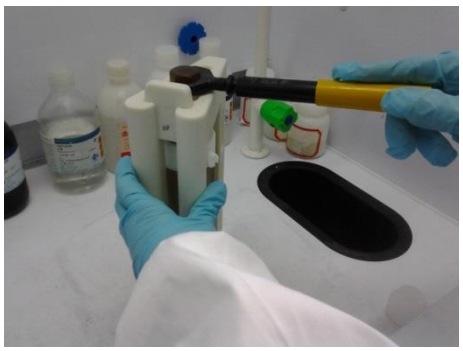


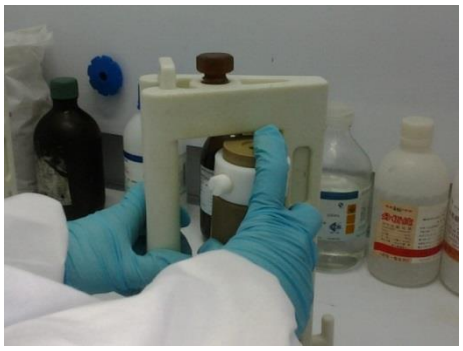
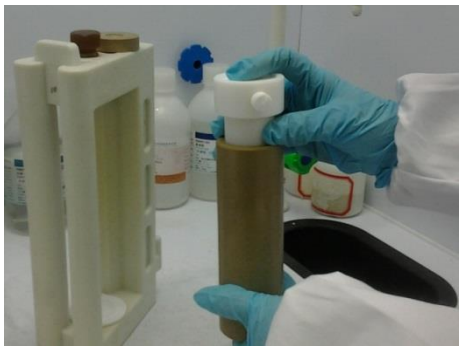
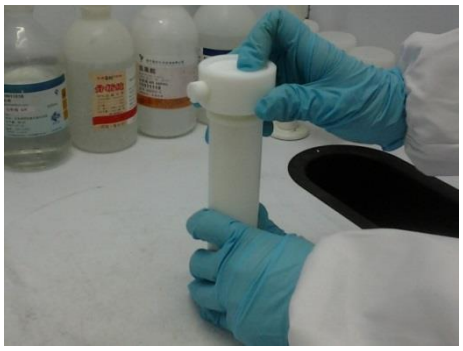
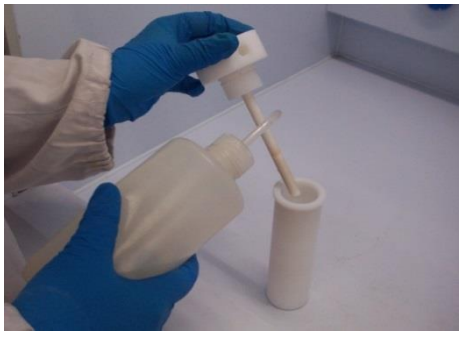
3. Place digestion vessel on the sample cup rack to keep digestion vessel stable. Prevent digestion vessel from falling down when moving it or adding the acid, which may result in acid leakage or injury because of acid splashing on operator.	
4. Move the remaining vessel components to fume hood to prepare for assembling. Note: The framework has been installed bottom cushion and top plug, The half circle of the bottom pad should face towards the frame. (see the right side of picture in right)	
5. Weigh the sample: use tool (scissors etc.) to smash the samples and stir them evenly. Use white paper as pad. (the smaller the sample pellet is, the better) Note: Do not contaminate the samples during smashing them.	
6. Plug the power, turn on scale, put the sample cup on the tray of the scale and press tare button.	
7. Weigh 0.1 g sample precisely. (The reproducible precision is 0.01 g) Note: The weight of organic sample is no more than 0.5g.If organic sample contains water, and when the weight of solid is less than 0.5g, the maximum sample weight should be less than 2.0g.The weight of inorganic sample is no more than 1.0g. Note: When microwave digestion, the weight of unknown sample should be strictly limited in 0.1g.You can try to do from 0.05g. If you are not sure the safety of unknown sample, experiment and operation, please connect Preekem	

applied technology center. Note: The sample must be loaded into control vessel (#0 vessel). Empty control vessel cannot be used. Note: Avoid the samples adsorption on the inner wall of digestion vessel. If this happens, flush the sample to the bottom of digestion vessel when adding the solvent.	
8. Add proper solvent in digestion vessel, pick up accurate amount of solvent with pipette, and add them in indigestion vessel. Note: For same batch of digestion: the sample and solvent added in each vessel must be same in types, weight and volume. Note: The minimum amount of solvent added is 3 mL. The total volume of acid solution added should be control within 15 mL. Note: Nitric acid, hydrochloric acid, hydrofluoric acid: in the vessel limited volume, you can mix them at any proportion; hydrogen peroxide: hydrogen peroxide is easy to occur fast and furious reaction with Oxidation of organic compounds, so don't add hydrogen peroxide when the sample may contain Oxidation of organic compounds or unknown specific components! Sulfuric Acid and phosphate: they are high-boiling acid, so you must control the temperature strictly and you should be very careful when you are using! Perchloric acid: It's dangerous to use in the seal container. Forbidden! Note: After adding acid, if the reaction is furious, you can wait for at least 10 minutes until Level doesn't change significantly, and then put on lid and digestion.	
9. Use expander to expand the opening: insert the expander into the skirt of control seal lid to the bottom, about 1/2 of the skirt, rotate it for 3~4 circles until the opening is expanded and then remove the expander from the lid. Note: The expander cannot be pulled off directly from seal lid, because the uneven force will deform the skirt and lead to failure of expanding the opening.	
10. Align the control seal lid with inner vessel; twist the skirt vertically in it, so that the entire skirt gets into the opening of the sample cup. Press down the vessel lid hard to put the opening of the sample cup gets into the round sealed slot of seal lid. Note: When closing the lid, the vessel should be placed on horizontal surface. When closing the lid, keep the vessel vertical to avoid poor seal quality due to skirt deformation.	

Note: There may be a chance that the seal lid will push up because of the air ompression when closing the seal lid of standard vessel. The operator can loosen the deflation screw to release some air, close the lid, and twist the deflation screw again.	
11. Confirm if the sleeve has inner pad installed. If no pad is found, must install the inner pad in the sleeve first, and then put the control vessel with lid covered into the sleeve. Note: Do not move the digestion vessel with hand holding the lid, to avoid vessel dropping and danger.	
12. Install the control top pad on the lid of control vessel. Note: Top pad to be installed in the slot of the vessel lid, face up the center of top pad where the slot locates.	
13. Place the framework on the horizontal bench top and the number label side should face towards the operator. Push the control vessel horizontally into the framework from the notch side (right side), to allow the sleeve cling with the bottom cushion flange to locate the sleeve. Note: The round holes that connect the air-guide tube fittings of the pressure sensor are located at one side. It is recommended that place the hole at skilled hand side to facilitate tightening the air-guide tube fittings.	
14. Insert the location pin into the top plug, put it through the top cushion to temperature sleeve for positioning, and then twist the top plug downwards by hand to complete the positioning. Note: It is to ensure accurate positioning and thus facilitate the insertion of temperature sensor.	

15. Tighten the top plug with torque wrench. The click or handle bending indicates tightening has been finished. Over tightening will exert excessive force on the vessel, resulting in the framework aging or broken. Note: Hold the bottom of the torque wrench with finger (see right picture), to ensure the mechanical effect.	
16. The installation method of the standard vessel is the same as the method of control vessel, recommending to put the deflation screw toward one side. Note: It is suggested that put the deflation screw towards the skilled hand side and do not put it too close to the edge of the frame to facilitate the twisting.	
17. First install the control vessel first (#0 vessel). Put control vessel at #0 position, hold the vessel by hand, insert the temperature probe from the hole at the top of the top plug of control vessel, lift the chuck, place the control vessel on the turntable, push it all the way through after aligning it with the dowel pin, align the vessel position with the dowel hole at the downside of the chuck, and then put down the chuck to fix them together. Tighten the air-guide tube fittings for pressure sensor. Note: Tighten the connector of the air-guide tube as tight as possible.	 
18. Install the digestion vessel in the oven chamber according to the centrifuge principle and keep the turntable stable. Twist the locknut on it. Note: The air-guide tube should not be stuck by vessel.	

19. Press the turntable button and examine the rotation of the turntable. It should rotate 360 degree in both directions, with no wire entangling. Note: Wire entangling means the temperature wire and pressure wire are entangled with each other.	
20. At first, confirm that the reaction vessel has been cooled to a safe temperature (≤ 80°C). Open the safety door, remove the locknut, lift the chuck, move the standard vessel to fume hood. Note: Open the vessel in fume hood. The released acid gas can be exhausted in time by fume hood, to avoid polluting the lab and harming operators. Note: Volatile elements such as arsenic, mercury and selenium. It is suggested that you shouldn't open digestion vessel until cooling to room temperature.	
21. Unscrew the deflation screw of the standard vessel in the fume hood to release the gas, until the deflation sound 'zzz' cannot be heard.	
22. Loosen the air-guide tube fittings, close the safety door, and wait until the pressure drops to zero. If it does not happen, further loosen the fittings, until the pressure drops to zero. Then completely unscrew the air-guide tube fittings. Move the control vessel to fume hood.	
23. Use torque wrench to unscrew the top plug. Note: If the gas is deflated thoroughly, loosen the top plug a little, and the vessel will be loose in the framework. If the gas is not deflated thoroughly, the inner vessel will clog the framework. If this happens, loosen the deflation screw until the gas is completely released.	

24. Remove the sleeve and inner vessel from the frame. Note: When removing the sleeve and inner vessel, press the lid with finger, to avoid the remaining pressure pop up the lid. (see the right picture)	
25. Push the inner vessel gently by hand from the bottom upward, remove the inner vessel and move it to the rack. Note: When removing the digestion inner vessel, hold the sample cup with hand. Do not move the digestion inner vessel by holding the vessel lid, in case the sample cup will drop during moving, and cause accident or injury.	
26. Place the inner vessel on rack or horizontal bench top; open the lid of the digestion vessel. Note: Do not operate without any support, in case the unstable force during opening the lid results in splashing and injury.	
27. Move the digested sample to container.	
28. Rinse the digestion vessel by immersing it in dilute acid. Note: During rinsing, no solution is allowed to flow into the well of the control seal lid. If there is solution in it, dry it before operation.	

Appendix 2: Examples of Digestion Methods (Reference)

The following examples of digestion methods are for reference only, you can use the same method for reference if you have the similar sample; but the same sample may have different components, so the digestion method may be different. you can make a relative adjust according to the actual condition.

Food

Name of sample	Weight
soybean oil, whole milk powder	0.2g
walnut fruit, hazelnut pulp	0.4g
flour, rice flour, sweet twist, fried bread stick, flour of millet, corn flour, buckwheat flour, wheat gluten, oatmeal, hot dry noodles, canned eight-treasure porridge, potatoes, dried vegetables, dry fruits, pulled figs, fresh edible fungus, soybean meal, mung bean flour, pea flour, lingonberry, spirulina, sesame powder, peanut powder, sunflower seed, essence of chicken, tahini, white granulated sugar, starch sugar, cornstarch, pea starch, concentrated juice, plum paste, ice-cream, ice lolly, Teething Biscuit, steamed bun, rice flour, jelly, egg oil, donkey-hide gelatin, golden syrup, caramel color, milk powder	0.5g
fresh vegetables, kidney bean class, leaf vegetables, fresh fruits, berry, small fruits	1.0g
yogurt, white wine, soy sauce, vinegar, sweetened condensed milk, palm oil	0.5mL
orange juice, grape, milk, yogurt	1mL

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	80	10	3
		2	120	20	3
		3	150	30	3
		4	180	40	10
Note: white wine, grape must be pretreated 120 minutes in the condition of 80°C					

Name of sample	Weight
ground pork, pork liver, bacon, Sausage, Instant Noodle seasoning packet, sesame oil essence, plant hydrolysis protein	0.3g
coffee, cheese, pidan, bread, cake, salmon, tomato mud, dried meat floss, potato chips, tea-leaves, honey, flower powder, tyrosine, ascorbyl palmitate	0.5g
eggs	0.5mL

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	120	10	3
		2	150	20	3
		3	180	30	3
		4	200	40	10

Note: Add reagent into sesame oil and liver sample wait 2 min until no obvious reaction, then start subsequent operation	
Name of sample	Weight
oyster(fresh), Ribbonfish(fresh), clam(fresh), eel(fresh)	0.3g
beef extract, river prawn(fresh), crab(fresh)	0.5g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	120	10	3
		2	150	20	3
		3	180	30	3
		4	210	40	10

Name of sample	Weight
wild pepper	0.5g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	7mL	1	120	10	3
HF	1mL	2	150	20	3
		3	180	30	3
		4	210	40	10

Name of sample	Weight
garlic	0.5g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	120	10	3
HF	0.2mL	2	150	20	3
		3	180	30	3
		4	210	40	15

Medicine

Name of sample	Weight
hard capsule shells (not contain sunscreen)	0.5g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	120	35	2
		2	150	35	3
		3	180	35	10

Note: If it contains sunscreen, you can add 0.2 mL HF during the digestion	
Name of sample	Weight
honeysuckle flower, astragalus root powder, peony powder, skullcap powder, liquorice root powder, hawthorn fruit powder, ginseng powder, salvia root powder, chinese angelica powder, saposhnikovia powder, dahurian angelica root powder, rehmannia root powder, tuber fleeceflower root powder, rsm extraction, safflor yellow extraction, meglumine	0.5g
Large leaves injectio	5mL

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	120	35	2
		2	150	35	3
		3	180	35	15

Note :If it has flocculent precipitate after digestion, you need to wait for some time or centrifugation in order to get clear liquid, or you can add 0.2 mL HF when digestion. And large leaves injectio must be pretreated to 1 mL in the condition of 80°C

Name of sample	Weight
soft capsule shell, refined fish oil	0.5g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	60	35	2
		2	120	35	2
		3	150	35	3
		4	180	45	10

Note: If the soft capsule shell contains sunscreen, you can add 0.2 mL HF or you can wait for some time to get clear liquid for testing.

Name of sample	Weight
1,4-bis(4-fluorbenzoyl)-benzene	0.1g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	3mL	1	130	15	3
H_2SO_4	3mL	2	160	20	3
		3	180	25	3
		4	200	30	3
		5	225	35	20

Biological samples

Name of sample	Weight
human hair powder	0.2g

pork bones, snake bones	0.25g
beef liver, urine , tissue fluid	0.5g
blood serum, bull's blood	1mL

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	120	10	3
		2	150	20	3
		3	180	30	3
		4	200	40	10

Name of sample	Weight
biological powder	0.25g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	120	10	3
H_2O_2	2mL	2	150	20	3
HF	0.5mL	3	180	25	3
		4	200	30	3
		5	225	35	10

Plastic

Danger: You must add HF or HBF4 for unknown components of sample, or it may have the danger of melting the master tank liners or sample cup.

Name of sample	Weight
polypropylene (PP), polyethylene (PE), polyamide (Nylon, PA), polybutylene terephthalate (PBT), plastic (PVC)	0.1g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	6mL	1	140	15	3
		2	170	25	3
		3	190	35	3
		4	210	40	15
Note: If it has supplement, you can add 0.5mL HF.					

Environmental samples

Name of sample	Weight
soil, precipitation, river mud, soil polluted by oil	0.1g

Solvent	Volume	Step	Temperature	Pressure	Time

	(mL)		(°C)	(atm)	(min)
HNO_3	6mL	1	150	15	3
HCl	2mL	2	180	25	3
HF	2mL	3	200	30	3
		4	220	35	15
Note: This method is suitable for testing element such as: Pb, Cr, Cd, Cu, Zn, Ni.					

Name of sample	Weight
soil, precipitation, River mud, soil polluted by oil	0.1g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	2mL	1	150	15	3
HCl	6mL	2	180	25	3
		3	200	30	3
		4	220	35	15
Note: This method is suitable for testing As, Hg, S					

Name of sample	Weight
glass fiber filters	0.3g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	6mL	1	120	10	3
HF	2mL	2	150	15	3
		3	180	25	3
		4	200	30	3
		5	220	35	15
Note: Cut up filters into pieces, add acid, and place it at least 10 min.					

Name of sample	Weight
glass fiber thimbles	0.5g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	9mL	1	120	10	3
HF	3mL	2	150	15	3
		3	180	25	3
		4	200	30	3
		5	220	35	15
Note: Cut up thimbles into pieces, add acid and place them for the whole night, which will make the mixture has a food mobility.					

Name of sample					Weight
microporous membrane					0.05g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	3mL	1	120	15	3
H_2O_2	2mL	2	150	25	3
		3	180	30	3
		4	200	35	15

Name of sample					Weight
finished water and incoming water from waste water plant					5-10mL
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	120	15	3
		2	140	25	3
		3	170	30	3
		4	190	35	15

Name of sample					Weight
coal					0.1g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	4mL	1	120	10	3
HF	1mL	2	150	15	3
		3	180	25	3
		4	200	30	3
		5	220	35	15

Name of sample					Weight
circuit board					0.1g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	120	10	3
HCl	1mL	2	150	15	3
HF	1mL	3	180	25	3
		4	200	30	3
		5	220	35	15
Note: Digest two times, the first time you need to add HNO_3, the second time you need to add HCl and HF.					

Light industry

Name of sample					Weight
pigment yellow (P.Y.12)					0.1g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	4mL	1	120	10	3
HF	1mL	2	150	20	3
		3	180	25	3
		4	200	30	3
		5	220	35	15

Name of sample					Weight
metal honeycomb type					0.1g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	4mL	1	120	10	3
HCl	2mL	2	150	30	3
		3	180	40	10

Name of sample					Weight
palladium on carbon					0.05g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	3mL	1	120	10	3
HCl	2mL	2	150	20	3
		3	180	25	3
		4	200	30	3
		5	220	35	15

Name of sample					Weight
catalyst (contains 80%TiO_2)					0.1g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	4mL	1	120	10	3
HF	2mL	2	150	20	3
		3	180	25	3
		4	200	30	3
		5	220	35	15

Name of sample	Weight

Ru-Zn alloy catalyst					0.1g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HCl H_2O_2	6mL 2mL	1	120	10	3
		2	150	20	3
		3	180	25	3
		4	200	30	3
		5	220	35	20

Name of sample					Weight
galvanized HSF board					0.1g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3 HCl	4mL 1mL	1	120	10	3
		2	150	20	3
		3	180	25	3
		4	200	30	3
		5	220	35	20

Name of sample					Weight
paint environmental protection board					0.1g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HCl H_2SO_4	5mL 0.5mL	1	120	10	3
		2	150	20	3
		3	180	25	3
		4	200	30	3
		5	225	35	15

Name of sample					Weight
lubricating oil					0.1g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	120	10	3
		2	150	20	3
		3	180	30	3
		4	200	40	10

Name of sample					Weight
purple paint, coal					0.1g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	4.5mL	1	120	10	3
HF	0.5mL	2	150	20	3
		3	180	25	3
		4	200	30	3
		5	225	35	15

Name of sample	Weight
exterior construction paint	0.1g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	8mL	1	120	10	3
H_2O_2	2mL	2	150	20	3
		3	180	25	3
		4	200	30	3
		5	225	35	15

Note: After adding HNO_3, you must pre-digest one hour in the condition of 120°C. If you don't do like that, it may have the danger of melting liner tube.

Name of sample	Weight
LiF	0.1g
thickening agent, fluorescent brightening agent, Stiffening agent	0.2g
colored printing paper	0.3g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	120	10	3
		2	150	20	3
		3	180	25	3
		4	200	30	3
		5	225	35	15

Name of sample	Weight
tinplate (Tinned Steel Sheet)	0.1g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	2mL	1	120	10	3
HCl	4mL	2	150	20	3
		3	180	30	10

Note: place it at least 10 minutes until the liquid level has no obvious change

Name of sample					Weight
lithium iron phosphate					0.1g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	1.5mL	1	120	10	3
HCl	4.5mL	2	150	20	3
		3	180	25	3
		4	200	30	3
		5	225	35	15

Name of sample					Weight
biomass in coal tar and coal tar					0.1g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	4mL	1	120	10	3
HF	1mL	2	150	20	3
H_2O_2	1mL	3	180	25	3
		4	210	35	15

Name of sample					Weight
tipping paper for cigarette					0.2g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	100	10	3
HF	1mL	2	130	20	3
HCl	1mL	3	160	25	3
H_2O_2	1mL	4	190	35	15

Name of sample					Weight
quartz					0.3g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HF	5mL	1	100	10	3
		2	130	20	3
		3	160	25	3
		4	190	35	15

Name of sample					Weight
printing ink					0.1g
Solvent	Volume	Step	Temperature (°C)	Pressure (atm)	Time (min)

	(mL)				
HNO_3	4mL	1	120	10	3
H_2O_2	1mL	2	150	20	3
HCl	1mL	3	180	25	3
		4	200	30	3
		5	220	35	20

Name of sample	Weight
liquid cosmetic (lotion, body lotion)	0.5g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	3mL	1	120	10	3
H_2O_2	2mL	2	150	20	3
		3	170	25	3
		4	190	30	3
		5	210	35	20

Note: Cosmetic which contains volatile stock such as alcohol should be put in 100°C water bath or put on electric heater to vaporize (forbidden vaporizing to dry). Fat and oil, cream powder should be pretreated 20 minutes in the condition of 100°C after adding 0.5-1.0mL ultrapure water and acid.

Name of sample	Weight
semi-fluid makeup (frost, honey, gel type)	0.3g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	4mL	1	120	10	3
H_2O_2	2mL	2	150	20	3
		3	170	25	3
		4	190	30	3
		5	210	35	20

Note: Cosmetic which contains volatile stock such as alcohol should be put in 100°C water bath or put on electric heater to vaporize (forbidden vaporizing to dry). Fat and oil, cream powder should be pretreated 20 minutes in the condition of 100°C after adding 0.5-1.0mL ultrapure water and acid.

Name of sample	Weight
solid cosmetic (foundation cream, pressed powder, lip sticks)	0.3g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	120	10	3
H_2O_2	1mL	2	150	20	3
		3	170	25	3
		4	190	30	3

		5	210	35	20
Note: Cosmetic which contains volatile stock such as alcohol should be put in 100°C water bath or put on electric heater to vaporize (forbidden vaporizing to dry). Fat and oil, cream powder should be pretreated 20 minutes in the condition of 100°C after adding 0.5-1.0mL ultrapure water and acid.					

Name of sample					Weight
soft light invisible powder, face triming grease					0.2g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	5mL	1	120	10	3
H_2O_2	1mL	2	150	20	3
HF	0.5mL	3	170	25	3
		4	190	30	3
		5	210	35	20
Note: The special cosmetic must add HF, or it may have the danger of melting digestion vessel.					

Metallurgy and steel

Name of sample					Weight
screw (toy)					0.3g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	4mL	1	120	10	3
HCl	1mL	2	150	30	3
		3	180	40	15
Note: Place it at least 10 minutes until the liquid level has no obvious change, then digest it.					

Name of sample					Weight
9Cr18, Cr4Mo4V, GCr15					0.1g
Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	1.5mL	1	120	10	3
HCl	4.5mL	2	150	20	3
HF	0.5mL	3	180	25	3
		4	200	30	3
		5	220	35	20

Name of sample					Weight
W					0.1g
titanium tungsten powder , TIO2					0.2g
Solvent	Volume	Step	Temperature	Pressure	Time

	(mL)		(°C)	(atm)	(min)
HNO_3	3mL	1	120	10	3
HF	2mL	2	150	20	3
		3	180	30	3
		4	200	35	15

Name of sample	Weight
special welding materials, gold-germanium alloys, lanthanum-gold alloy	0.1g

Solvent	Volume (mL)	Step	Temperature (°C)	Pressure (atm)	Time (min)
HNO_3	2mL	1	120	10	3
HCl	6mL	2	150	20	3
		3	180	30	3
		4	200	35	15

Note: After the gold-germanium alloys digesting, they will produce highly volatile $GeCl_4$, so you need to cool them to room temperature and then open the digest vessel.

PreeKem Scientific Instruments Co., Ltd.

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

Postal code: 201108

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

Fax: 86(21) 5442 7063

Web: www.preekem.com

E-mail: service@preekem.com

Technical Maintenance File

Operation Manual

Edition: 201603007