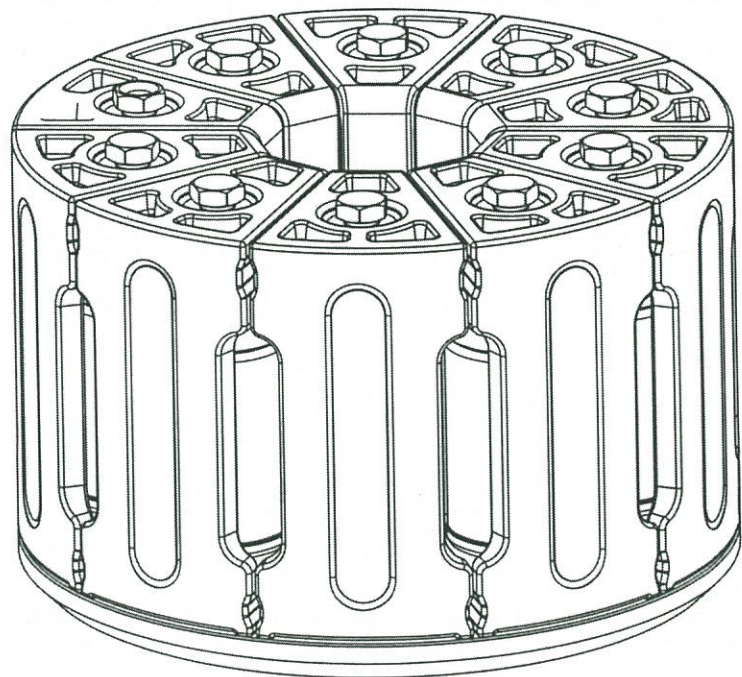




# Operation Manual

KJ-180



## 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.

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#### 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.

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## **KJ-180 Digestion Vessel 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 vessel components, leading to possible vessel damage.

**ATTENTION**

1.2 DO NOT heat concentrated alkali or salt solutions inside the vessel. PreeKem suggests that the concentration of alkali or salt should 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 damage.

**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 vessels may damage.

**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 installed in the instrument when the digestion is occurred in sealed vessels, and the control vessel must not be blank.

**DANGER**

1.7 In one batch 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 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.   |
| 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 splashing 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 splashing 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), which 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 puts 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-180 vessel is an airtight reaction vessel, which is designed for processing variety types of sample reaction including synthesis, extraction and digestion.

The KJ-180 vessel is made up of anti-corrosion inner vessel and strengthened framework. Upmost 10 KJ-180 vessels can be installed for reaction simultaneously to work in with different turntable assembly.

KJ-180 vessel can be divided into two kinds of different digestion vessels, one is the control digestion vessel and another is standard digestion vessel.

The control digestion vessel is possessed of special structure, which can be connected with the temperature sensor assembly and the pressure sensor assembly, achieving the real-time measurement in temperature, pressure and only one control digestion vessel included in one set of KJ-180.

The standard digestion vessel can together be installed into the cavity and simultaneously complete reaction with the control digestion vessel, the temperature and the pressure value can be referred to the measured value of control digestion vessel. The digestion vessels using temperature range is 0-250°C, Pressure range is 0-4MPa.

KJ-180 digestion vessel can be applied to COOLPEX smart microwave reaction system and WX-6000 microwave digestion system.

Following introductions are based on the turntable of 10 vessels.

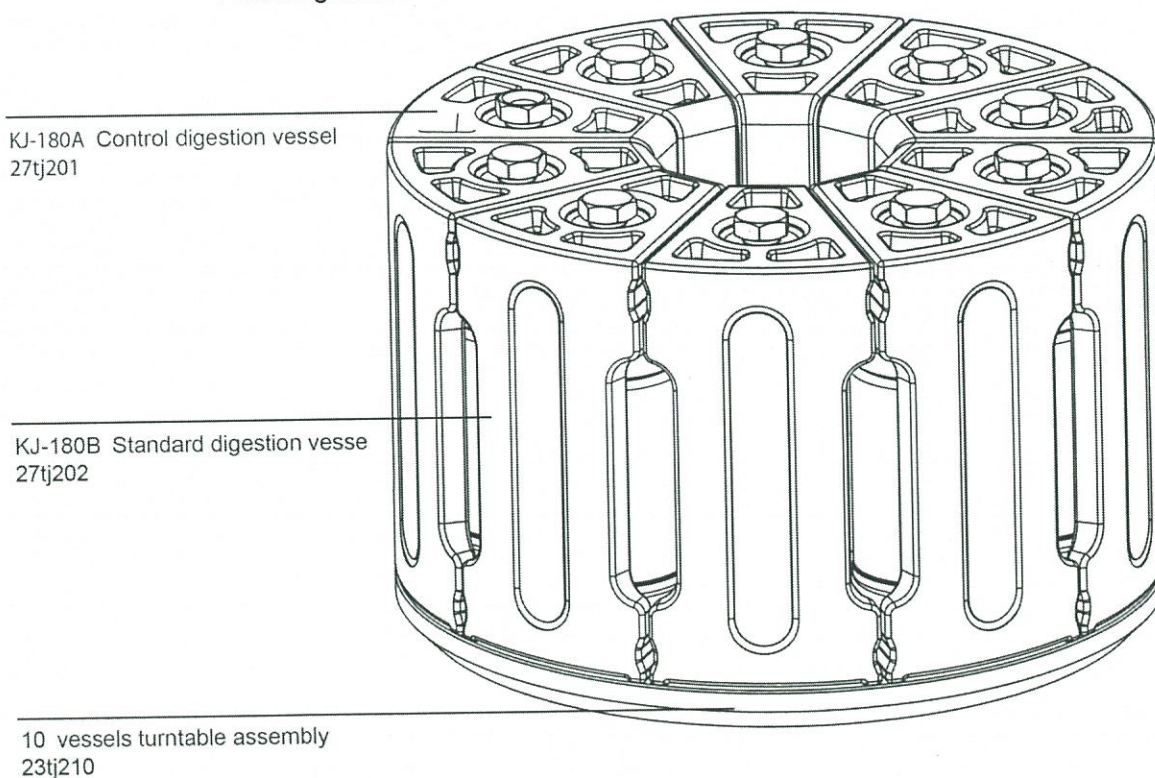


Figure 2-1: KJ-180 Digestion Vessel

Relation table between the different types of instrument with the turntable assembly:

	6 positions of turntable (27tj203)	10positions of turntable (23tj210)
COOLPEX	x	√
WX-6000	√	x

## 2-2 Technical Performance

Inner vessel material: TFM (3M®)

Sleeve material: PEEK (victrex®)

The volume of the digestion vessel: 100mL

Maximum working pressure: 4MPa

Maximum tolerance pressure: 15MPa

Maximum working temperature: 250°C

Maximum tolerance temperature: 300°C



### 3 The usage of rotor

The following contents are based on the installation of 10 positions of digestion vessel, which will elaborately introduce the usage method of the KJ-180 digestion vessel.

#### ATTENTION

**Attention should be taken in choosing different types of turntables and half couplings when applying KJ-180 to different types of instruments.**

**The digestion vessels number will differ based on different types of turntables.**

The relation table between the maximum installing positions of the digestion vessel with the turntables and half coupling

	6 positions of KJ-180 digestion vessel	10 positions of KJ-180 digestion vessel
<b>Turntable</b>	27tj203	23tj210
<b>Half coupling</b>	01fa103	09ca027

#### 3-1 The installation of turntable

Related components:

- Half coupling (09ca027)
- 10 vessels turntable assembling (23tj210)

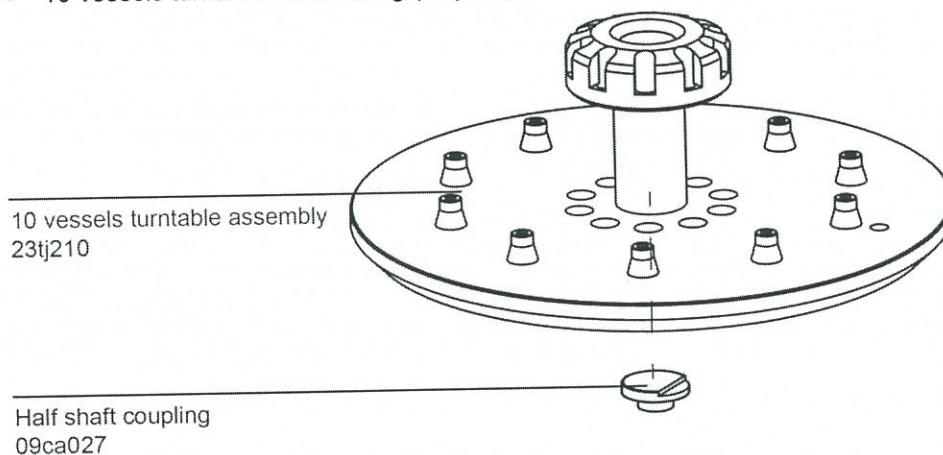


Figure 3-1: Turntable installation scheme

Procedure:

1. Prior to install the turntable ,the instrument door should be opened.
2. Insert the half coupling into the central hole of the bottom of the cavity inner.
3. Aligning the central gap with the coupling shaft when Install the whole turntable into the cavity

#### ATTENTION

**The turntable can be left in the cavity and needn't to be installed every time use.**



**ATTENTION**

The turntable installation method between the WX-6000 microwave digestion system and the COOLPEX smart microwave reaction system is the same, the only difference between them is that the allowable installation numbers of digestion vessel.

### 3-2 The installation of KJ-180A control digestion vessel

Related component:

- Control seal lid (27tj401)
- Sample cup (27ca001)
- Sleeve (27ca002)
- Framework (27ca003)
- Bottom cushion (27ca006)
- Control top cushion (27ca007)
- Control top plug (27ca008)
- Torque wrench (00fb701)
- Location pin assembly (01tj307)
- Opening expander (27ca009)

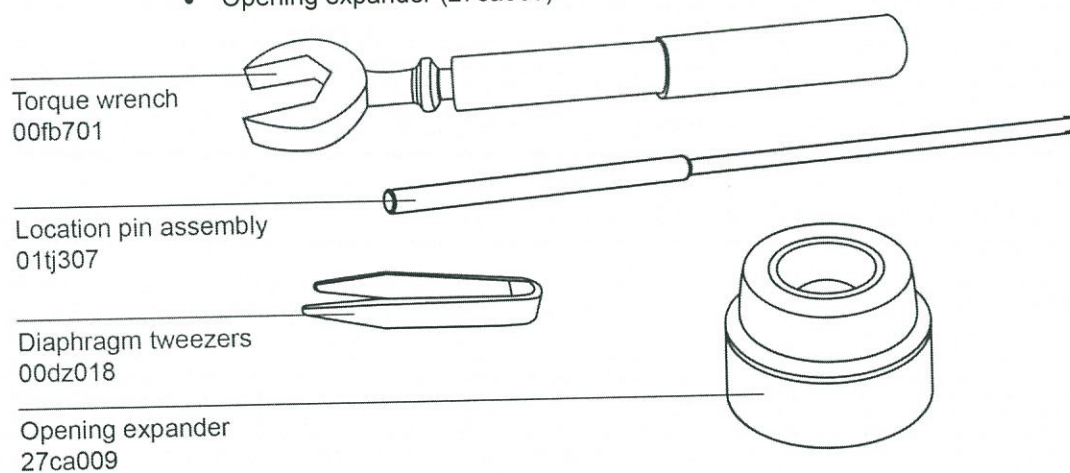


Figure 3-2: Tools scheme

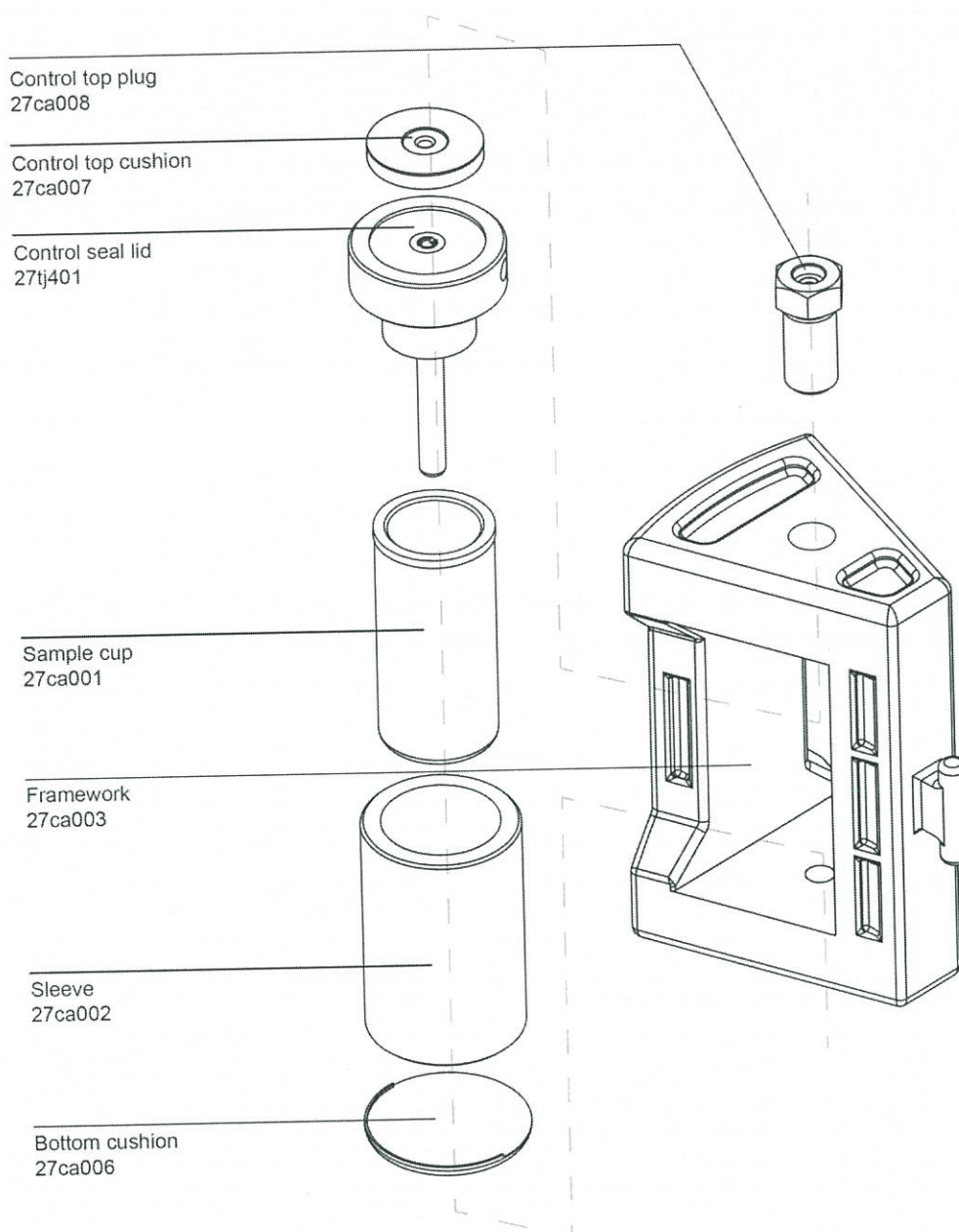


Figure 3-3: KJ-160A control digestion vessel

Procedure:

1. Weigh the sample via the balance and Place it into the sample cup.

**ATTENTION**

**The sample should be placed into the bottom of the sample cup to avoid the sample deposits attached at the side walls.**

**DANGER**

**The sample weight should be controlled for unknown samples, which should be less than 0.1g to avoid explosion caused by fierce reaction.**

2. Move the sample-contained sample cup into the fume hood and add appropriate amount of acid solvent, immerse the sample with acid solvent. Slightly shake the sample cup or stir it, dispersing the sample to evenly contact with the

reagent, if necessary putting the sample-contained and acid-reagent-contained cup into the electrical heater for a 15-30 min pretreatment.

**ATTENTION**

Visually observe the reaction between the sample with the reagent is fierce or not, if the reaction is fierce, wait the reaction to subside completely before capping the vessel.

**ATTENTION**

The volume range of the acid solvent for digestion reaction is 3-10 mL. The reagent volume range for other microwave chemical reaction should be 3-15 mL.

3. Insert the opening expander into the bottom of the seal lid skirt, rotating for 2-3 circles to expand the skirt. The sealing performance will greatly be improved through the expander which will effectively avoid the loss of the sample or elements during the reaction process.

**ATTENTION**

Do not tilt the axis of the opening expander in order to avoid the unevenly expanding or cracking and warping the skirt.

**ATTENTION**

Quickly cover the sample cup with the expanded seal lid, otherwise the skirt will recover to the original shape and lose the expanding effect, one more expanding step is needed.

**ATTENTION**

The tube of the temperature sensor was attached to the seal lid, great attention should be taken when insert or take out the opening expander to avoid touching with the tube or to break it.

4. Cover the seal lid (The rim of the sample cup should completely insert into the matched seal groove cavity) and insert the sample cup into the sleeve from top to down. Place the top cushion into the round-shape groove of the seal lid, the groove of the top cushion should be faced upward.

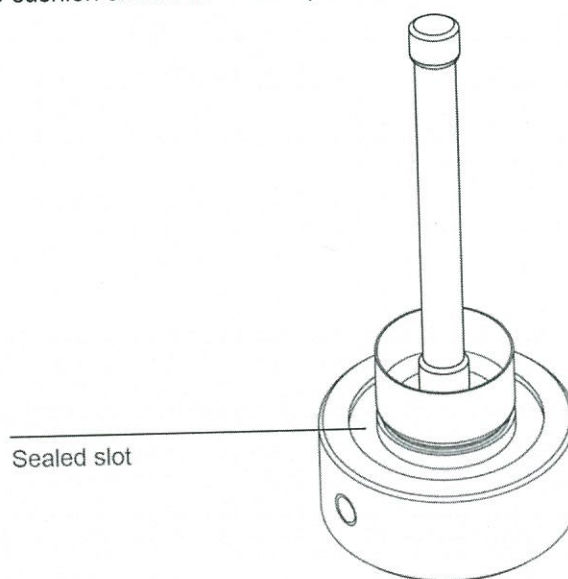


Figure 3-4: Sealed slot scheme



5. Insert the bottom cushion into the bottom of the framework and adjust the indentation side of the bottom cushion facing the same direction with the front of the framework (The framework front are without supporting arms, which protrude from the framework.).

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**ATTENTION**    **The digestion inner vessel is included of two parts: The control seal lid and the sample cup.**

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6. To ensure the accuracy of the installation position, inserting the location pin into the temperature sensor hole to make sure it is smooth.

Tighten top plug with torque wrench, when the torque reach the setting value, a sound of "KA" will be heard meaning the screw was tightened, then take out the location pin.

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**ATTENTION**    **If resistance exists when insert in the location pin, checking whether the digestion vessel was positioned on the center of the cushion, if not, loose the top plug and readjust the position and reinsert the location pin.**

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**DANGER**        **Torque wrench only! Do not use other common wrench to loose the top plug to avoid the framework damage.**

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### 3-3 The installation of KJ-180B standard digestion vessel

Related components:

- Common seal lid (27ca004)
- Deflation screw (23ca011)
- Safety diaphragm (09ca050)
- Sample cup (27ca001)
- Sleeve (27ca002)
- Framework (27ca003)
- Bottom cushion (27ca006)
- Standard top cushion (27ca005)
- Standard top plug (27ca014)
- Torque wrench (00fb701)
- Tweezers (00dz018)
- Opening expander (27ca009)

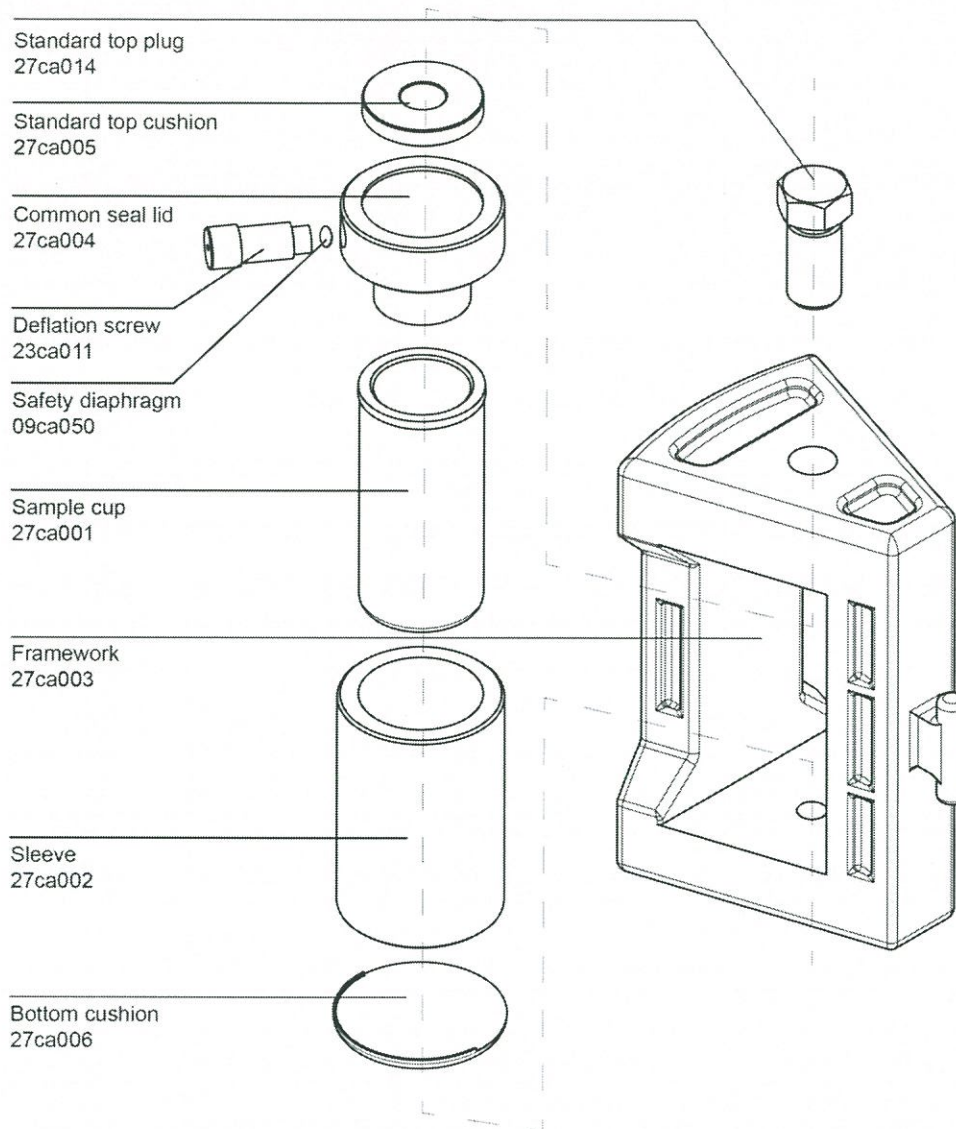


Figure 3-5: KJ-180B standard digestion vessel

## Procedure:

1. Weigh the sample by the balance and place them into the sample cup.

**DANGER**

The sample weight of the standard digestion vessel should be the same with the sample weight put inside the control digestion vessel.

**ATTENTION**

The sample should be placed in the bottom of the sample cup to avoid the sample deposits attached at the side walls.

**DANGER**

For unknown sample, the sample size should be less than 0.1g to avoid the explosion.

2. Move the sample-contained cup into the fume hood and add appropriate amount of acid reagent, immerse the sample with acid reagent. Slightly shake the sample cup or stir it, dispersing the sample to evenly contact with the reagent, if

necessary putting the sample-contained and acid-reagent-contained cup onto the electrical heater for a 15-30 min pretreatment.

**DANGER** The reagent volume of the standard digestion vessel should be the same with reagent volume of the control digestion vessel.

**ATTENTION** Observe the reaction between the sample and the reagent, if it is fierce, waiting till the reaction basically disappeared, then cover the seal lid.

**ATTENTION** The volume range of the acid reagent for digestion reaction is 3-10 mL. The reagent volume range for other microwave chemical reaction should be 3-15 mL.

3. Screw off the deflation screw from the seal lid and load the safety diaphragm into the deflation screw after checking the smooth of the air-bleed hole (Specified safety diaphragm only, allow only one piece loaded), ensuring the safety diaphragm placed flat on the groove of the deflation screw and the tail nod (Refer to diagram) faced the indentation of the deflation screw.

Screw the deflation screw into the seal lid, make sure the screw was tightly compress the safety diaphragm to avoid the gas leakage but shouldn't be over tightened.

The sealing property of safety diaphragm is very important, which invalid seal will lead to the difference temperature and pressure between the control digestion vessel and the standard digestion vessel and will cause the failure of experiment.

**ATTENTION** The standard seal lid means the common seal lid which installed with the deflation screw.

**ATTENTION** During the sample is digested, if pressure release or gas leakage occur near the safety diaphragm under the pressure less than 2.5MPa, please check the installation of the safety diaphragm, ensuring the tail nod of safety diaphragm facing to the indentation of the deflation screw.

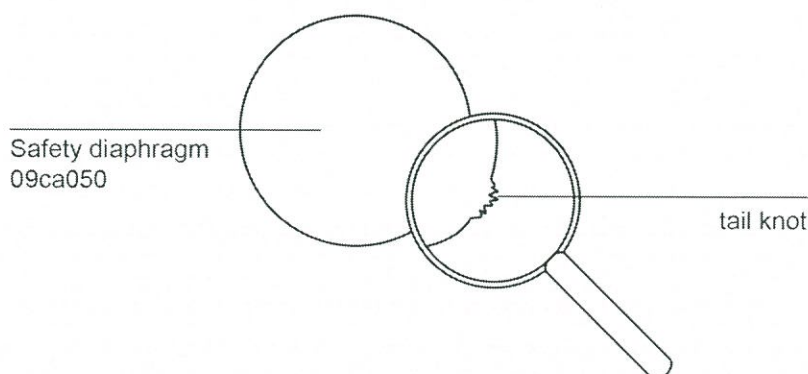


Figure 3-6: The Tail nod of the safety diaphragm



4. Insert the opening expander into the skirt and to the bottom of the seal lid, rotating it for 2-3 circles to expand the skirt. The seal property of the digestion vessel can greatly be improved through the expanding and effectively avoid the lose of sample or element during the reaction process.

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**ATTENTION** Do not tilt the axis of the opening expander in order to avoid the unevenly expanding or cracking and warping the skirt.

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**ATTENTION** Quickly cover the sample cup with the expanded seal lid, otherwise the skirt will recover to the original shape and lose the expanding effect, one more expanding step is needed.

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5. Cover the seal lid (The rim of the sample cup should completely insert into the matched seal groove cavity) and insert the sample cup into the sleeve from top to down. Place the top cushion into the round-shape groove of the seal lid, the groove of the top cushion should be faced upward.

6. Insert the bottom cushion into the bottom of the framework and adjust the indentation side of the bottom cushion facing the same direction with the front of the framework. (The framework front are without supporting arms, which protrude from the framework)

7. Tighten top plug with torque wrench, when the torque reach the setting value, a sound of "KA" will be heard meaning the screw was tightened, then take out the location pin.

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**Danger** Torque wrench only! Do not use other common wrench to loose the top plug to avoid the framework damage.

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### 3-4 Install the digestion vessel into the cavity

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**ATTENTION** The KJ-180 is different in temperature sensor assembly and the Air-guide tube assembly from model to model. The following material numbers only refer to the COOLPEX smart microwave reaction system. The Material number represents the serial of the component.

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Related components:

- KJ-180A control digestion vessel (27tj201)
- KJ-180B Standard digestion vessel (27tj202)
- Turntable assembly(10 Positions) (23tj210)
- Temperature sensor assembly (27tj204)
- Air-guide assembly (23tj203)
- Turntable assembly (6 positions) (27tj203) (Using together with WX-6000)

Procedure:

1. Check the temperature sensor assembly and air-guide assembly make sure the right installation of the temperature sensor assembly and air-guide tube assembly matching with KJ-180.

**ATTENTION**

**Please refer to the related chapter of the operation manual for the installation methods of the temperature sensor assembly and the air-guide tube assembly.**

**DANGER**

**Other model's temperature sensor assembly and air-guide tube can not be applied to KJ-180 digestion vessel, which will cause the abnormal of the instrument.**

2. Insert the temperature probe into the central hole of the assembled KJ-180A digestion vessel, then install the KJ-180A digestion vessel onto the turntable which position is labeled with 0.

3. Screw the connector of the air guide tube assembly into the seal lid, which should be tightly screwed.

4. Separately place the assembled digestion vessel into the suitable position of the turntable, the digestion vessel should be symmetrical and evenly placed to ensure the smooth running of the turntable.

**ATTENTION**

**Before delivery, the air guide tube assembly has already been adjusted and passed the seal capacity test, there is no need to tear the assembly apart during the operation, if need to be replaced for some special circumstance, please contact us ,our service engineer will guide you the disassemble process.**

### 3-5 The uninstall of the digestion vessel

Take out the standard digestion vessel with framework from the cavity when the temperature value down to 80°C and pressure value down to 0.05MPa.

If the temperature down to 80°C while the pressure inside the vessel is still higher than 0.05MPa, gas may burst out when open the seal lid, thus attention should be taken when operator work under this circumstance.

**DANGER**

**Before opening the digestion vessel, the operator should be equipped with the anti-acid glove, the eye protection, the mask and the lab coat. Be attention to the danger of corrosion and scald.**

**DANGER**

**Uninstall the digestion vessel is not allowed when the temperature is above 80°C.**



<b>DANGER</b>	<b>Taking out the digestion vessel from cavity is prohibited when the temperature is above 120°C.</b>
<b>ATTENTION</b>	<b>Place all the components into the cupboard when disassembly finished to avoid missing.</b>
	<p>Related components:</p> <ul style="list-style-type: none"> <li>• KJ-180A control digestion vessel (27tj201)</li> <li>• KJ-180B standard digestion vessel (27tj202)</li> <li>• Turntable assembly (23tj210)</li> <li>• Temperature sensor assembly (27tj204)</li> <li>• Air-guide tube assembly (23tj203)</li> <li>• Turntable assembly (27tj203) (Use together with WX-6000)</li> </ul> <p>Procedure:</p> <ol style="list-style-type: none"> <li>1. Take the standard digestion vessel from the turntable to the fume hood.</li> <li>2. Loose the deflation screw to release the gas in the fume hood.</li> </ol>
<b>ATTENTION</b>	<b>To make sure the fume hood is under the working condition and the acid can be exhausted before loosening the deflation screw.</b>
<b>DANGER</b>	<b>When the deflation lose, the acid gas will be released from the deflation screw hole ,pay attention to the corrosion and scald of the acid gas.</b>
	<ol style="list-style-type: none"> <li>3. When the temperature down to 80°C slowly loose the connector of the air-guide tube assembly which is connected with the control digestion vessel, screw off the connector when the gas released.</li> </ol>
<b>ATTENTION</b>	<b>The control digestion vessel can be moved to the back of the cavity, thus the released acid gas can be exhausted by the backing exhausting system.</b>
	<ol style="list-style-type: none"> <li>4. Take the control digestion vessel from the turntable and pull the temperature sensor assembly out and move the digestion vessel into the fume hood.</li> </ol>
<b>ATTENTION</b>	<b>Lower the control digestion vessel or place it at an angle of 45° after taking the digestion vessel from the turntable, leaving enough space to pull the temperature sensor assembly out.</b>
	<ol style="list-style-type: none"> <li>5. Twist the top plug with the torque wrench and pull the digestion vessel out from the framework including digestion inner vessel, sleeve inner pad and the sleeve .Please slightly press the seal lid to avoid the seal lid being pushed by the</li> </ol>

residual gas which may possibly cause the falling of the seal lid, the loss of sample and the damage of seal lid.

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<b>ATTENTION</b>	<b>Screw off the deflation screw in the fume hood before opening the standard digestion vessel, waiting till all the gas released then take off the top plug and take out the inner digestion vessel, otherwise the operator will be in danger of acid liquid splash caused by inner pressure of the digestion vessel.</b>
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<b>ATTENTION</b>	<b>After Loosing the top plug and releasing all the gas, a gap can be observed between top cushion and the framework, if the seal lid raise up and touch the framework ,continuing to lose the deflation screw till the gas been completely released.</b>
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6. Move the top cushion off and hold the sleeve by one hand pushing the digestion inner vessel a little bit out from the bottom and taking it to the sample holder.

7. Holding the sample cup placed in the sample holder and taking off the seal lid, after that the solution can be transferred to other container.

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<b>DANGER</b>	<b>Opening the seal lid in the air is prohibited, otherwise will possibly result in the acid solution splashing and damage caused by ununiformed stress.</b>
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<b>ATTENTION</b>	<b>All the components of the framework should be rinsed and dried prior to repeatable usage.</b>
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## Appendix 1: Operating specification of KJ-180 digestion vessel

Before install the digestion vessel, the operator should be equipped with the **anti-acid glove**, the **eye protection**, the **mask** and the **lab coat**.

### Preparation

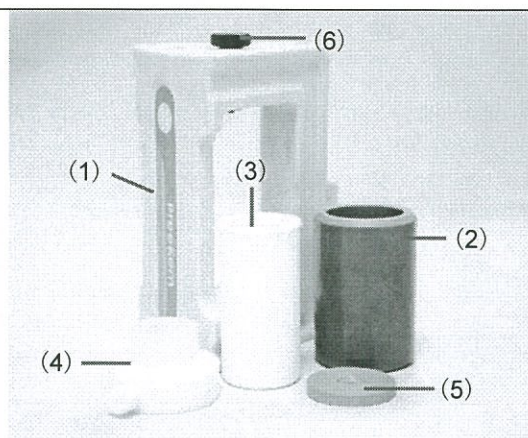
1. Check the framework, digestion vessel, and all the components and ensure that all of which are dry and no crack;

Further to make sure the skirt of the seal lid is round and no damage, the screw thread is in good shape and the gas release channel is smooth.

Note: 1. Make sure all components are dry (For inner vessel: only the exterior of the inner vessel need to be keep dry), Drops of liquid or particles will absorb microwave energy, causing localized heating which may char vessel components, leading to possible vessel damage.

2. Component name: (1)Framework (2)Sleeve (3)Sample cup (4)Seal lid (5)Top cushion (6)Top plug.

3. The hole in the top plug, the top cushion and the seal lid of the control digestion vessel (No.0) is the big difference compared to the standard digestion vessel.

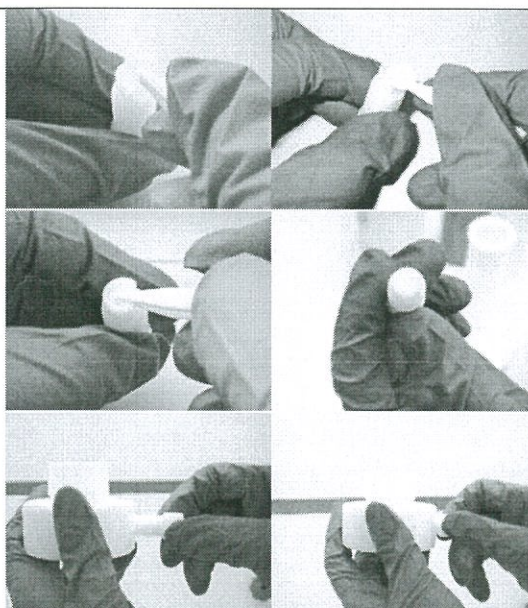


2. Screw off the deflation screw from the seal lid of the KJ-180B standard digestion vessel, take off the safety diaphragm from the deflation screw by tweezers and observe whether there is any bubble, if apparent bubble can be seen or the safety diaphragm broken with a hole, the safety diaphragm should be replaced.

The deflation screw should be horizontally upward faced when inserting the safety diaphragm into the bottom of the deflation screw via the tweezers after checking the smooth of the gas release channel then screw the deflation screw into seal lid to compress the safety diaphragm ensuring no gas leakage.

Note: 1. Screw the deflation screw to the bottom of the seal lid, stop when you feel the screw is tight by your hand and do not over-tight, otherwise the screw will lose effect.

2. Only one safety diaphragm allowed to be



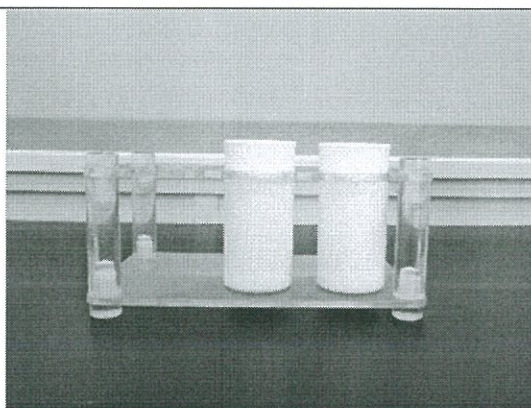
inserted to the deflation screw, if more than one safety diaphragms loaded, the safety pressure system will lose effect.

If without safety diaphragm, the seal lid can't be sealed and will cause the failure of the reaction.

3. Check the safety diaphragm of the standard digestion vessel before every operation, the control digestion vessel needn't to be loaded with safety diaphragm.

4. Ensuring the safety diaphragm tail nod faced the indentation of the deflation screw.

3. Place the digestion vessel into the sample holder and level it, the acid solution may possibly spill out during the moving or adding acid process if the digestion vessel is tilt.

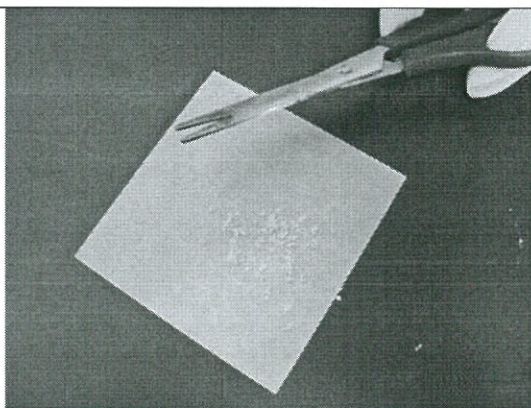


4. Place the other parts of the vessel into the fume hood ready for digestion vessel installation.

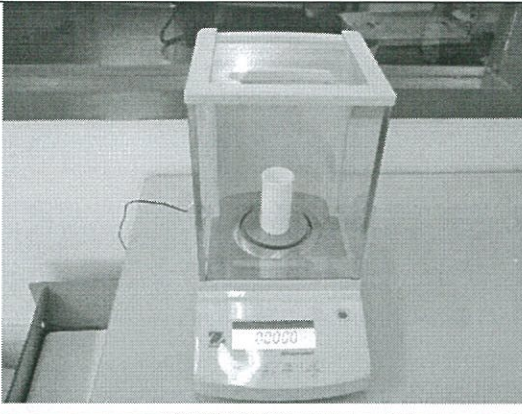
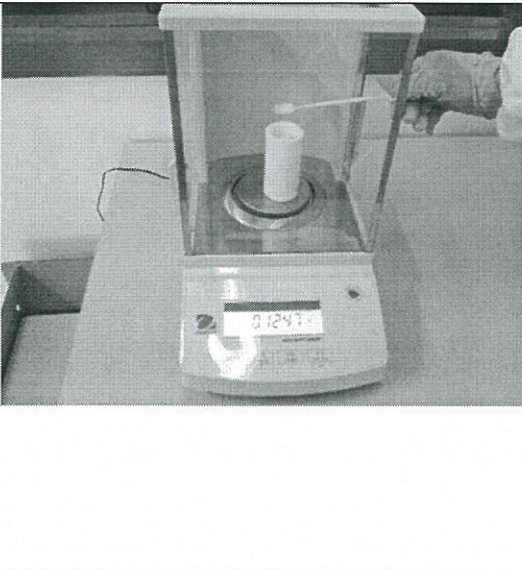
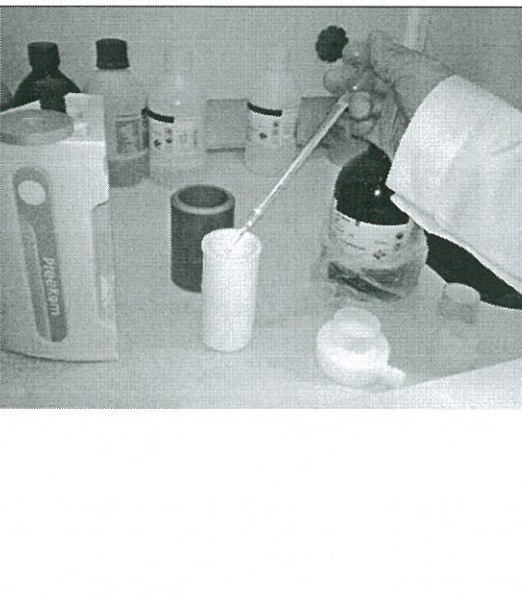
## Weigh the sample

5. Smash the sample by tool (Like scissors) and stir it to mix well on the white paper. (the sample should be the smaller the better)

Note: Avoid to contaminate the sample during the smashing process.



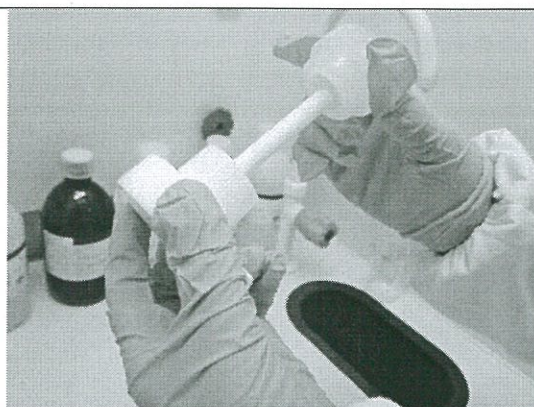


<p>6. Connect the balance with the power supply and switch it on, place the sample cup onto the balance to weigh the net weight.</p>	
<p>7. Weigh 0.1g sample (the weight should be accurate to 0.01g)</p> <p>Note: during the weighing process, do not touch the table of the balance to ensure the accuracy.</p> <p>2. The sample weight can be changed according to the actual situations, the normal range should be from 0.1-0.5g.</p> <p>3. The control digestion vessel (No.0 vessel) must be contained with sample which can not be used as blank.</p> <p>4. Avoid adhering the sample onto the wall of the digestion inner vessel, if happens, wash the sample down to the bottom of the digestion inner vessel by reagent.</p>	
<h2>Vessel install</h2>	
<p>8. Adding suitable reagent via suction pipet or pipette into the digestion vessel.</p> <p>Note: 1. The reagent volume should at least be 3mL the whole volume of the reagent should be controlled within 15mL.</p> <p>2. The acid solution for digestion: Advice to use the acid of Nitrate acid, hydrochloric acid, Hydrogen peroxide-acid, hydrofluoric acid. While perchloric acid is prohibited, concentrated sulfuric acid and phosphoric acid can't be used solely, which should be mixed with other acid otherwise explosion may occur.</p> <p>3. For the same batch of the sample and the reagent should be same in terms of the type, the weight and the volume.</p>	



9. Expanding the skirt with the opening expander, inserting the opening expander into the half of the skirt with rotation of 3-4 circles to achieve the expanding effect, then rotating the opening expander out of the skirt.

Note: The opening expander can't be directly pull out from the skirt, the direct pull out may cause the uneven force leading to the warping of the skirt and lose the expanding effect.

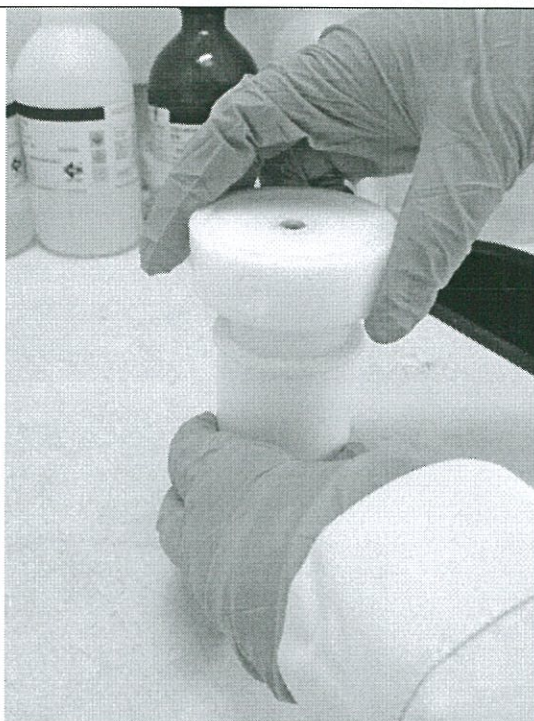


10. Align the control seal lid with the digestion inner vessel and rotate skirt vertically and wholly into the sample cup. Compress the seal lid to fit the sample cup into the seal groove cavity of the seal lid.

3. Note: Levelly place the seal lid to the sample cup during the sealing process.

2. Keeping the seal lid vertical with the sample cup to avoid the skirt warping and lead to the bad sealing property.

3. When sealing the standard digestion vessel, the seal lid may be propped up by the gas inside the sample cup, if this happens, loosening the deflation screw to release the gas and screw it back again when the seal lid inserted.

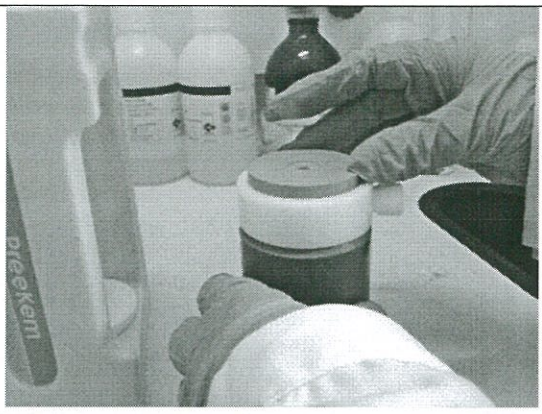

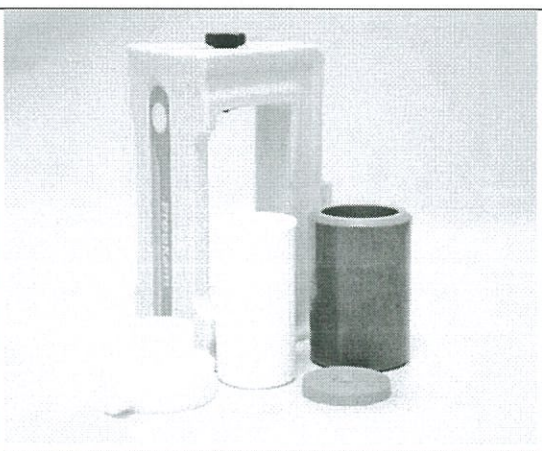
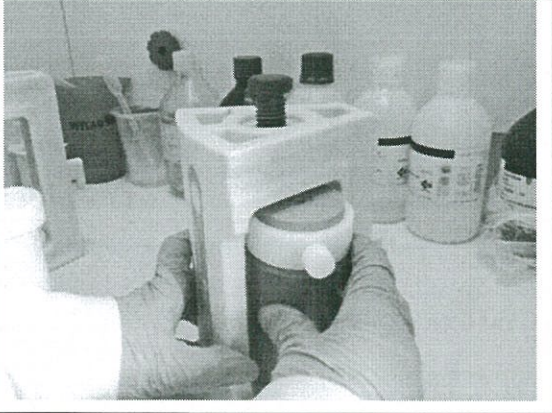


11. Insert the sealed control digestion vessel into the sleeve.

Note: Holding the seal lid to move the digestion inner vessel is prohibited, which will cause the dangerous of the falling of the vessel.



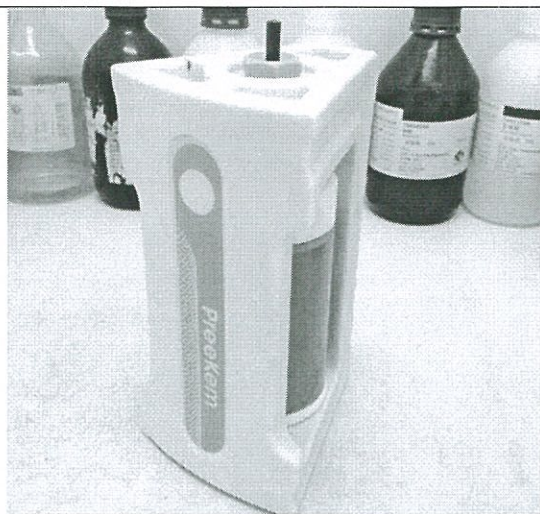


<p>12. Install the top cushion onto the control seal lid.</p> <p>Note: Insert the top cushion into the groove of the seal lid, the indentation of the seal lid should be faced upward.</p>	
<p>13. Install the bottom cushion and the top plug into the framework.</p> <p>Note: The indentation side of the bottom cushion faced with the front of the framework (the framework front refer to side without arms protruding out, see the right picture)</p>	
<p>14. Make the framework labeled with Number side face to the operator.</p> <p>Note: For the convenient of the further procedure.</p>	
<p>15. Place the framework horizontally on the table, push into the control digestion vessel from the front of the framework, the sleeve should tightly touch the flange of the bottom cushion. (The framework front is without arm, which is protruding out.)</p> <p>Note: Advice the hole of the connector of the air guide tube assembly facing the main hand, which will be convenient for screwing.</p>	



16. Insert the location pin from the top plug into the top cushion and then through the tube of the temperature sensor, finish the location step by screw tight the top plug.

Note: To ensure the correct location for the convenient of inserting the temperature sensor.



17. Screw the top plug with the torque wrench, when heard a sound of "KA" which means the plug is tightened. Do not over tighten the top plug, for it may cause over pre-tightening force and lead to the broken or cracking of the framework.

Note: Holding the torque wrench by the end (See picture in right side), to ensure the Mechanic effect.

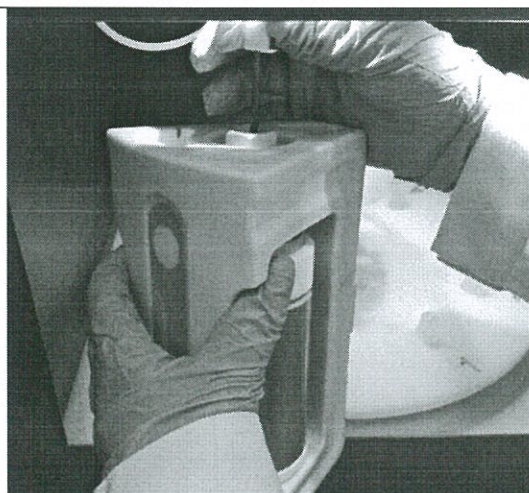


18. The installation of the standard digestion vessel is the same with the control digestion vessel, advice the deflation screw facing the same direction.

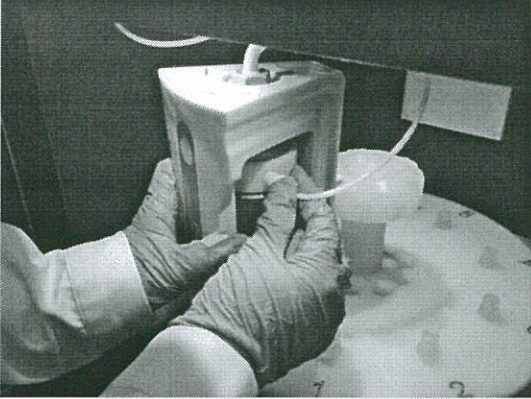
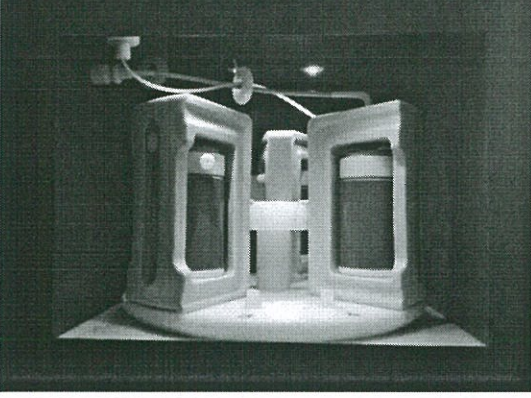

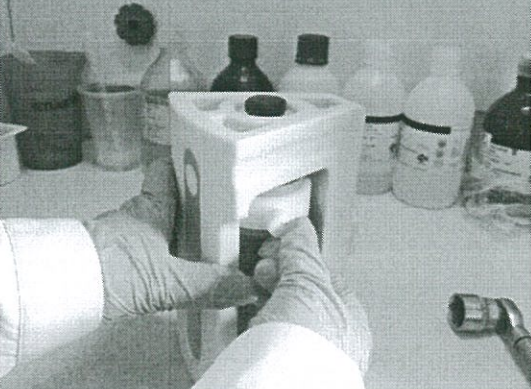
Note: The deflation screw advised to be faced with the main using hand and do not be closed with the framework.

19. Install the control digestion vessel (No.0) first and put it onto position 0, holding the vessel by hand, insert the temperature sensor probe into the hole of the top plug, place the control vessel onto the turntable and connecting it with the air guide tube assembly.

Note: Tighten the connector of the air-guide tube assembly as tight as possible.



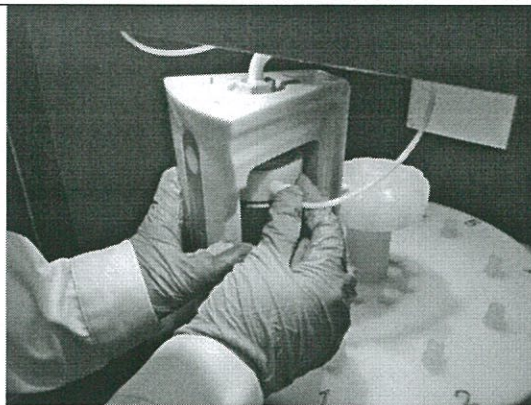


	
<p>20. Press the turntable button and observe the rotation condition of the turntable.</p> <p>The turntable should rotate clockwise and anticlockwise in 360 degrees without wire entangling.</p> <p>Note: wire entangling refers to the temperature wire entangled with the pressure wire.</p>	
<h2>Open the vessel</h2>	
<p>21. Firstly, to confirm the reaction vessels have been cooled down to safe temperature (<math>\leq 80^{\circ}\text{C}</math>). Open the safety door, move the standard vessels into fume hood.</p> <p>Note: Open the vessel in the fume hood, so the released acid gas can be exhausted out, avoiding the contamination to both of lab and human.</p>	
<p>22. Keep loosening the deflation screw to release gas in the fume hood until the sound of "Zi" can't be heard.</p>	



23. Loose the connector of the air-guide tube assembly and close the door, waiting till the pressure value is down to zero, if the value can't go down to zero, open the cavity door and further to loose the connector and close the door to check value again.

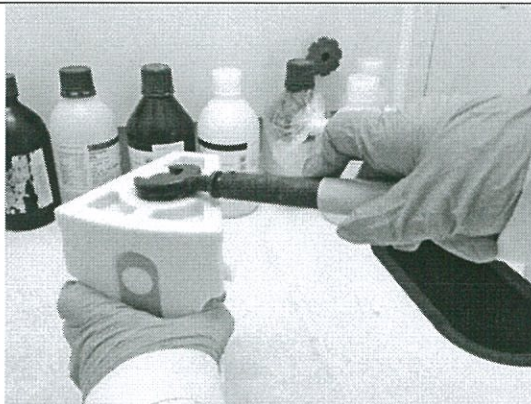
When the value is down to zero, screw off the connector and move the control digestion vessel into the fume hood.



24. Screw off the top plug via torque wrench.

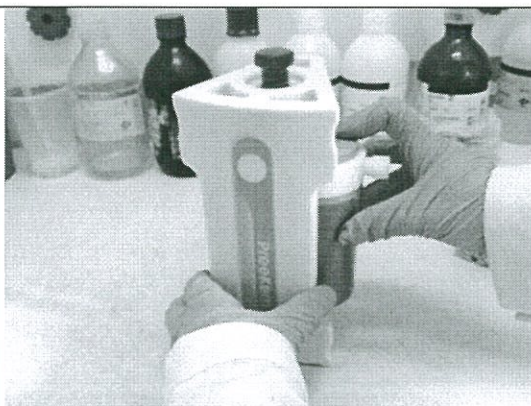
Note: If the gas has been thoroughly released, the vessel will be loosed by a slight loose of the top plug.

If the gas hasn't been completely released the inner vessel will be propped up to the framework which means the top plug need to be further loosed.



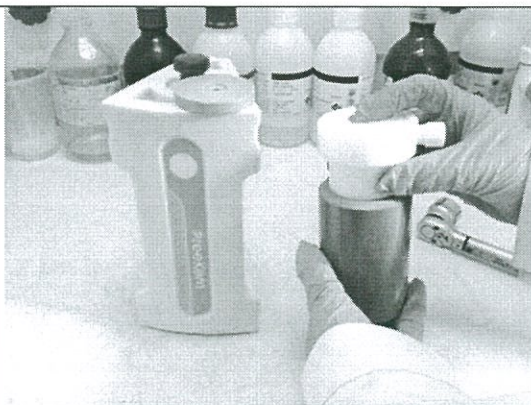
25. Take out the sleeve and the digestion inner vessel

Note: When taking out the sleeve together with the digestion inner vessel, pressing the seal lid with finger to avoid seal lid pop up by the remaining gas. (Refer to the right picture)



26. Slightly push the inner vessel from bottom to up and move it to the sample holder.

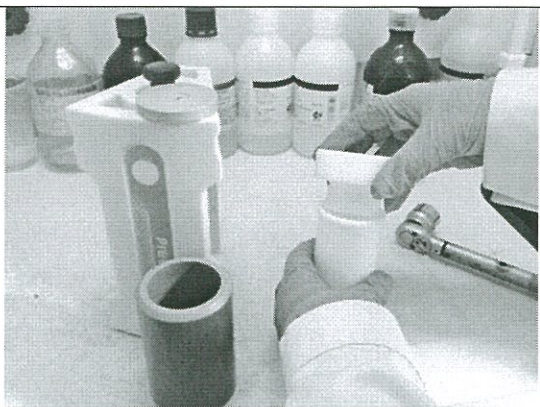
Note: when taking out the inner vessel the hand must hold the body of the sample cup, holding the seal lid is prohibited, which will possibly cause the accident or injury by the sample cup falling.



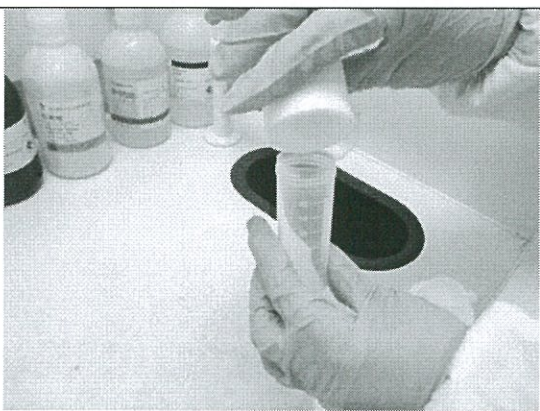


27. Place the inner vessel into the sample holder or on the level table, then open the seal lid.

Note: Opening the seal lid in the air is prohibited, which will cause unstable force resulting in acid solution splashing and injury.



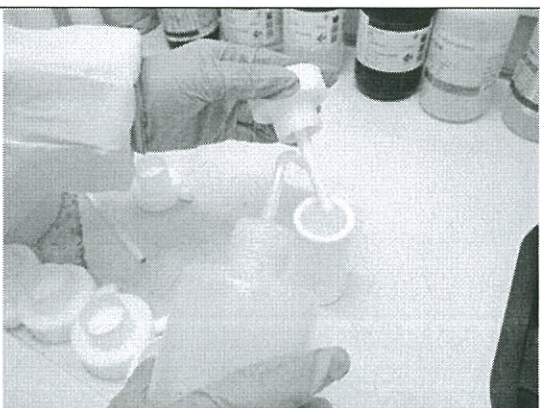
28. Move the digested solution to the container.



## Rinse

29. Soak and rinse the digestion vessel into the dilute acid solution.

Note: a great attention should be taken when rinsing the seal lid of the digestion vessel, the tube of control vessel should be keep dry, if it is dipped with solution, dry it prior to the next operation.



## Appendix 2: Examples of Digestion Methods (Reference)

Examples of Digestion Methods (Reference)									
No	Sample	Weight (g)	Solvent	Volume (mL)	Temp (°C)	Pressure (atm)	Time (min)	Note	Results
<b>Food</b>									
1	Flour	0.2	HNO <sub>3</sub>	5	130	15	2		Clear
					150	25	2		
					180	35	6		
2	Rice	0.2	HNO <sub>3</sub>	5	120	15	2		Clear
					160	20	6		
3	Milk powder	0.2	HNO <sub>3</sub>	5	80	15	2		Clear
					120	20	2		
					160	25	2		
					200	30	4		
4	Fresh fruit	1.0	HNO <sub>3</sub>	4	120	10	2		Clear
					150	15	2		
					180	20	4		
5	Fresh vegetable	1.0	HNO <sub>3</sub>	4	120	10	2		Clear
					150	15	2		
					180	20	4		
6	Dried vegetable	0.2	HNO <sub>3</sub>	4	120	15	2		Clear
					150	20	2		
					180	25	4		
7	Cooking oil	0.2	HNO <sub>3</sub>	5	100	15	2	Pretreatment at 120°C for 20 min if needed	Clear
					140	20	2		
					170	25	4		
					200	30	5		
8	Cake	0.2	HNO <sub>3</sub> H <sub>2</sub> O <sub>2</sub>	4 1	100	8	2		Clear
					130	10	2		
					180	20	3		
9	Soy sauce	0.3	HNO <sub>3</sub>	4	120	8	2		Clear
					160	12	5		
10	Vinegar	0.3	HNO <sub>3</sub>	4	100	6	2		Clear
					150	10	5		



Examples of Digestion Methods (Reference)									
N o	Sample	Weigh t (g)	Solven t	Volume(mL )	Tem (°C)	Pressur e (atm)	Tim e (min )	Note	Result s
11	Chilli sauce	0.2	HNO <sub>3</sub>	4	120	15	2		Clear
					150	18	2		
					180	25	4		
12	Royal jelly	0.3	HNO <sub>3</sub> H <sub>2</sub> O <sub>2</sub>	4 0.5	100	10	2		Clear
					130	15	2		
					160	20	2		
					190	24	4		
13	Ice cream without chocolate	0.2	HNO <sub>3</sub>	5	120	10	2	Pretreatmen t at 120°C for 20 min	Clear
					170	15	2		
					200	20	5		
14	Jelly	0.5	HNO <sub>3</sub>	5	100	10	2		Clear
					140	20	6		
15	Drink	0.5	HNO <sub>3</sub>	5	100	10	2		Clear
					150	20	5		
16	Beer	0.5	HNO <sub>3</sub>	5	120	10	2		Clear
					150	15	5		
17	Grape wine	0.5	HNO <sub>3</sub>	5	120	10	2		Clear
					150	15	5		
18	Rock candy	0.3	HNO <sub>3</sub>	6	130	15	2		Clear
					150	25	2		
					170	35	2		
					190	45	5		
19	Chewing gum	0.2	HNO <sub>3</sub>	5	130	15	2		Clear
					150	25	2		
					180	35	2		
					200	40	5		
20	Concentrate d Fruit juice	0.3	HNO <sub>3</sub>	8	100	20	5		Clear
					130	30	2		
					150	35	2		
					180	40	5		



Examples of Digestion Methods (Reference)									
No	Sample	Weight (g)	Solvent	Volume(mL)	Tem. (°C)	Pressure (atm)	Time (min)	Note	Results
Fisheries									
1	Ribbonfish(fresh)	0.5	HNO <sub>3</sub>	6	150	15	4		Clear
					180	20	4		
2	Squid(fresh)	0.5	HNO <sub>3</sub>	6	150	15	4		Clear
					180	22	4		
3	Clams(fresh)	0.5	HNO <sub>3</sub>	6	150	15	4		Clear
					180	22	4		
4	Mussels(fresh)	0.5	HNO <sub>3</sub>	6	150	15	4		Clear
					180	20	4		
5	Eel(fresh)	0.5	HNO <sub>3</sub>	6	150	10	4		Clear
					180	18	4		
					200	28	4		
6	Squid (dried)	0.1	HNO <sub>3</sub>	5	150	10	4		Clear
					180	18	4		
					200	25	4		
Plant									
1	Bush (GBW07603)	0.2	HNO <sub>3</sub>	4	150	15	4		Clear
					180	20	4		
2	Tea (GBW07605)	0.2	HNO <sub>3</sub>	4	150	18	4		Clear
					180	24	4		
3	Honeysuckle	0.2	HNO <sub>3</sub>	4	140	18	4		Clear
					180	25	4		
4	Salvia(dried)	0.2	HNO <sub>3</sub> HF	4 0.5	140	18	4		Clear
					180	25	4		
5	Konjac flour	0.2	HNO <sub>3</sub>	4	150	15	4		Clear
					180	22	4		
6	Radix powder	0.2	HNO <sub>3</sub>	4	150	15	4		Clear
					180	22	4		
7	Ginseng(dried)	0.2	HNO <sub>3</sub> HF	4 0.5	140	18	4		Clear
					180	25	4		

Examples of Digestion Methods (Reference)									
No	Sample	Weight (g)	Solvent	Volume (mL)	Temp. (°C)	Pressure (atm)	Time (min)	Note	Results
Biological samples									
1	Human hair	0.2	HNO <sub>3</sub>	4	150	18	4		Clear
					180	22	4		
2	Meat	0.2	HNO <sub>3</sub>	4	150	15	4		Clear
			H <sub>2</sub> O <sub>2</sub>	1	180	25	5		
3	Liver	0.2	HNO <sub>3</sub>	4	150	15	4		Clear
			H <sub>2</sub> O <sub>2</sub>	1	180	25	5		
4	Blood	0.2	HNO <sub>3</sub>	4	150	15	4		Clear
					180	25	4		
5	Bone	0.2	HNO <sub>3</sub>	4	150	16	4		Clear
					180	22	4		
Plastic									
1	Polypropylene (PP)	0.1	HNO <sub>3</sub>	4	140	15	4	Add a little HF if containing fiberglass	Clear
					170	20	4		
					200	26	5		
2	Polyethylene (PE)	0.1	HNO <sub>3</sub>	4	140	15	4	Add a little HF if containing fiberglass	Clear
					170	20	4		
					200	26	5		
3	Nylon	0.1	HNO <sub>3</sub>	5	160	18	4	Add a little HF if containing fiberglass	Clear
					190	26	4		
4	PA	0.1	HNO <sub>3</sub>	4	160	18	4	Add a little HF	Clear
					190	25	4		
5	Plastic (PVC)	0.2	HNO <sub>3</sub> HCl	4.5 1.5	150	18	4	Add a little HF if containing fiberglass	Clear
					180	22	4		
					210	30	5		
5	Plastic (ABS)	0.1	HNO <sub>3</sub> HF	4 1	160	18	4		Clear
					190	25	4		
					220	30	5		
6	Silicone rubber	0.1	HNO <sub>3</sub> HF	4 1	160	18	4		Clear
					190	22	4		
					220	38	5		

Examples of Digestion Methods (Reference)									
N o	Sample	Weigh t (g)	Solven t	Volume(mL )	Tem (°C)	Pressur e (atm)	Time (min )	eNo t	Results
Environmental samples									
1	Soil (Total)	0.2	HNO <sub>3</sub> HCl HF	1 3 1	140	10	2		Clear
					170	18	2		
					190	25	2		
					220	30	8		
2	Soil(Leaching)	0.2	HNO <sub>3</sub> HCl	1 3	160	12	4		Need to filter sedimen t
					190	20	5		
3	Surface water	10mL	HNO <sub>3</sub>	5	120	10	2		Clear
					150	15	6		
4	Power plant Mud sediment	0.2	HNO <sub>3</sub> HF HCl	4 1 2	140	5	2		Clear
					180	15	2		
					220	22	6		
5	River mud	0.1	HNO <sub>3</sub> HF H <sub>2</sub> O <sub>2</sub>	3 1 1	160	15	2		Clear
					190	18	2		
					210	22	6		
6	Fly ash	0.1	HNO <sub>3</sub> HCl HF	3 1 0.5	150	10	2		Clear
					170	15	2		
					190	22	2		
					210	30	6		
Inorganic samples									
1	Silica	0.1	HF HNO <sub>3</sub>	3 2	160	15	4		Clear
					190	20	8		
2	High purity quartz	0.2	HF	4	160	5	4		Clear
					190	10	4		
					220	15	5		
3	Ash	0.1	HNO <sub>3</sub> HCl HF	3 1.5 1	150	5	4		Yellow Clear
					170	10	3		
					190	15	3		
					210	24	5		



Examples of Digestion Methods (Reference)									
No	Sample	Weight (g)	Solvent	Volume (mL)	Temp (°C)	Pressure (atm)	Time (min)	Note	Results
4	Slag	0.1	HNO <sub>3</sub> HCl	1 3	150	15	3		Yellow Clear
					190	20	3		
					220	26	8		
5	Coal	0.1	HNO <sub>3</sub> HF	3 0.5	160	15	4		Clear
					190	20	4		
					210	25	6		
6	Zeolite	0.1	HNO <sub>3</sub> HF	4 0.5	140	8	4		Clear
					170	15	3		
					200	20	3		
					210	25	6		
7	Scheelite	0.1	HNO <sub>3</sub> HF	4 1	160	5	4		Clear
					190	10	4		
					210	15	5		
8	Iron ore	0.1	HNO <sub>3</sub> HCl HF	3 1 1	140	10	3		Clear
					170	15	3		
					200	20	3		
					220	25	5		
9	Beryl	0.1	HCl HF	4 1	140	10	4		Clear
					180	18	4		
					220	28	5		
10	Ilmenite	0.2	HCl HNO <sub>3</sub> HF	3 1 1	140	10	4		Clear
					180	18	4		
					220	28	15		
11	Chrome ore	0.1	HNO <sub>3</sub> HF	3 1	140	10	3		Clear
					170	15	3		
					200	20	3		
					220	25	5		
12	Magnesium refractories	0.1	HNO <sub>3</sub> HCl H <sub>2</sub> O <sub>2</sub>	2 2 1	140	8	3		Yellow Clear
					170	12	3		
					200	18	3		
					220	25	5		

Examples of Digestion Methods (Reference)									
No	Sample	Weight (g)	Solvent	Volume (mL)	Temp. (°C)	Pressure (atm)	Time (min)	Note	Results
13	Cerium dioxide	0.1	HNO <sub>3</sub> HCl	1 3	140	8	3		Clear
					170	12	3		
					200	18	3		
					220	24	5		
14	Alloy steel	0.1	HNO <sub>3</sub> HCl	1 3	150	10	4	Keep it for 10 min	Clear
					180	15	5		
15	Aluminum	0.1	HNO <sub>3</sub> HCl HF	1 3 0.5	150	5	2	Keep it for 10 min	Clear
					180	10	2		
					210	16	6		
16	Co-based alloys	0.2	HNO <sub>3</sub> HCl HF	2 4 0.5	150	10	4		Clear
					190	18	4		
					220	25	10		
17	High purity aluminum	0.2	HCl	4	150	10	4		Clear
					190	16	4		
					220	20	6		
18	Coating Toner	0.2	HNO <sub>3</sub> HF	4 1	150	10	4		Clear
					190	16	4		
					220	20	6		

\* Predigest for 15 min if react violently after adding acid until reaction slow down, prior to closed microwave digestion.

\* If too much organics in the sample or large amount of sample need to be digestion at once, you can put the sample with acid added on electric heater for 20-30 min at low temperature around 140°C.

\* If some silicate in the sample, a little HF (0.5-1.0 mL) may be added. After digestion, the vessels can be put on electric heater to vaporize acid. Add a little HClO<sub>4</sub> for vaporize acid till white mist appear. We don't suggest to vaporize HF, but adding 1:6 saturated boric acid to neutralize HF instead if volatile elements, such as As, Hg are need to be tested.

\* The digestion methods above are just for reference only. They may be applied in similar samples; But the digestion methods may be different because the compositions might be different even for the same kind of samples. Customers can adjust the methods according to the actual.

