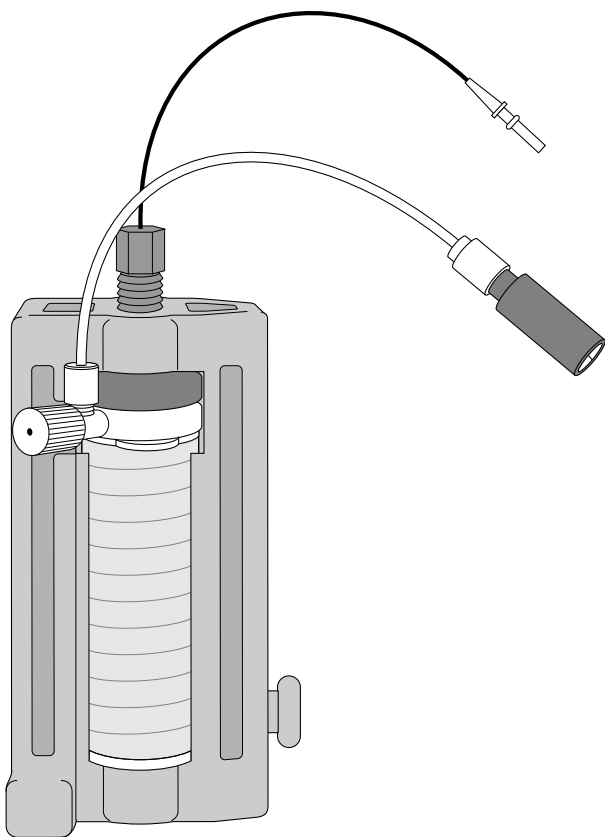


HP-500 Plus, XP-1500 Plus & OMNI

Vessel Accessory Sets & Autovent Options



Instructions for Use

CEM

600493
Rev. 6
12/01

Description

The HP-500 Plus and XP-1500 Plus High Pressure Digestion Vessel Accessory Sets permit simultaneous processing of up to 12 (XP-1500 Plus) or 14 (HP-500 Plus) pressure and temperature microwave digestion samples inside CEM MARS 5 microwave systems.

The vessel sets consist of a chemically resistant inner liner and cover to contain and isolate a sample solution from an advanced composite-reinforced pressure sleeve. All the materials used in the vessel's construction are transparent to microwaves so that liquid samples can absorb the maximum amount of incident microwave energy.

Specifications

	HP-500 Plus	XP-1500 Plus
Vessel Volume:	100mL (liner)	100mL (liner)
Liner Weight:	95 g	95 g
Maximum Control Pressure and Temperature:	350 psi 210 C	800 psi 240 C
Maximum Organic Sample Size:	< 0.5 grams	<0.5 grams
Materials of Construction:		
Liner	Teflon® PFA	Teflon® TFM
Sleeve Assembly:	Advanced Composite	Advanced Composite
Cover:	Teflon® PFA	Teflon® PFA
Support Module	Polypropylene w/ TFM® Heat Shield	Polypropylene w/ TFM® Heat Shield
Module Screw:	Polypropylene	Polypropylene
Vent Fitting:	Teflon® PFA	Teflon® PFA
Ferrule Nuts:	Teflon® PFA	Teflon® PFA
Load Disk:	Ultem®	Ultem®

Note: The HP-500 Plus vessel and its design are protected under the following U.S. Patent Nos. – 5,427,741 and 5,369,034

Teflon is a registered trademark of the E. I. DuPont Company
TFM is a registered trademark of Hoechst

Safety Guidelines

Microwave sample preparation imposes a unique set of safety considerations beyond the basics of good laboratory practice. Guidelines for safe operation of HP-500 Plus and XP-1500 Plus vessels are presented below.

1. All vessel 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.
2. Ensure that the vessels are completely vented of built-up reaction gases and completely depressurized before removing liner and internal components from the polypropylene support module. Failure to do so could potentially result in ejection of acid and injury to the operator.
3. Never install more than one (1) safety membrane in either an HP-500 Plus or XP-1500 Plus.
4. **Never** attempt to digest samples larger than the recommended maximum (refer to specifications) if the organic content and composition of the sample is unknown. Unknown samples should always be predigested for a minimum of 15 minutes in an unsealed vessel without any heating prior to attempting closed vessel microwave digestion.
5. **Do not** heat concentrated base or salt solutions inside either type of vessels. Microwave heating of such solutions causes precipitation of salts and formation of crystal deposits which absorb microwave energy and may char into the vessel or electrically arc, leading to possible vessel failure.
6. **Do not** heat high boiling point acids (concentrated sulfuric or phosphoric acids) inside either type of vessels without using temperature control limited to the recommended maximum or damage may result.
7. HP-500 Plus vessels have a maximum recommended hold time of 30 minutes at 210 degrees C. XP-1500 Plus vessels have a maximum recommended hold time of 30 minutes at 240 degrees C. Hold times longer than 30 minutes at the maximum temperature or higher may result in softening of the bottom of the support module and may damage the vessel.
8. For digestions using high concentrations of HF in an acid mixture, use the CEM HF-Master™ Thermowell. The thermowell is made of an HF-resistant industrial gem material. A Teflon-coated thermowell may be used; however, the lifetime may decrease as HF concentration increases.

WARNING

Acid decomposition of certain chemical compounds or types of samples constitutes unreasonable, hazardous misuse of CEM microwave digestion systems and vessels. The classes of compounds listed below are unsuitable for closed vessel microwave digestion because they are highly reactive with oxidizing acids and/or may become nitrated and potentially explosive. Absence of a particular chemical compound from this list does not imply microwave acid decomposition of such a sample is safe under all conditions. CEM will not be responsible for damage to equipment and facilities or personal injuries resulting from microwave digestion of such compounds/samples.

Compounds Unsuitable for Closed Vessel Microwave Digestion

- Explosives (TNT, Nitrocellulose, etc.)
- Propellants (Hydrazine, Ammonium Perchlorate, etc.)
- Pyrophoric chemicals
- Hypergolic mixtures (Nitric Acid and Phenol, Nitric Acid and Triethylamine, Nitric Acid and Acetone, etc.)
- Animal Fats (Esters of glycerol capable of nitration and the formation of nitroglycerin or other nitrated organic compounds)
- Aviation Fuels (JP-1, etc.)
- Acetylides
- Acrolein
- Glycols (Ethylene Glycol, Propylene Glycol, etc.)
- Perchlorates (Ammonium, Potassium, etc.)
- Ethers (Cellosolve - Ethylene Glycol Phenyl ether, etc.)
- Lacquers
- Alkanes (Butane, Hexane, etc.)
- Ketones (Acetone, Methyl Ethyl Ketone, etc.)

HP-500 Plus and XP-1500 Plus Standard Vessel

Assembly Procedures

1. If safety membranes are to be used, unscrew and remove the Teflon vent fitting from the cover of a standard (non-control) vessel. Using the safety membrane installation tool, remove a safety membrane from the storage case by gently pressing the black button and placing the suction tip of the tool on a safety membrane. Place the membrane into the fitting and press firmly to release it into the vent fitting. Always use the proper safety membrane. Ensure that the membrane is lying flat on the bottom of the vent fitting. Failure to seat the membrane in a standard vessel will result in leakage. On the control vessel, failure to seat the membrane properly will result in a faulty pressure reading.

CAUTION

Never install more than one (1) safety membrane in the vent fitting.

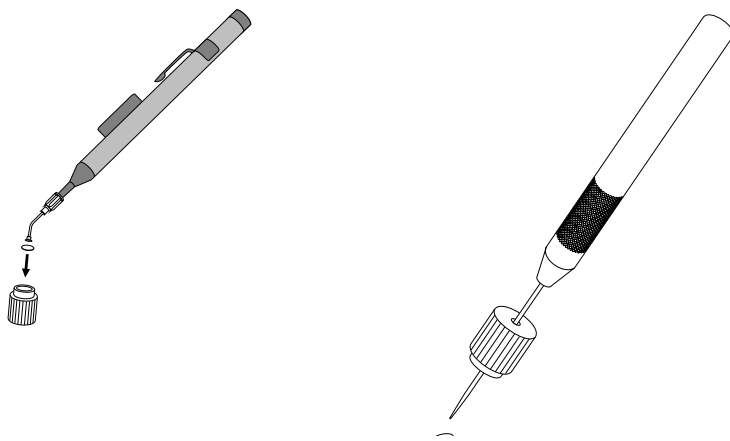


Figure 1. Insertion & Removal of Rupture Membrane

2. Weigh the sample and place it into the vessel liner. The sample must be placed in the bottom of the liner so that it will be completely covered by acid. The side walls of the liner must not have sample deposits on them.
3. Add the acid solution to the vessel liner, immersing the sample. Visually observe for a reaction. If a reaction occurs, allow the reaction to subside completely before capping the vessel.

CAUTION

A predigestion step must be performed on samples of unknown composition or those suspected of containing easily oxidized and highly reactive compounds such as alcohols, ketones and glycols. Predigestion is performed without microwave heating for a minimum of 15 minutes in an open, unsealed vessel.

4. Place the vessel cover on top of the vessel liner. Place the brown load disk on the cover with the side with the circular depression facing upward.

CAUTION

Do not use an HP-500 Plus or XP-1500 Plus vessel without a heat shield in the bottom of the vessel support module and a sleeve between the liner and support module.

5. Insert the liner into a clean, dry sleeve. Install the completed vessel assembly into the support module.
6. Screw the Teflon vent fitting onto the threaded stem of the cover (figure 2). Rotate the vessel so that the vent fitting is positioned toward the front of the support module (end with label). With the module screw centered in the recessed area in the control cover, tighten the support screw finger tight. Using the torquing tool, tighten the screw an additional half turn.

Note: If using a preset torque wrench, ensure that it is set at 5 ft. lbs.

7. Repeat steps 1 through 7 for remaining standard sample vessels.

Note: If preferable, the turntable can be installed into the instrument cavity prior to vessel installation.

8. Place each vessel into the turntable by inserting the bullet-shaped locking tab of the support module into a slot in the raised center of the turntable. If fewer than 14 vessels (HP-500 Plus) or 12 vessels (XP-1500 Plus) are being used, arrange the vessels symmetrically. Place the turntable into the microwave system.

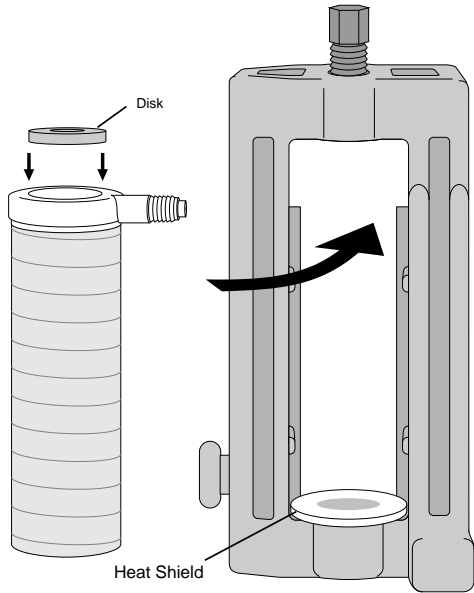
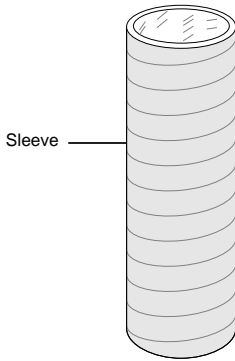
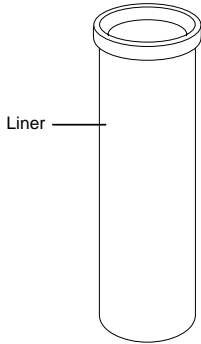
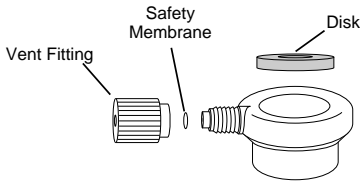


Figure 3. Insertion of Standard Vessel into Support Module

Figure 2. Assembly of Standard Vessel

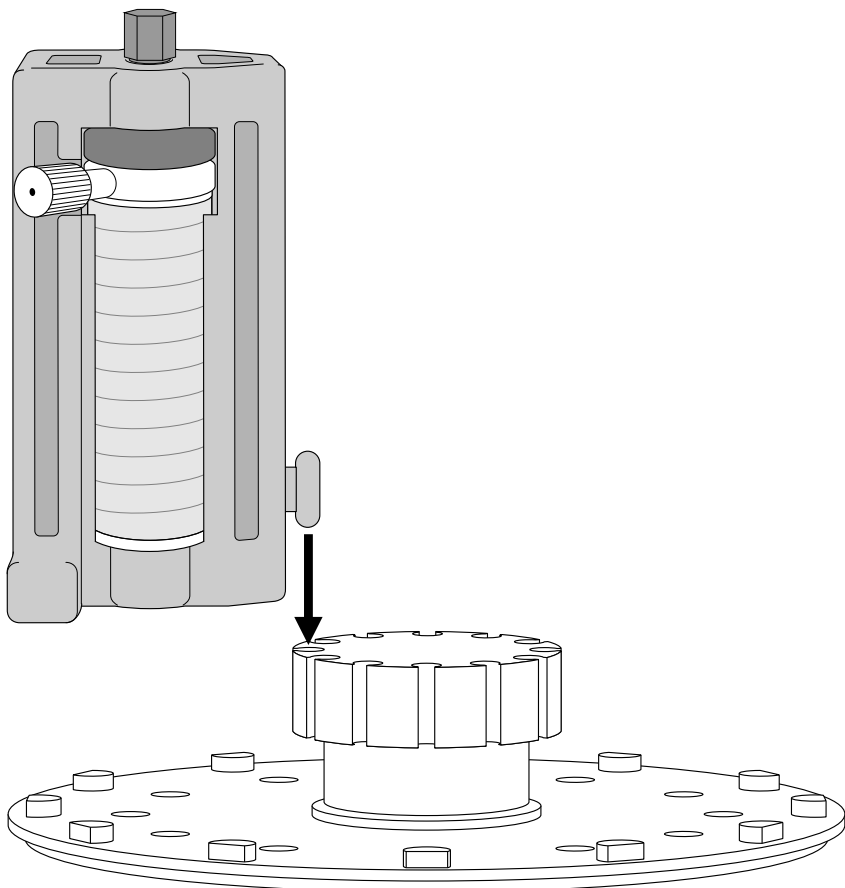


Figure 4. Insertion of Completed Vessel Module onto Turntable

HP-500 Plus and XP-1500 Plus Control Vessel

Assembly and Connection Procedures

1. Insert the open end of the thermowell (recommended CEM HF-Master) into the locking ferrule nut and slide it through the temperature port in the cover. Tighten the locking ferrule nut finger tight. *For the XP-1500 Plus only, also slide the secondary ferrule nut over the thermowell on the underside of the cover and tighten finger tight.* Verify that the thermowell is locked securely by holding the cover and slightly pulling on the thermowell. The thermowell should not feel loose.
2. If safety membranes are to be used, install a safety membrane in the Teflon vent fitting as outlined in step 2 of standard vessel instructions and as shown in figure 1.

CAUTION

Do not install and tighten the thermowell ferrule nut in the cover assembly prior to installation of the thermowell. If the thermowell ferrule nut is tightened without the thermowell being installed, the opening in the cover will be distorted, making it difficult or impossible to install the thermowell for future use. Also, the distortion may cause leakage.

3. Weigh and place the sample into the vessel liner, ensuring that the acid covers the sample.
4. Place the vessel cover on top of the vessel liner. Place the Teflon spacer containing the brown load disk on the control cover with the side of the spacer with the circular depression facing upward. If using temperature control, the spacer and load disk should have a hole in the center.
5. Thread the Teflon vent fitting onto the vent stem of the vessel cover as shown in figure 5. Tighten the pressure tubing leading from the ESP-1500 Plus to the pressure port on the control cover. Rotate the vessel so that the vent fitting is positioned toward the front of the support module (end with label).

CAUTION

The ESP-1500 Plus and the EST-300 Plus must be assembled to and removed from the control vessel prior to installation of the turntable and vessels into the instrument cavity.

Note: If using a preset torque wrench, ensure that it is set at 5 ft. lbs.

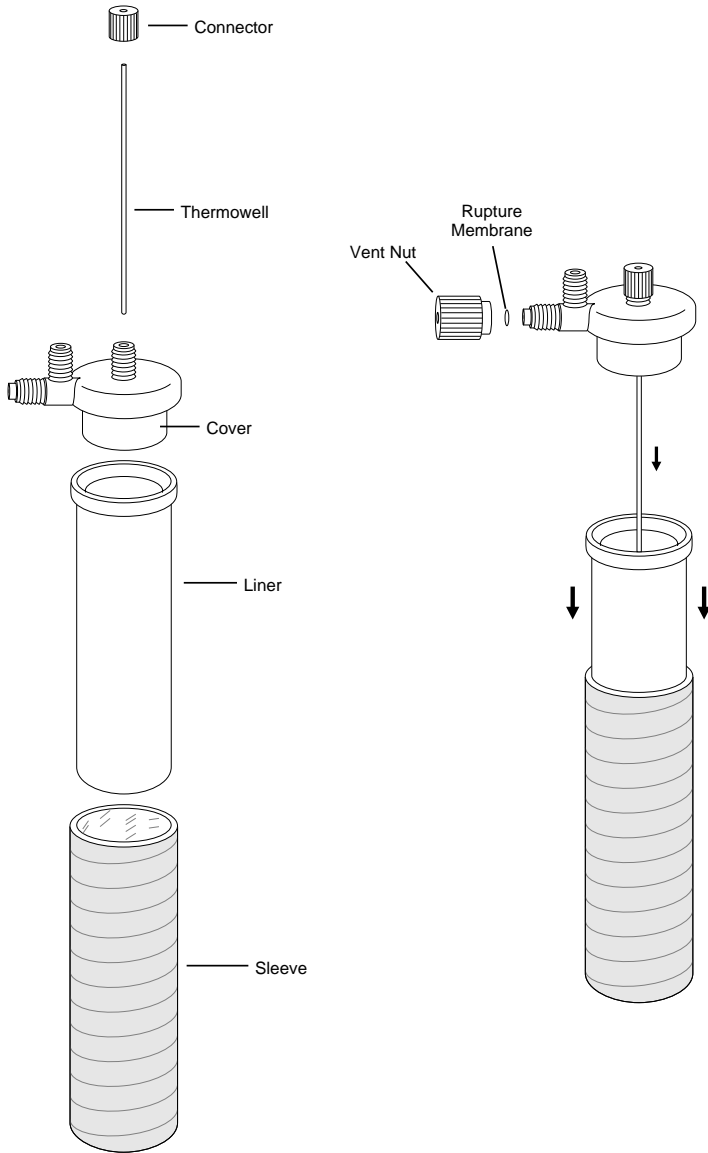


Figure 5. Assembly of Control Vessel for Pressure/Temperature Control

6. If using temperature control, the module screw will have a small hole in the center. Slide the probe holder onto and up the EST-300 Plus probe approximately 6 - 7 inches. Insert the EST-300 Plus through the module screw. Slide the probe holder down the EST-300 Plus and gently press it into the hole in the center of the module screw.

- Place the control vessel in the turntable in the position labeled "Control Vessel." Install the vessel retaining ring on the vessels with the notch of the retaining ring resting on the control vessel. Install the turntable into the instrument cavity, aligning the straight line on the label in the center of the turntable with the flat edge of the turntable lug. Press the turntable key to rotate the turntable. The control vessel should be positioned in the back left hand corner of the instrument. Connect the ESP-1500 Plus pressure controller to the connector on the right side of the instrument cavity (facing instrument) by rotating the ESP-1500 Plus while gently pushing it into the connector port until it slips into the correct position. This rotation is to align the ESP-1500 Plus and the connector port. Once the connector is properly aligned, push the ESP-1500 Plus into the connector port until the polypropylene guard is fully seated against the connector port. Connect the EST-300 Plus into the connector in the top of the cavity, ensuring that it does not cross the EST-1500 Plus cable. Permit the turntable to rotate 2 or 3 times to ensure that the pressure sensing tubing and temperature probe do not become entangled.

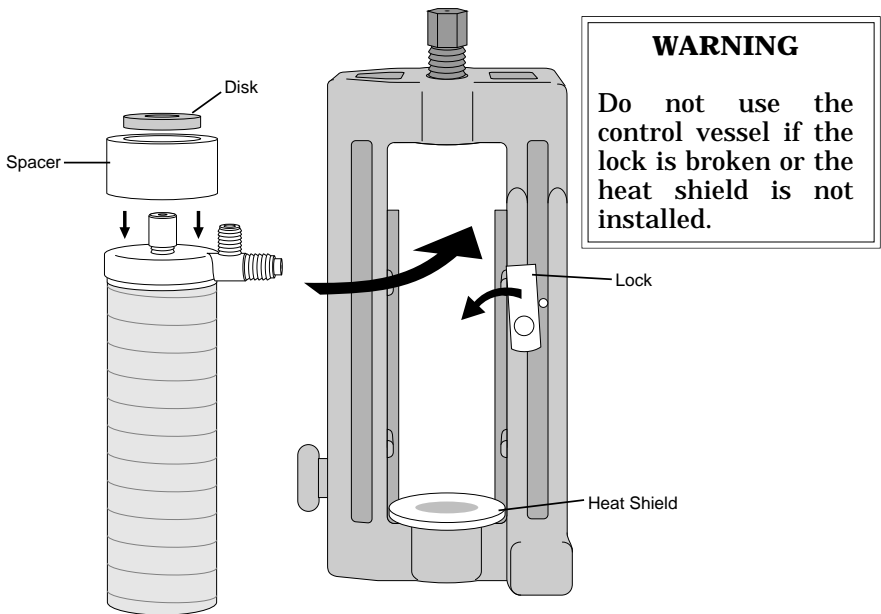


Figure 6. Assembly of Control Vessel for Pressure/Temperature Control

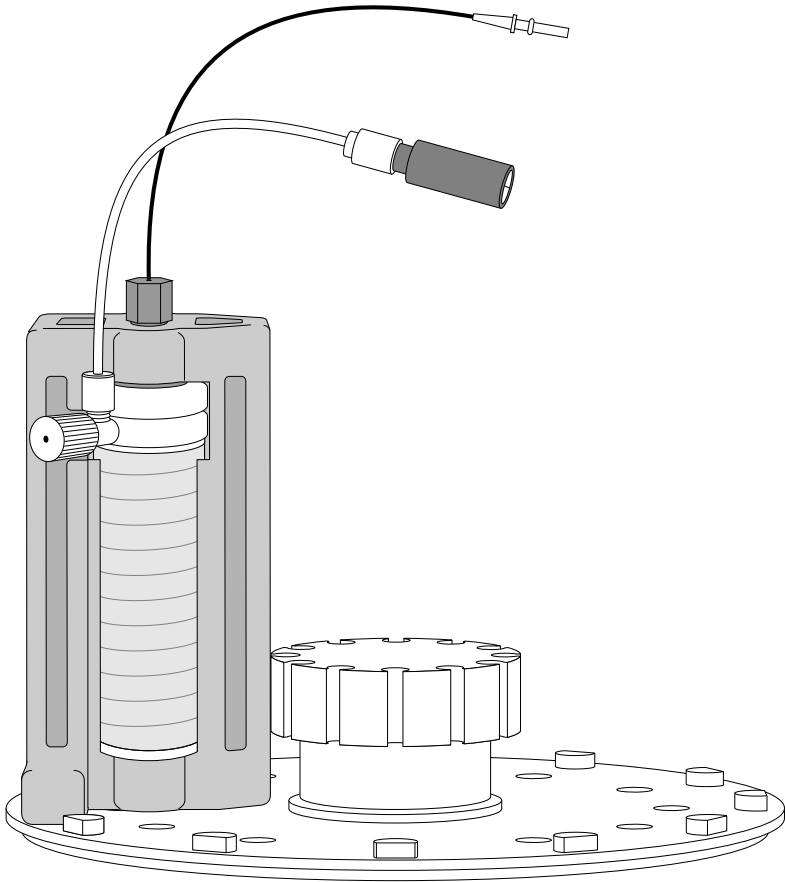


Figure 7. Pressure/Temperature Control Vessel with Insertion of Temperature and Pressure Sensor

Omni Vessel

The Omni vessel utilizes the XP-1500 Plus frame and liner, but has a specialized sleeve, cover and load disc.

Use of Omni Vessel

1. Assemble the Omni vessel in the manner outlined for the XP-1500 vessel utilizing the specialized sleeve, cover and load disc provided.
2. If using temperature and/or pressure control, assemble the Omni control vessel in the manner outlined for the XP-1500 Plus control vessel utilizing the specialized sleeve, cover and load disc provided.

3. Tighten the vessel with a preset torque wrench set at 5 ft. lbs.

WARNING

Omni vessel load discs are manufactured to ensure that they cannot be used with sealed XP-1500 vessels. Do Not attempt to use these parts because it could constitute a safety hazard.

Autovent Option for HP-500 Plus and XP-1500 Plus Vessels

Both the HP-500 Plus and XP-1500 Plus vessels can be converted to autovent vessels by replacing the vessel cover with the autovent cover. Autovent covers do not utilize safety membranes. They are designed to vent excess pressure.

Use of Optional Autovent Covers

1. Assemble the standard HP-500 Plus or XP-1500 Plus vessels as outlined in this manual with the exception of the covers. Replace the standard covers with the autovent covers (figure 8).
2. If using temperature and/or pressure control, assemble the HP-500 Plus or XP-1500 Plus control vessel as outlined in this manual with the exception of the cover. Replace the control cover with the autovent control cover (figure 9).
3. Tighten the vessels with a preset torque wrench set at 5 ft. lbs.
4. Refer to the setup instructions in the instrument operation manual and turn the ReactiGuard sensor off prior to performing a method with vessels assembled with the autovent option. Autoventing can activate the sensor.

CAUTION

The ReactiGuard sensor must be turned off prior to performing a sample analysis when using the autovent option with HP-500 Plus or XP-1500 Plus vessels.

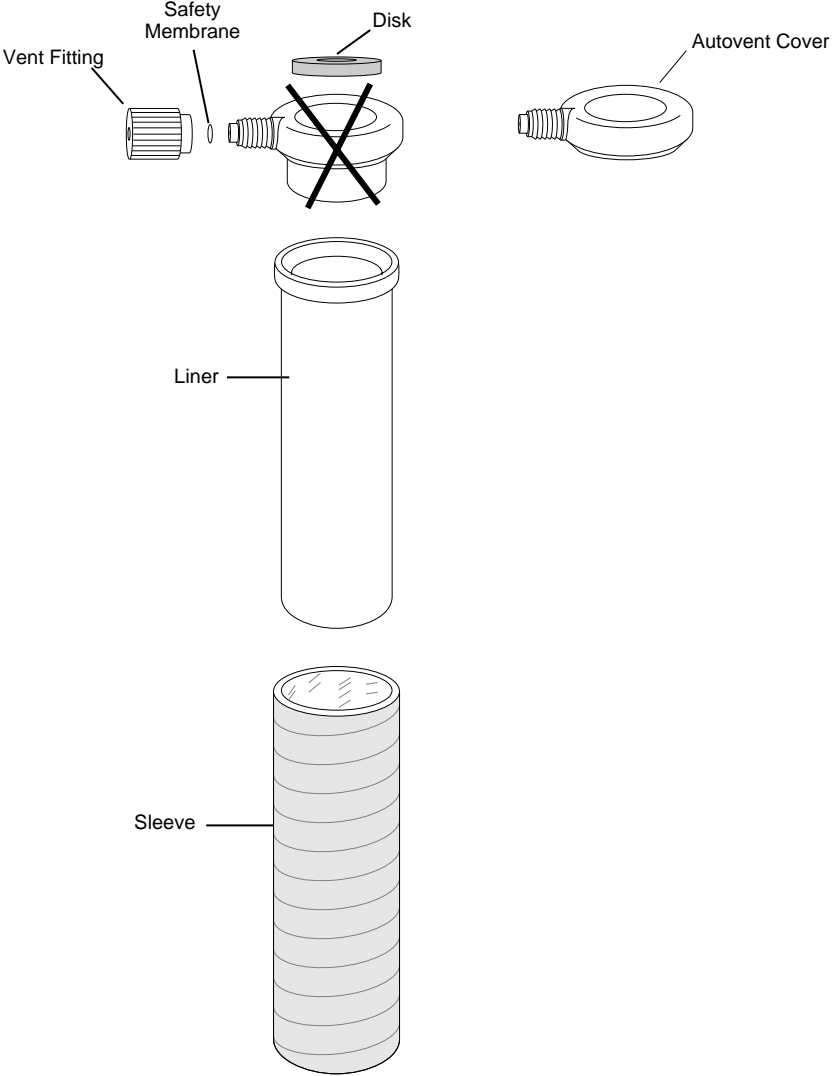


Figure 8. Assembly of Standard Vessel with Autovent Option

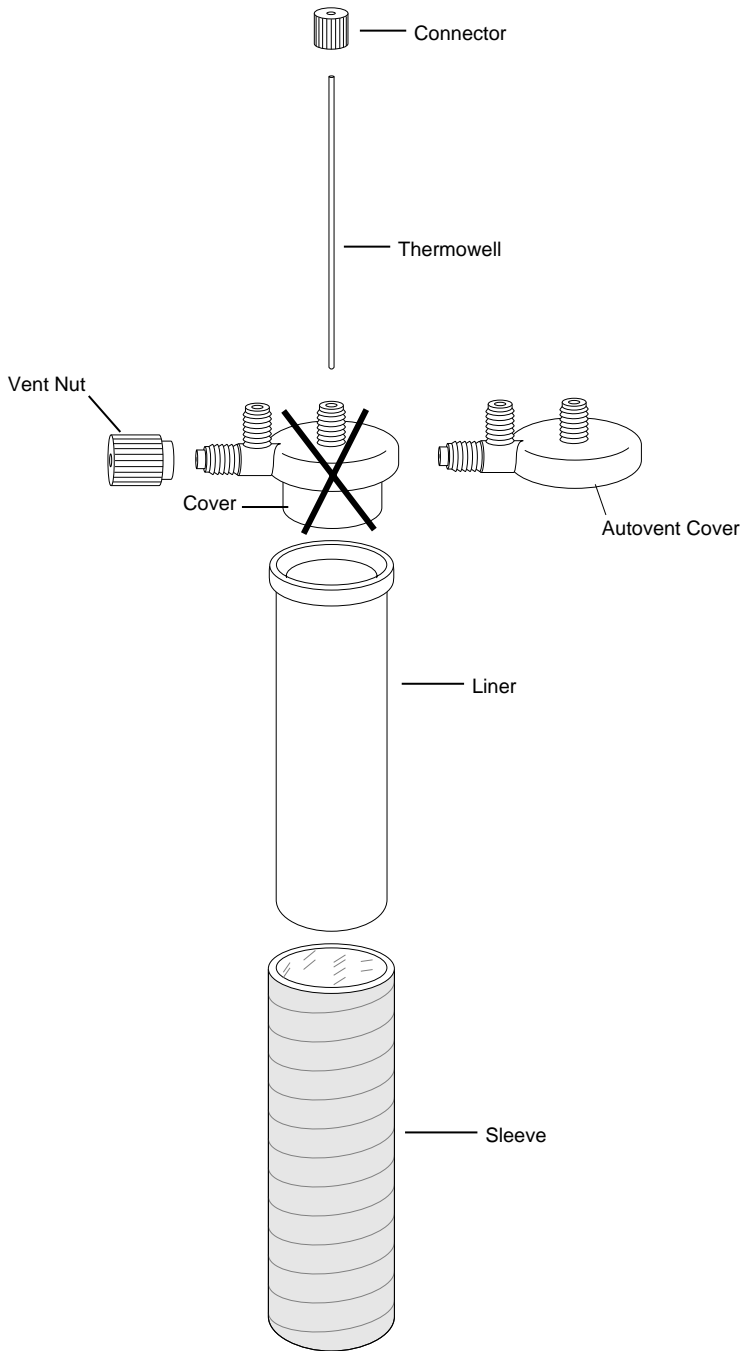


Figure 9. Assembly of Control Vessel with Autovent Option

Cooling Procedures

CAUTION

Do not immerse vessels in water for cooling. If vessels are immersed, they must be dried in an air oven for several hours prior to reuse.

The software of the MARS 5 microwave system includes a five-minute cooling step at the conclusion of each method. However, this cooling step will not return the vessels to room temperature conditions. CEM recommends at least 15 minutes cooling time for HP-500 Plus and XP-1500 Plus vessels.

Venting and Opening Procedures

WARNING

Always wear gloves, a lab coat and eye protection when handling/venting HP-500 Plus and XP-1500 Plus vessels.

1. Following completion of a microwave heating method, permit the vessels to cool until the pressure inside the control vessel drops below 200 psig (13.8 bar) and/or 100degrees C prior to venting.

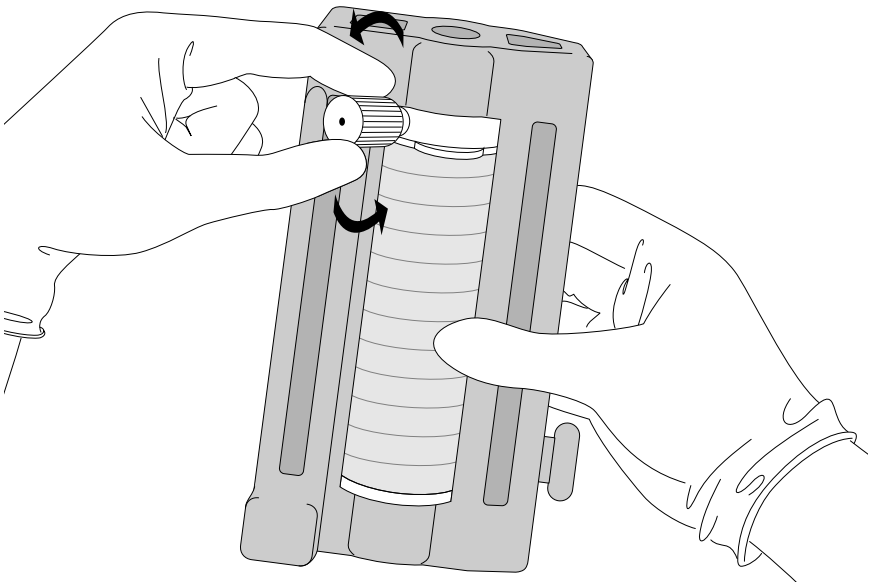


Figure 10. Venting an HP-500 Plus

WARNING

Ensure that the vessel is completely vented and depressurized prior to removing the liner and internal components from the polypropylene support module. Failure to do so could result in ejection of acid and injury to the operator.

2. Vent vessels by grasping the support module with one hand and loosening the Teflon vent fitting by slowly turning it in a counterclockwise direction as illustrated in figure 10. A series of slow, partial turns of the fitting will permit decomposition gases and acid vapor to escape through the vent fitting. Do not rapidly loosen the vent fitting by multiple turns. This will permit gases to exit down the threads of the fitting.

WARNING

Ensure that the vessel is completely vented and depressurized prior to removing the liner and internal components from the polypropylene support module. Failure to do so could result in ejection of acid and injury to the operator.

3. If applicable, remove the EST-300 Plus. Remove the vessel from the support frame. Carefully remove the sleeve. Then remove the cover of the vessel. The cover should be removable with only modest force required.

WARNING

Avoid tipping the vessel sideways to avoid spilling the contents of the vessel.

Cleaning and Inspection Procedures

Liner

These fluoropolymer materials soften at temperatures above 300 degrees C and should not be permitted to reach this temperature during digestion.

Do not permit prolonged contact of the Ultem® disk with acid. Disks should be rinsed thoroughly with water after each use or if contact with acid occurs.

Various procedures can be used to clean the liners. The method and rigor of cleaning will be determined by the particular level of analyte detection (% , ppm, ppb, and ppt).

Example: Place the acid mixture and volume of acid required for sample preparation in the liners. Assemble the vessels and place them in the instrument for sample preparation. Create a Cleaning Method as follows:

Stage - 1

% Power - 100

Pressure (psi) - 100

Ramp Time (min.) - 15:00

Temperature (Degrees C) - 200

Hold Time (min.) - 8:00

Load and perform the method to heat the acid. Permit the vessels to cool. Vent and remove the vessel covers.

Remove the acid from the vessels. Rinse the liners and covers with deionized water, and dry both parts thoroughly. For low level analysis, it may be necessary to repeat this process. For major element analysis, a detergent soak and rinsing may be adequate.

The user should determine the cleaning procedure which will provide adequate cleaning of vessels by analysis of a blank test prepared as outlined above in the Cleaning Method. Dilution and actual analysis of the blank as samples will help the user determine the most suitable cleaning procedure for its particular application(s).

The liner, cover and locking ferrule nut (control vessel only) can be damaged by excessive heat and pressure. Pressure and temperature control during method development will reduce the possibility of damage resulting from excessively high temperatures and pressures.

Support Module (Polypropylene)

A yellow-colored support module indicates acid degradation. If the surface of the support module becomes deep yellow in color, it should be replaced because it will be brittle and low in strength. Degradation can be the result of:

1. Spilling acid on the support module.
2. Not properly tightening the module screws.

Always keep the support module free of acid spills. The support module should be cleaned with a laboratory detergent and hot water, rinsed with water, and dried thoroughly after each use.

Advanced Composite Sleeve

Inspect the sleeve for any damage or distortion. The sleeve may be cleaned by wiping it with a damp cloth after each digestion. If peeling or other damage is observed, the sleeve should be replaced. Ensure that the sleeve and heat shield are dry prior to a digestion.

CAUTION

Do not soak sleeves in water. Trapped water can damage the composite layers during heating.

Vent Fitting and Safety Membrane (PFA Teflon, Fluoropolymer)

Safety membranes should be used only once, then discarded.

To clean vent fittings, soak them in hot water and detergent. Rinse them thoroughly with hot tap water. Permit them to dry completely prior to use. Incomplete drying of vent fittings can cause leakage when under pressure.

Ensuring Long Vessel Life

1. Follow the proper inspection and cleaning recommendations.
2. Optimize methods for a particular sample type, analyte, and method of analysis. Do not digest samples at temperatures and pressures greater than those required to provide good analytical results. Optimize time of digestion.
3. Keep the vessels clean. Unless cleaned promptly and thoroughly, all non-fluoropolymer parts of the vessel may be damaged by exposure to acids. All components should be inspected after each use for signs of excessive wear such as dark yellow staining, cracking, pitting, grooving or other signs of use-related stress.
4. Lifetime of vessel components cannot be guaranteed due to substantial variance of usage conditions and operator maintenance procedures. Inspect vessels for wear after each use. For best performance and safety, under normal usage conditions and with regular inspection and maintenance, components in direct contact with the sample and acid mixtures should be replaced after one hundred (100) microwave heating cycles. All other components should be inspected regularly for wear or damage, and should be replaced if signs such as cracking or heavy discoloration are observed.

Refer to the MARS 5 operation manual for instrument programming and operating instructions.

CEM

P.O. Box 200 • 3100 Smith Farm Rd.
Matthews, NC 28106-0200

Sales & Applications Hot Line: (800) 726-3331
Sales Fax: (704) 821-5185
Applications Fax: 821 -7894