



## Mars 6 Synthesis Research



The GlassChem20, EasyPrep™ Plus, EasyPrep™, and GreenChem™ Plus are the ONLY pressurized vessel sets recommended for synthetic research in a Mars 6. Open vessel research may also be performed in a round bottom flask.

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## Vessel Chart

The chart below gives an overview of the different vessels and will help determine which vessel set is right for your application. Additional information can be found in the operations manual for each vessel set (GlassChem20 PN 600788, EasyPrep™ Plus PN 600274, EasyPrep™ PN 600218, GreenChem™ Plus PN 601662, or Open Vessel PN 601155). Please contact CEM applications support with any questions or concerns.

	<b>GlassChem20</b>	<b>EasyPrep™ Plus</b>	<b>GreenChem™ Plus</b>	<b>Open Vessel</b>
				
<b>Minimum Ramp Time</b>	5 min	5 min – EasyPrep Plus 20 min – EasyPrep	5 min	5 min
<b>Maximum Number of Vessels</b>	24 or 36	12	14	1
<b>Vessel Volume</b>	20mL	100mL	100mL	250mL to 5L
<b>Maximum Working Volume</b>	14mL	75mL	75mL	70% of round bottom flask
<b>Minimum Working Volume</b>	3mL	20mL	20 mL	100mL
<b>Maximum Control Temperature</b>	180°C	300°C	200°C	300°C
<b>Maximum Operating Pressure</b>	200psi	800psi	200psi	N/A
<b>Temperature Control Type</b>	Fiber Optic probe	Fiber Optic probe or Fiber Optic Probe and IR (DuoTemp)	Fiber Optic probe	Fiber Optic probe
<b>Recommended Stir Bar</b>	Teflon coated Micro or “flea”	Teflon coated Micro or “flea”	Teflon coated Micro or “flea”	Teflon coated
<b>Vessel</b>	Pyrex	Teflon® TFM 1700	Pyrex or Teflon	Standard Round Bottom
<b>Thermowell</b>	Sapphire	Sapphire (EasyPrep Plus) Teflon® TFM (EasyPrep)	Sapphire	Glass or PFA
<b>Ideal Chemistry</b>	Organic, inorganic, or teaching laboratories	EasyPrep Plus: Inorganic and materials research labs EasyPrep: Alkaline solution	Organic, inorganic, or materials research laboratories, providing a larger reaction scale in fewer vessels than the GlassChem set	Organic, inorganic, materials research or teaching laboratories

## General Microwave Tips

- Cover all solids in the reaction vessel with liquid
  - Metal catalysts can be used, but ensure they are wetted or in solution
  - Finely divided (powder) metals are safer than bulk metals for heterogeneous catalysis
  - Solid State Reactions should not be performed in a Mars 6
- Never exceed the maximum working volume of the vessel
- Always use a stir bar that adequately mixes the reaction contents in the microwave
- When transitioning a conventional, open vessel reaction to a closed vessel reaction:
  - Increase reaction temperature 25°C above the highest boiling point solvent in vessel

## When to Exercise Caution

- Anything that would be a concern conventionally, regardless of reaction size
- High concentrations of acids, bases, or salts
  - Includes other ionic or very polar species
  - Bases react more readily in a microwave
  - Generally, 10% or less is recommended for glass vessels
- Gases formed during the reaction
  - Increase headspace (empty volume in vessel) to accommodate the generated gas

## How to Cautiously Microwave

- Start with the recommended power based on the number and type of vessels used
- Watch the reaction during the ramp and after the first few minutes of the hold
- Always prepare the Control (vessel with fiber optic probe) the same as the Standard vessels
  - **The Control vessel must contain the same solvent and reagents as used in the Standard vessels**
- Always distribute multiple vessels evenly around the turntable

## Solvent Chart

Microwave Absorbance	Solvent
High	DMSO, EtOH, MeOH, Propanols, Nitrobenzene, Formic Acid, Ethylene Glycol
Medium	Water, DMF, NMP, Butanols, Acetonitrile, HMPA, Methyl Ethyl Ketone, Acetone and other ketones, <i>o</i> -Dichlorobenzene, 1,2-Dichloroethane, 2-Methoxyethanol, Acetic Acid, Trifluoroacetic Acid
Low	Chloroform, Dichloromethane, Carbon Tetrachloride, 1,4-Dioxane, THF, Glyme, and other ethers, Ethyl Acetate, Pyridine, Triethylamine, Toluene, Benzene, Chlorobenzene, Xylenes, Pentane, Hexane and other hydrocarbons

### NOTE

Always use the Ramp-to-Temperature control type and allow for sufficient time to heat to temperature, especially for low absorbing solvents. This will help prolong the life of the magnetron and microwave unit.

## GlassChem20 Vessels

Refer to the GlassChem20 manual (PN 600788) for complete instructions.

### Power Guidelines (MARS 6):

1-4 vessels: 500W      5-10 vessels: 1000W      11-24 vessels: 1800W

### Minimum Cavity Load:

50mL with low & medium microwave absorbing solvents

10mL with high microwave absorbing solvents



### WARNING

Low absorbing solvents (see solvent chart on page 4) should be microwaved with caution as they do not heat well. Do not microwave low absorbing solvents without adding proper amounts of reagents. Please contact applications support for questions.

### Vessel & Turntable Setup:

1. Following the appropriate safety precautions for the reagents used and guidelines in the charts above, place all the reaction components and Teflon® coated magnetic stir bar into the GlassChem 20 vessel.

**All vessels must contain the exact amount of reaction components and the total volume must meet the minimum cavity load.**

2. Place the vent plug inside the top of the reaction vessel. Place the vessel top on the reaction vessel and tighten finger tight. For the control vessel, place the fully assembled control top with an installed control plug and thermowell onto the reaction vessel and tighten finger tight.



Standard Vessel



Control Vessel



Torque Tool

3. Using the preset torque tool, tighten the top until an audible click can be heard from the tool.
4. Insert the composite sleeves into the receptacles of the turntable. Place the control vessel in position "1" and evenly space the remaining vessels throughout the turntable, meeting the minimum cavity load outlined above. (See Example spacing below)



● = Empty Position    ● = Standard Vessel    ● = Control Vessel    ● = Peg

- Place the turntable shield on the turntable. Slide the four pins into place through the four pegs.



- Place the turntable into the MARS 6 cavity. Ensure that the turntable is properly seated on the drive lug.
- Insert the fiber optic probe by sliding the probe into the thermowell nut and close the door of the MARS 6 instrument.
- Select or create the desired method. For instructions on method creation, refer to the MARS 6 Operation Manual (PN 600284).
- Press **Start** to begin the method.

### Vessel Removal:

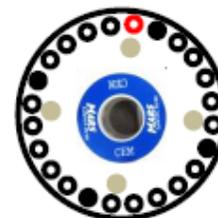
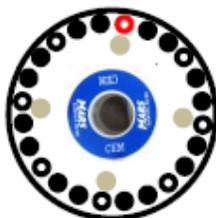
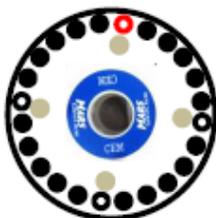
- Following completion of a microwave heating method, allow the vessels to cool below 50 °C.
- Remove the fiber optic probe from the control vessel. Then remove the turntable from the microwave cavity; followed by the turntable shield.
- Open the vessels slowly with the top pointed toward the back of a fume hood. Do not rapidly loosen the vessel top to prevent permitting gases to escape quickly, resulting in potential injury.
- Perform the reaction work-up.

### Break-in Procedure:

#### NOTE

The break-in procedure is required only for newly acquired vessels or vessel parts.

- Place 10 mL of water and a Teflon® coated stir bar into each of the vessels.
- Place the vent plugs on each of the vessels followed by the vessel tops. Tighten the vessel tops with the preset torque tool until an audible click can be heard from the tool. For the control vessel, place the fully assembled control top with an installed control plug and thermowell onto the reaction vessel and tighten finger tight. Using the preset torque tool, tighten the top until an audible click can be heard from the tool. (Refer to the instructions for properly assembling reaction vessels.)
- If 23 vessels or fewer are being used, evenly space the vessels throughout the turntable.



- Heat the reaction vessels to 150 °C with a 5-minute ramp time and hold at this temperature for 5 minutes.
- Allow the vessels to cool to approximately 70 °C and loosen the tops. Using the preset torque tool, reseal the vessel tops while the vessels are still warm.
- Reheat the vessels to 150 °C as in step 4.
- If the vessels leak during the second heating, repeat the procedure in step 5.

Once the vessel tops have been broken in, the vessels are ready for regular use.

# EasyPrep™ & EasyPrep™ Plus Vessels

Refer to the EasyPrep™ Plus manual (PN 600274) or the EasyPrep™ manual (PN 600218) for complete instructions.

## Power Guidelines (MARS 6):

1-3 vessels: 400W    4-6 vessels: 800W    7-9 vessels: 1200W    10-12 vessels: 1800W

## Minimum Cavity Load:

50mL with medium microwave absorbing solvents

20mL with high microwave absorbing solvents



## WARNING

Low absorbing solvents (see solvent chart on page 4) should be microwaved with caution as they do not heat well. Do not microwave low absorbing solvents without adding proper amounts of reagents. Please contact applications support for questions.

## Vessel & Turntable Setup:

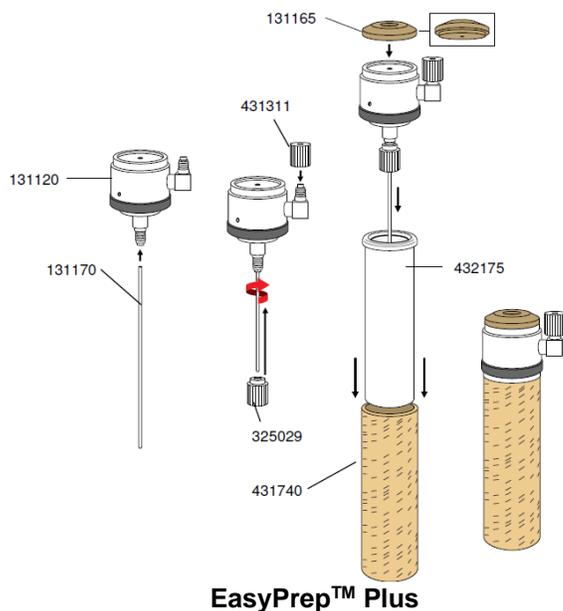
1. Following the appropriate safety precautions for the reagents used and guidelines in the charts above, place all solids and Teflon® coated magnetic stir bar into the vessel. The reagents must be added to the bottom of the liner so that they will be completely covered by solvent. Add the solvent to the vessel.

**All vessels must contain the same amount of reaction components and the total volume must meet the minimum cavity load.**

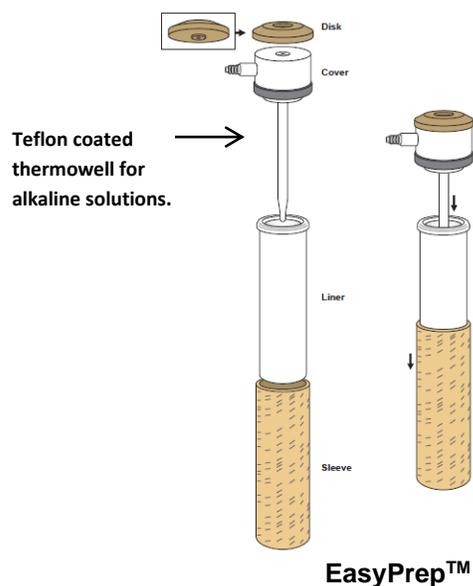
2. Place the reaction vessel inside the composite sleeve. Place the seal cover followed by the brown load disk on the top of the reaction vessel.

## NOTE

The thicker seal cover and brown load disk with the hole in the center are required for the control vessel.



EasyPrep™ Plus



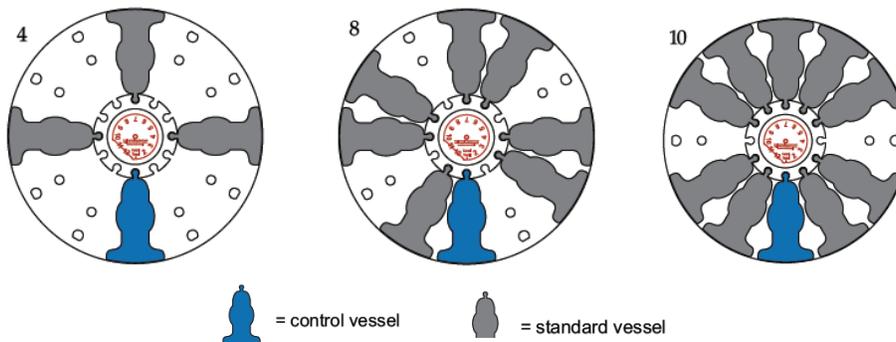
EasyPrep™

3. Place the assembled vessel inside the vessel frame. The larger vessel frame is required for the control vessel.

#### NOTE

The EasyPrep™ and EasyPrep™ Plus control vessel parts should not be interchanged.

- For the control vessel, connect the ESP-1500 Plus pressure controller to the pressure port of the control vessel (if applicable). Screw the Teflon vent fitting onto the threaded stem of the cover, ensuring that a rupture membrane is in position.
- 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 preset torque tool, tighten the screw until an audible click is heard.
- Place the control vessel into the position labeled “Control Vessel”. Place the remaining vessels 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 12 vessels are being used, evenly arrange the vessels around the turntable. (See example spacing below)



- Place the turntable into the MARS 6 cavity. Ensure that the turntable is properly seated on the drive lug.
- If using pressure control, 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.
- Insert the fiber optic by sliding the probe into the thermowell nut and close the door of the MARS 6 instrument.
- Select or create the desired method. For instructions on method creation, refer to the MARS 6 Operation Manual (PN 600284).

#### NOTE

Be sure to select the correct vessel type (EasyPrep™ or EasyPrep™ Plus) when programming a *Classic* method.

- Press **Start** to begin the method.

### Vessel Removal:

- Following completion of a microwave heating method, allow the vessels to cool below 50 °C.
- Remove the fiber optic probe from the control vessel and the pressure controller if applicable. Then remove the turntable from the microwave cavity.
- Vent the control vessel by grasping the support module with one hand and loosening the Teflon vent fitting by slowly turning it counterclockwise. For the remaining vessels, open the vessels slowly with the top pointed toward the back of a fume hood. Do not rapidly loosen the vessel top to prevent permitting gases to escape quickly, resulting in potential injury.
- Loosen the support screw and remove the assembled vessel from the vessel frame. Remove the load disk and seal cover.
- Perform the reaction work-up.

## Break-in Procedure:

### NOTE

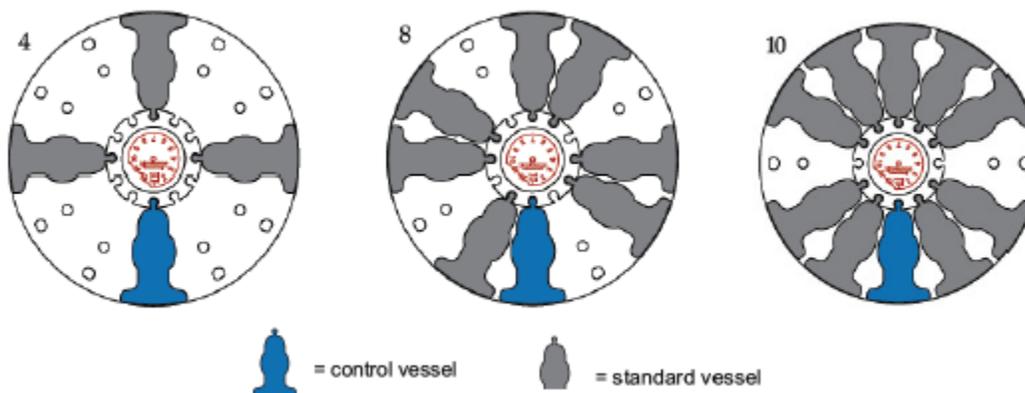
The break-in procedure is required only for newly acquired vessels or vessel parts.

1. Place 20 mL of water and a Teflon® coated stir bar into each of the vessels. If only running one EasyPrep vessel, use 50mL of water to meet the minimum cavity load requirement. Refer to the instructions above for properly assembling reaction vessels.
2. For the control vessel, connect the ESP-1500 Plus pressure controller to the pressure port of the control vessel (if applicable). Screw the Teflon vent fitting onto the threaded stem of the cover, ensuring that a rupture membrane is in position.

### NOTE

The EasyPrep™ and EasyPrep™ Plus control vessel parts should not be interchanged.

3. 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 preset torque tool, tighten the screw until an audible click is heard.
4. Place the control vessel into the position labeled “Control Vessel” and insert the fiber optic probe. If fewer than 12 vessels are being used, evenly arrange the vessels around the turn table.



5. Select and load the “EasyPrep Clean” method from the One Touch Directory.
6. With the door open, rotate the turntable. Make sure the pressure line and fiber optic probe are properly aligned.
7. Close door and run preprogrammed “EasyPrep Clean” method. Observe vessels for venting.
8. After completion of the program, allow vessels to cool for a minimum of 15 minutes. Unplug sensors and remove the turntable from MARS 6.
9. Carefully vent and open each vessel. Once cooled, discard the water and rinse vessel liners and covers with deionized water. Allow vessels to dry before performing next synthesis.

Once the vessel tops have been broken in, the vessels are ready for regular use.

# GreenChem™ Plus Vessels

Refer to the GreenChem™ Plus manual (PN 601662) for complete instructions.

## Power Guidelines (MARS 6):

1-3 vessels: 400W   4-6 vessels: 800W   7-9 vessels: 1200W   10-14 vessels: 1800W

## Minimum Cavity Load:

50mL with medium microwave absorbing solvents

20mL with high microwave absorbing solvents



## WARNING

Low absorbing solvents (see solvent chart on page 4) should be microwaved with caution as they do not heat well. Do not microwave low absorbing solvents without adding proper amounts of reagents. Please contact applications support for questions.

## Vessel & Turntable Setup:

1. Following the appropriate safety precautions for the reagents used and guidelines in the charts above, place all solids and Teflon® coated magnetic stir bar into the GreenChem vessel. The reagents must be added to the bottom of the liner so that it will be completely covered by solvent. Add the solvent to the vessel.  
**All vessels must contain the same amount of reaction components and the total volume must meet the minimum cavity load.**
2. Place the reaction vessel inside the composite sleeve. Place the seal cover followed by the brown load disk on the top of the reaction vessel.

## NOTE

The seal cover and brown load disk with the hole in the center is required for the control vessel.

3. Place the assembled vessel inside the vessel frame. For all vessels, screw the Teflon vent fitting onto the threaded stem of the cover, ensuring that a rupture membrane is in position.

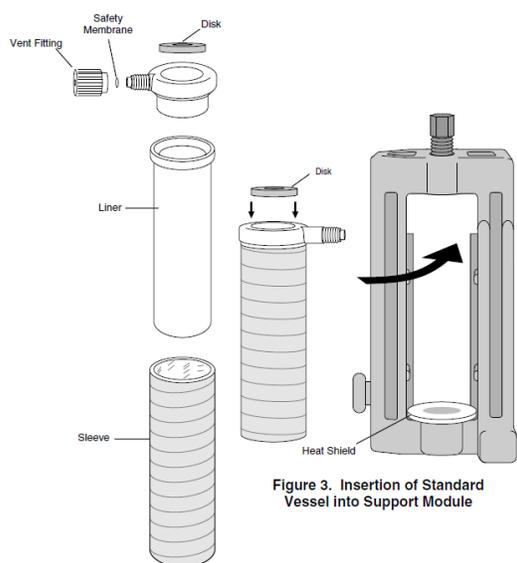
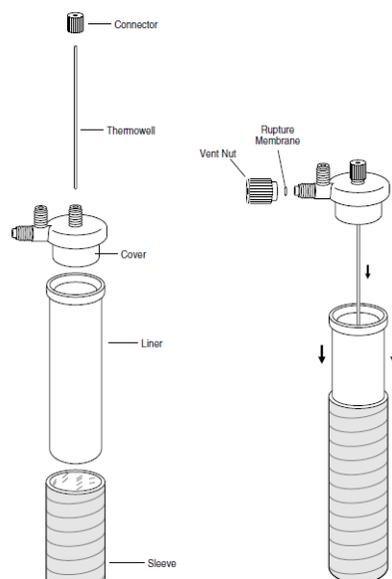


Figure 3. Insertion of Standard Vessel into Support Module

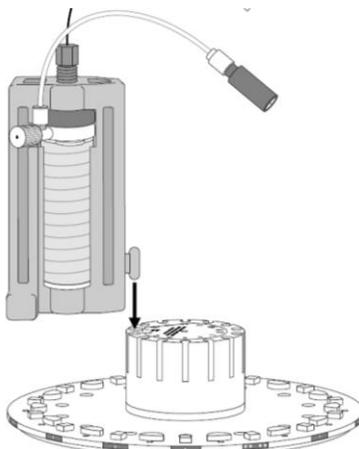
## Standard Vessel



## Control Vessel

4. Rotate the vessel so that the vent fitting is positioned toward the front of the support module (end with label). If using pressure control, tighten the pressure tubing leading from the ESP-1500 Plus to the pressure port on the cover of the control vessel. If not using pressure control, ensure that the plug nut is securely inserted into the pressure port on the cover of the control vessel.

- With the module screw centered in the recessed area in the control cover, tighten the support screw finger tight. Using the preset torque tool, tighten the screw until an audible click is heard.
- Place the control vessel into the position labeled "Control Vessel". Place the remaining vessels 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 are being used, evenly arrange the vessels around the turntable. Install the vessel retaining ring on the vessels with the notch of the retaining ring resting on the control vessel.



- Place the turntable into the MARS 6 cavity. Ensure that the turntable is properly seated on the drive lug.
- If using pressure control, 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.
- Insert the fiber optic by sliding the probe into the thermowell nut and close the door of the MARS 6 instrument.
- Select or create the desired method. For instructions on method creation, refer to the MARS 6 Operation Manual (PN 600284).
- Press **Start** to begin the method.

### Vessel Removal:

- Following completion of a microwave heating method, allow the vessels to cool below 50 °C.
- Remove the fiber optic probe from the control vessel and the pressure controller if applicable. Then remove the turntable from the microwave cavity; followed by the vessel retaining ring.
- Vent vessels by grasping the support module with one hand and loosening the Teflon vent fitting by slowly turning it counterclockwise.
- Loosen the support screw and remove the assembled vessel from the vessel frame. Remove the load disk and seal cover.
- Perform the reaction work-up.

### Break-in Procedure:

#### NOTE

The break-in procedure is required only for newly acquired vessels or vessel parts.

- Place 20 mL of water and a Teflon® coated stir bar into each of the vessels. If only running one EasyPrep vessel, use 50mL of water to meet the minimum cavity load requirement. Refer to the instructions above for properly assembling reaction vessels.
- Rotate each vessel so that the vent fitting is positioned toward the front of the support module (end with label).
- If using the pressure control, tighten the pressure tubing leading from the ESP-1500 Plus to the pressure port on the cover of the control vessel. If not using pressure control, ensure that the plug nut is securely inserted into the pressure port on the cover of the control vessel.
- With the module screw centered in the recessed area in the control cover, tighten the support screw finger tight. Using the preset torque tool, tighten the screw until an audible click is heard.
- Place the control vessel into the position labeled "Control Vessel". Place the remaining vessels 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 are being used, evenly arrange the vessels around the turn table before installing the retaining ring.

6. Create the GreenChem Break In method:
  - a. From the MARS 6 home screen, select “Classic Method”.
  - b. To create a method, select “+” at the top of the screen and type “GreenChem Break In” as the method name.
  - c. Touch “Select Control Type” and select “Ramp to Temperature” as the desired control type for the method.
  - d. Touch “Vessel Type” and select “Green Chem” as the desired vessel to be used with the method.
  - e. Select the scroll bar on “Temp Guard” and set to “Off”.
  - f. Select “Add/Edit Stages->” in the lower right corner of the screen.
  - g. Select “+” to add a stage to the method.
  - h. Enter the following method parameters:

<b>Ramp Time:</b>	<b>15:00</b>	<b>Power:</b>	<b>600</b>
<b>Hold Time:</b>	<b>10:00</b>	<b>Stirring:</b>	<b>OFF</b>
<b>Temperature:</b>	<b>180</b>		
7. Once all parameters are properly selected, select the disc icon to save the entered data. After the method has saved, the disk icon will become gray and the method name will be added to the top left corner of the screen.
8. Load the turntable and select and load the “GreenChem Break In” method from the Classic Method Directory.
9. With the door open, rotate the turntable. Make sure the pressure line and fiber optic probe are properly aligned.
10. Close door and run preprogrammed “GreenChem Break In” method. Observe vessels for venting.
11. After completion of the program, allow vessels to cool for a minimum of 15 minutes. Unplug sensors and remove the turntable from MARS 6.
12. Carefully vent and open each vessel. Once cooled, discard the water and rinse vessel liners and covers with deionized water. Allow vessels to dry before performing next synthesis.

Once the vessel tops have been broken in, the vessels are ready for regular use.

## Open Vessel

Refer to the Open Vessel manual (PN 601155) for complete instructions.

### Power Guidelines (MARS 6):

100mL-500mL volume: 500W    500mL-1.5L volume: 1000W

1.5L-3.5L volume: 1800W

### Minimum Cavity Load:

100mL with low, medium and high microwave absorbing solvent



### WARNING

Low absorbing solvents (see solvent chart on page 4) should be microwaved with caution as they do not heat well. Do not microwave low absorbing solvents without adding proper amounts of reagents. Please contact applications support for questions.

### Vessel Setup:

1. Place all reagents into a round-bottom flask (250mL or larger) with a stir bar.
2. Ensure the cavity is clean.
3. Place Teflon® disk flat in the cavity with the center hole placed over the drive lug. The additional riser may be required for smaller round bottom flasks.
4. Place the round bottom flask with the reaction solution and a stir bar into the cavity.
5. If required, attach the applicable extension glassware and a condenser.
6. Install the fiber optic probe.
  - a. If a condenser is not used, then feed the fiber optic probe directly into the round bottom flask. Ensure that the tip of the probe is submerged into solution, but not in the path of the stir bar.
  - b. If a condenser is used, then a glass adapter (PN 135025) and Teflon probe insert (PN 325216) may be needed in order to insert the fiber optic probe below the condenser. A double neck glass adapter is also available (PN 135040).



**Condenser Not Used**



**Condenser Used**

### NOTE

CEM recommends the use of a thermowell to protect the fiber optic probe.  
Glass thermowell (PN SP-1178) or plastic thermowell (PN SP-1550).

7. Close the door of the MARS 6 instrument.
8. Select or create the desired method. For instructions on method creation, refer to the MARS 6 Operation Manual (PN 601155).

### WARNING

Do not program temperature any higher than the boiling point of the lowest boiling solvent being utilized.

9. Press **Start** to begin the synthesis.

### Vessel Removal:

1. Wait until the cool-down cycle has terminated and the reaction has returned to a safe temperature. Do not remove the flask from the instrument cavity until the reaction has completed the cool-down cycle and the flask is at a safe temperature to remove it from the microwave cavity.

**WARNING**

To prevent the possibility of severe burns, wear insulated gloves and protective gear as outlined in the user's safety program.

2. Open the door of the MARS 6 instrument.
3. Remove the fiber optic probe.
4. If applicable, remove the condenser.
5. Remove the round bottom flask.
6. Perform the reaction work-up.