

CEM MARS5 Microwave

Updated August 5, 2009

Instrument instructions can be found at:

<http://academic.bowdoin.edu/chemistry/resources/instructions.shtml>

If you have any problems with the instrument or would like to get trained, please contact Celeste

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1. Protocol

- a. **Read instructions carefully before using instrument.** Reading the bold sentences in each category will tell you what you need to know to run the instrument.
 - i. Bullets are under the bold sentences when more detail is required.
 - ii. At the end of the instructions is a frequently asked questions/troubleshooting section.

2. Startup Procedure

- a. **Read precautions.**
 - i. Proper PPE (Personal Protective Equipment) is required—Safety goggles, neoprene gloves (near the instrument), and lab coat.
 - ii. 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.
 - iii. **Never** heat liquids in a sealed vessel or container that is not equipped with a pressure relief valve.
 - iv. **Never** attempt to digest samples larger than 0.5 grams if the organic content and composition of the sample are unknown.
 - v. Minimum volume for the MARS 5 cavity is 10 mL of acid or 50 mL of water.
 - vi. When working with an unknown sample, always perform a predigestion step in an unsealed, open vessel, allowing a minimum of 15 minutes time for the reaction of volatile or easily oxidized compounds to subside before sealing the vessel and microwave heating.
 - vii. Microwave heating of alkaline or salt solutions in open or closed vessels will concentrate these solutions, causing precipitation of salts and formation of crystal deposits on vessel walls. These crystal deposits will absorb microwave energy, causing localized heating which may char and damage vessel components, leading to possible failure.
 - viii. Do not heat high boiling point acids (concentrated sulfuric or phosphoric acids) inside microwave digestion vessels. These acids will heat to temperatures beyond the melting point of the polymeric materials of construction.
 - ix. There are several chemical compounds or types that are not suitable for closed vessel microwave digestion. Please consult the Operation Manual (page 3) for a list of them.

- x. Do not turn the instrument off within 15 seconds after operation with microwave power.
- b. **Turn on instrument.**
 - i. Wait until Main Menu (CEM Method Menu) is displayed before proceeding.
- c. **Enter sample information into microwave log sheet.**

3. Create Method

- a. **The “MILK” method is pre-loaded and sufficient for standard microwave digestions.**
- b. **New methods can be created in the User Directory only.**
- c. **Select “Edit/Create Method”.**
 - i. Using the right/left arrow keys on the four-way directional keypad, highlight “Edit/Create Method”.
 - ii. Press **Select** on the keypad.
- d. **Select “User Directory”.**
 - i. Use the right/left arrow keys to highlight “User Directory”.
 - ii. Press **Select** on the keypad.
- e. **Select “New Method”.**
 - i. Use the up/down arrow keys to highlight “New Method”.
 - ii. Press **Select** on the keypad.
- f. **Select vessel type.**
 - i. Our instrument uses OMNI/XP1500.
 - ii. Press **Select** on the keypad.
- g. **Select control type.**
 - i. Standard control is the one typically used.
- h. **Enter a method name.**
 - i. Highlight the letter or number for the new name.
 - ii. Press **Select** on the keypad.
 - iii. Continue until you have finished the method name.
 - iv. Press **Next** key on the keypad.
- i. **Enter method information.**
 - i. Highlight “Sample Description” and press the Select key. This will pull up an alphabet that will allow you to enter the name.
 - ii. Continue until all the method information is entered.
- j. **Enter method parameters.**
 - i. Either enter a value for the parameter or hit the Select key to pull up the options for that parameter.
 - ii. Continue until all the parameters are set.
 - iii. General guidelines for selecting wattage.

1. 1-2 vessels	300 watts
2. 3-5 vessels	600 watts
3. 6 or more vessels	1200 watts
- k. **When you are finished, the method you created will be loaded.**

4. Assemble Sample Vessels

- a. **Prepare the vessels in accordance with procedures outlined in the Vessel Manual.**
 - i. Use the following steps as guidelines, if you have never assembled a vessel, get trained before doing it based solely on these instructions.
 1. The liner should be in the sleeve.
 2. Pour sample into the liner.
 - a. Minimum volume is 10 mL of acid or 50 mL of water.
 3. Place white cover on top of sleeve/liner.
 4. Place brown disk on top of white cover.
 5. Insert vessel into vessel holder.
 6. The vent valves should be pushed to one side (same side for all vessels) so that all vessels will fit in the turntable.
 7. Place screw in top and tighten with fingers.
 8. When the screw is snug, take out the wrench and tighten until the handle clicks.
 9. Continue until all the vessels have been assembled.
 10. ****Note**-One of the vessels must be a “Blank” for quality assurance purposes.**

5. Assemble Control Vessel

- a. **Prepare the control vessel in accordance with the procedures outlined in the Vessel Manual. The control vessel must be an “actual sample” to accurately monitor the conditions during the digestion process.**
 - i. Use the following steps as guidelines, if you have never assembled a vessel, get trained before doing it based solely on these instructions.
 1. The liner should be in the sleeve.
 2. Pour sample into the liner.
 - a. Minimum volume is 10 mL of acid or 50 mL of water.
 3. Place white cover (that has the Thermowell) on top of sleeve/liner.
 4. Place white spacer on white cover (with the indented ring facing up).
 5. Place brown load disk on top of white spacer (with indented ring facing up)
 6. Insert vessel into the control support module. The control support module is the tallest one.
 7. Place screw (with hole) in top of the control support module and tighten with fingers.
 8. Gently insert the RTP-300 Plus temperature probe into the control vessel to make sure everything is lined up. If so, remove temperature sensor and tighten vessel with wrench. **(BE SURE THAT THE TEMPERATURE SENSOR IS NOT IN THE CONTROL VESSEL WHEN TIGHTENING WITH THE WRENCH---SENSOR WILL BE DAMAGED!)** If not, remove the temperature probe and realign the various parts. Repeat until the temperature probe can be fully inserted without any obstructions.

6. Load vessels

- a. **Install the turntable into the instrument, ensuring that the flat edge on the bottom of the turntable corresponds with the flat edge on the turntable lug.**
- b. **Install the sample vessels.**
 - i. Press the rotate key until the location where you want to place the sample vessel is facing the front.
- c. **Install the Control vessel.**
 - i. Gently insert the RTP-300 Plus temperature probe into the control vessel. This is a fiber optic cable, so don't force the sensor in. If it is difficult to insert, take apart the vessel and make sure everything is lined up properly.
 - ii. Connect the ESP-1500 Plus pressure sensor to the control support vessel. The tubing will screw into the port on the Control vessel. Finger tight is all that is needed.
 - iii. Press the rotate key until the location where you want to place the control vessel is facing the front.
- d. **Connect temperature probe to instrument.**
 - i. Insert the other end of the temperature probe into the roof of the cavity. The sensor will snap into place.
- e. **Connect pressure probe to instrument.**
 - i. Align the ESP with the connector port. Rotate the ESP-1500 Plus while gently pushing it into the connector port until it slips into position.
 - ii. 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.
 - iii. Position the pressure tubing in the guide ring mounted on the roof of the cavity.
- f. **Rotate the turntable while observing to the pressure/temperature cables to make sure they don't catch on anything.**

7. Run Method

- a. Press the Home key to return to the main menu.
- b. Press the Start key.

8. Shutdown Procedure

- a. **Do not turn the instrument off within fifteen seconds after operation with microwave power.**
- b. **Press the P/T key to display the current pressure and temperature. Do Not remove the vessels until these readings are at a safe level—Temp. 20-30°C**
- c. **Remove vessels and vent towards the back of the fume hood.**
- d. **When finished with the samples, clean and dry the vessels.**
- e. **Turn off the instrument.**