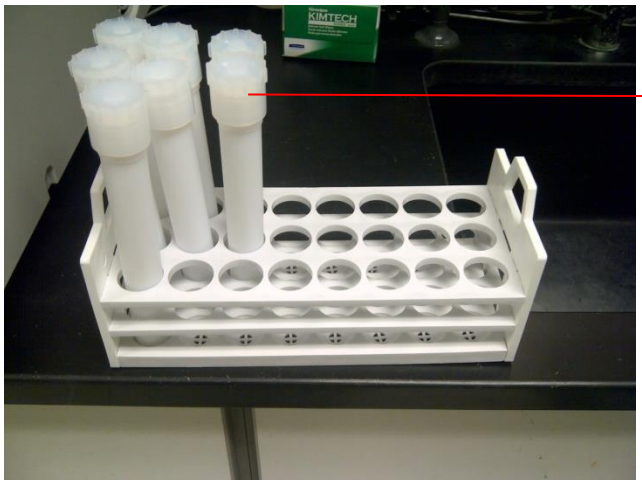
CEM Microwave Digester Protocol

Revision 10 7/2/2019

Before proceeding to microwave digestion of your samples please consult the "ICP-OES target detection range" or other appropriate instrumentation documentation to be sure you are digesting the appropriate mass for detection levels for the analysis.

It must also be known that your procedure may vary from this general protocol, and for safety reasons you must have confirmation from GEL staff that your procedure will be safe and effective. This is especially true if Hydrofluoric acid (HF) is being used for digestion. If you are using HF, it is required that you have HF safety training from EH&S. **When using HF, please read the "HF safety guidelines" on the instrument web page and contact Gwyneth Gordon or Sarah Kempkes for usage approval.**

All sample preparation should be performed in fume hood next to microwave**. Only the translucent vessels should be used as the white ones are more prone to rupture. Maximum temperature for the vessels is 210 C.** Higher temperatures may result in the vessel venting and cause loss of sample.



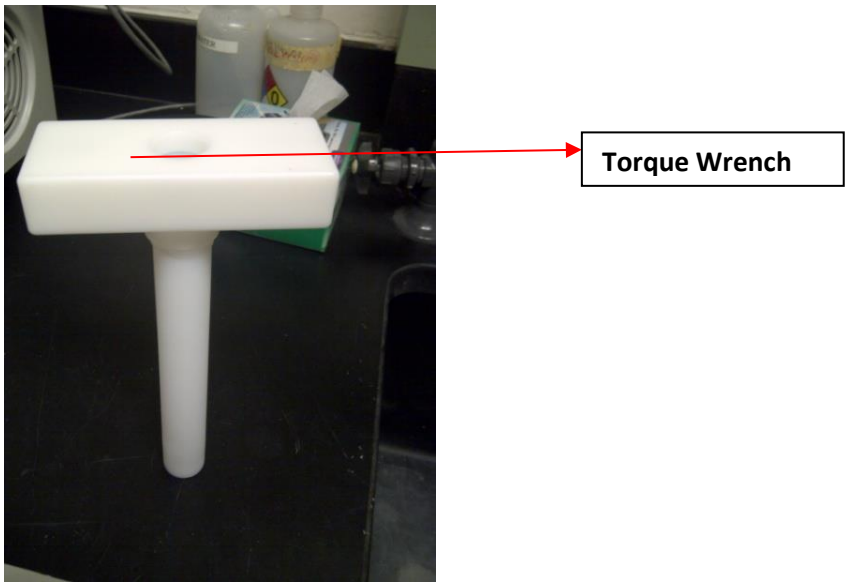
Warning: Acid decomposition of certain chemical compounds can be hazardous. Prior to use of the Mars instrument or MarsXpress vessels, consult the list of "Compounds Unsuitable for Microwave Digestion" in the instrument operation manual.

Tube preparation

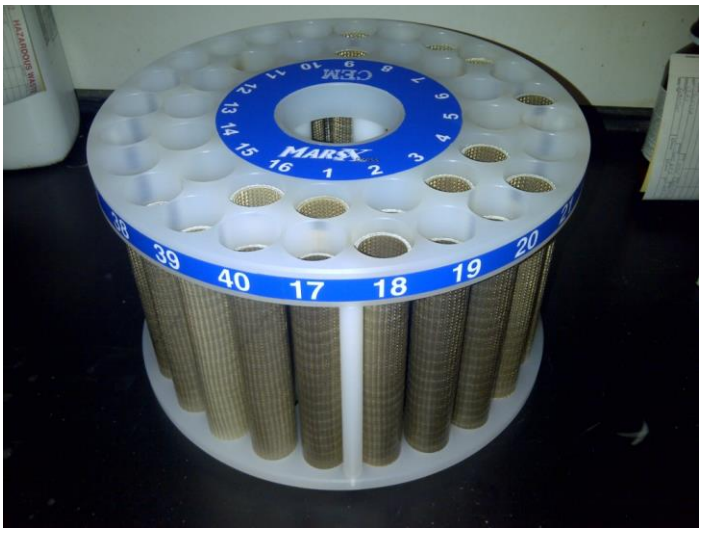
Microwave tubes or vessels are cleaned after each use by soaking in the 10% Nitric Acid bath in GWC 675 for at least 2 hours after each use. They should then be rinsed 3 times in Reverse Osmosis (RO) water. After this rinse set tubes in bins to dry. Tubes are Teflon lined so that they expel digested contents with minimal residue. However sometimes material may cake onto vessel and will require acetone rise or brushing to remove residue. Please use only the brush provided for cleaning as other brushes may scratch the surface of the tubes. Be sure to inspect vessels for any signs of wear such as bulging or internal blistering and do not use any suspect tubes.

Sample Preparation

1. Prepare to weigh sample and QC masses into clean, dry MARSXpress vessels. Holding tubes into light can make it easier to see that they are clean.
2. First deionize the tube to eliminate static buildup and then place the vessel on the balance (milligram precision). Tare out tube and add sample material to tube. Record exact weight and immediately cap to prevent contamination.
3. For every digestion procedure, 3 vessels of a certified compound for QC check, as well as 3 blank vessels should be prepared along with sample preparation.
4. After pipetting the appropriate acid volumes for the digestion method, using the fume hood next to microwave, wait a minimum of 15 minutes prior to using the Torque wrench to seal the vessels. This step is necessary to release product gases and mitigate excessive pressure buildup during digestion.
    a. Max volume for tubes is 20 mLs of liquid.
    b. Tops of tubes should be secured with Torque wrench until you hear the “click”



Note: It is essential that all of your vessels be inserted FULLY into each sleeve on the carousel before microwave use, for both safety and tube integrity. Vessels will expand and form bubbles, holes or swelling of walls where they are not contained by the sleeve. This will destroy the vessel and the sample will be lost.



c. A minimum of 8 vessels should be microwaved in one run, and these tubes should be spaced evenly around the carousel. If less than 8 samples then fill additional tubes with the same volume of digestion matrix and place those vessels in carousel. This will ensure consistent temperature measurement throughout the digestion.
d. There is a specific pattern in which to distribute the vessels on the carousel, see the vessel distribution document at the workstation in GWC 673 or obtain a copy from GEL staff.

5. Open microwave by pressing button on top right corner.
6. Press the turntable key on the instrument to rotate the turntable drive lug so that the drive lug is parallel with the front of the cavity.
7. Install the turntable on the drive lug with vessel #1 positioned farthest from the back wall of the cavity (nearest to operator).
8. Ensure that the turntable properly engages the drive lug until it "clicks".

Software

1. Open the Microwave control shortcut link on the desktop
2. Enter preferred Method Name
3. Under control style select Ramp to Temperature
4. Under vessel type select Xpress
5. Make any additional adjustments based on your protocol and click OK
    a. Power level should be adjusted with the respect to the number of vessels.
        i. Guidelines for framed vessels are
            1. 1-2 vessels (400W)
            2. 3-6 vessels (800W)
            3. 7 or more vessels (1600W)

    The PERCENT of wattage applied can be adjusted when entering the method into the software to refine power strength.

6. Click "Start" on top of page
7. Keep monitoring system for temperature
8. When digestion has finished wait until the tubes have cooled (30 min- 1 hour) before removing the carousel and opening the vessels.
9. Some materials, especially those with high organic content, may effervesce when relieving the pressure following digestion. For ALL samples, rotate the cap **very slowly** using the Torque wrench, so that the sample does not effervesce and leak out of vent on top of vessel. You may have to leak pressure slowly by loosening and tightening the cap repeated times until sample is completely depressurized. This manual venting procedure <u>should be done when samples are at room temperature and performed in the fume hood.</u> Appropriate personal protective safety equipment should be worn to prevent any chemical burns. ALWAYS point the vent hole in the vessel away from you when venting. Any sample or volume lost during this procedure will compromise sample results.
10. Pour all of vessel contents into a labeled 50 ml conical vial for transfer.

11. Check all of the samples for complete digestion. If solution is not homogeneous and clear seek advice from GEL staff. Sometimes the digestion for desired element can be successful even with residual material in vessel or solution.

NOTE: Concentrated acid samples cannot be stored in these conical tubes. After analysis is complete these samples must be disposed of properly. See GEL staff if unsure of how to properly dispose of samples.

Additional important information:

1. In case the system overpressures you will hear a whistling sound. The digestion will need to be stopped immediately.
2. Reservations for the instrument should be made for a minimum of 2 hours to allow for digestion and cool down of samples.
3. You are responsible for cleaning of the vessels after digestion and disposal of your samples.