

Standard Operating Procedure

CEM Mars 5 Microwave Digestion (MARSXpress Vessels)

Introduction

The Microwave Accelerated Reaction System, CEM MARS5, is designed for digesting, dissolving, hydrolyzing, extracting, and drying a wide range of materials

Purpose

The purpose of this document is to introduce the user to the operation of the CEM Mars 5 Microwave Digestion system when using MARSXpress vessel available in the TRACES Centre. This document also outlines the special safety concerns introduced when completing microwave digestion.

Scope

This procedure applies to all users who wish to use the microwave digestion system. Individuals must complete training with TRACES staff and sign a prerequisite safety waiver prior to operating the system.

Referenced Documents

- CEM MARS 5 Operation Manual (p/n# 600122)
- MARSXpress Microwave Digestion Vessel Manual (p/n 600968)

Responsibilities

1. TRACES Users

- 1.1. All Users must obtain training with TRACES Staff prior to system operation. It is the responsibility of the User to ensure they have a good understanding of the instrument and all operation protocols.
 - 1.1.1. User is responsible for inspecting each digestion vessel for malfunction or cracks.
- 1.2. The User must submit a signed Microwave Safety Prerequisite Waiver prior to system operation.
 - 1.2.1. The Waiver must be approved by TRACES Staff.
 - 1.2.2. Passcode and/or Electrical switch to start the system will be granted to approved users.
- 1.3. If additional training sessions are needed it is the responsibility of the User to schedule these with TRACES Staff.
- 1.4. Instrument time must be booked by the User via the online booking system prior to system operation.

2. TRACES Staff

- 2.1. It is the responsibility of TRACES Staff to provide introductory and, if required, subsequent training to all users.

Equipment

- CEM MARS 5 Microwave Accelerated Reaction System
- MARSXpress 55 mL PFA (**P**erfluoroalkoxy **a**lkanes polymer similar Teflon™) vessels (12 available)
- MARSXpress Torque Tool

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Procedures

1. Sample Preparation

Samples should be prepared using the analytical balance and fume hood provided in EV216. Each User is responsible for bringing any materials needed for sample preparation.

Important Notes:

- The maximum allowable volume per vessel is 25 mL of liquid
- Samples that have not been previously run should have a starting weight of 0.1 grams.
- A minimum of 8 vessels must be microwaved during each analysis. If you have less than 8 samples fill additional vessels with the same volume of digestion matrix.
- For low level application requirements additional vessel cleaning may be necessary. Contact TRACES Staff to discuss.

- 1.1. Place a clean, dry MARSXpress vessel on the analytical balance.
- 1.2. Tare the balance and add sample material to the vessel. Try to place the sample at the bottom, avoiding the vessel walls.
- 1.3. Record the exact mass and immediately cap the vessel to prevent contamination.
- 1.4. In the fume hood pipette the appropriate acid volume into each vessel and wait a minimum of 15 minutes. This will mitigate excessive pressure buildup during digestion and allows for the release of product gases.
 - a) Make sure sample components, especially powders, are suspended in the acid and not on the wall of the vessel. This can create a hot spot and may lead to vessel failure.
- 1.5. Place the lid on and hand tighten each vessel. Following this use the torque tool (Figure 1) to tighten the lid until you hear a single click.
- 1.6. Distribute your samples evenly around the turntable. Ensure that each vessel is properly positioned.
 - a) Refer to Appendix A for a schematic of correct placement.

IMPORTANT: All vessels must be inserted FULLY into a composite sleeve on the turntable. During microwaving, 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.



Figure 1. Schematic of A) how to properly use torque tool and B) proper insertion of sample vessels

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2. Instrument Preparation

- 2.1. Inspect the instrument for any cracks, dents, or warping.
- 2.2. Inspect the door for any damage and for proper alignment. When closed, the door should sit firmly against the front of the microwave cavity. The push button on the top of the instrument must release and engage the door latch mechanism.

IMPORTANT: If damage is noted, do not attempt instrument operation. Contact TRACES Staff immediately.

- 2.3. Turn on the system using the power switch on the right side of the instrument.
- 2.4. Press the turntable key (Figure 3) on the instrument to rotate the turntable drive lug so that the drive lug is parallel with the front of the cavity.
- 2.5. Install the turntable on the drive lug (Figure 2) with vessel #1 positioned farthest from the back wall of the cavity (nearest to operator).
- 2.6. Ensure that the turntable properly engages the drive lug until it “clicks”.
- 2.7. Close the microwave door.

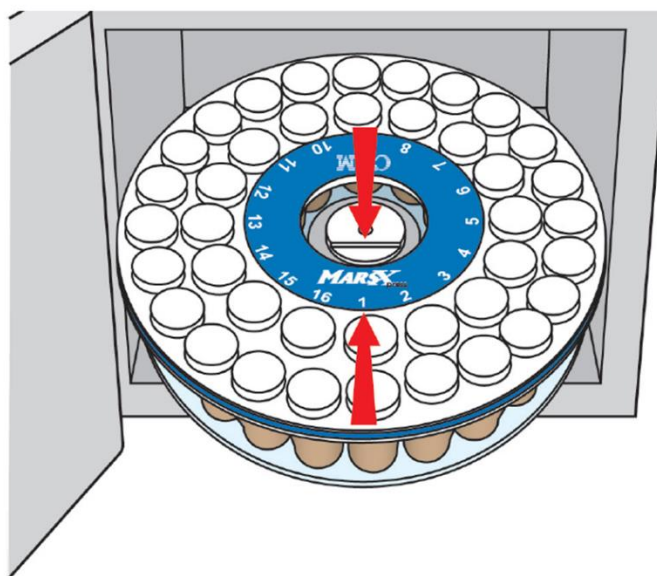


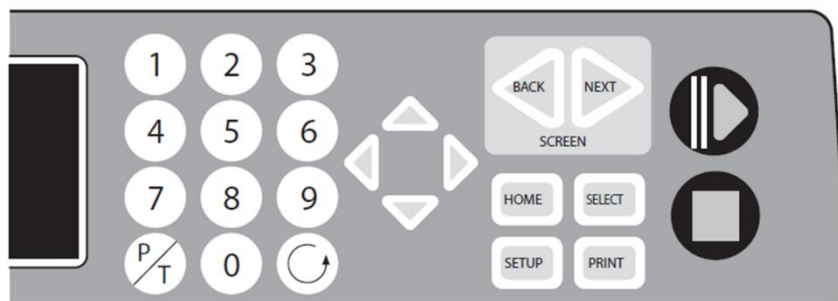
Figure 2. Proper positioning of the turntable during installation

3. Sample Analysis

3.1. Loading a Method:

- a) On the home screen select “Load Method”.
- b) Select “CEM Directory” or “User Directory” based on the location of your desired method.
- c) Use to arrow keys to scroll to the method you want and press the “Select” key.
- d) Start the method by pressing the “Play” key.
- e) Monitoring the system throughout analysis:
 - Depending on your method and the probes you are using you should monitor the temperature and/or pressure throughout the analysis.
 - If you hear a whistling sound the system is over pressurized. Stop the digestion immediately.

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Key	Function
Start/Stop	Begins the method currently displayed on the screen or pauses the operation during any stage of the method
Home	Returns to the main menu from any point in the software selections
Select	Enters selected parameter and in some instances advances to the next screen
Arrow Keys	Scroll through selections such as methods, vessels, etc.
Turntable Key (circle with arrow)	Rotates the turntable when the instrument door is open

Figure 3. Overview of important keyboard functions

4. Sample Removal

- 4.1. When the digestion has finished permit the tubes to cool undisturbed for at least 30 minutes before removing them from the turntable or opening the vessels. Venting should not be completed until samples have reached room temperature.
- 4.2. In a fume hood carefully open each vessel, any sample or volume lost during this procedure will compromise sample results. Rotate the cap slowly using the torque tool with the vent hole facing away from you.
 - a) You may have to release the pressure slowly by loosening and tightening the cap multiple times. Repeat this process until sample is completely depressurized.
 - b) Appropriate personal protective safety equipment should be worn to prevent any chemical burns.
- 4.3. Transfer the contents of each vessel into a labeled conical tube for transfer.
- 4.4. Check all samples for complete digestion. If the solution is not clear and homogeneous contact the TRACES Staff.

5. Cleaning

It is the responsibility of each User to clean the microwave vessels. Vessels should be cleaned directly following digestion. Failure to do so will result in an additional fee being added to your instrument time.

5.1. Cleaning PFA vessels:

- a) Use hot water, liquid detergent, and the foam cleaning brush provided in EV216 to clean all plastic vessel parts. Soak vessels for 10-15 minutes in the provided container.

IMPORTANT: Avoid abrasive cleansers and stiff brushes which can scratch the Teflon surface.

- b) Rinse each vessel thoroughly with DI water.

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- c) Inspect each vessel. If visible residue is noted additional cleaning is necessary. If vessels appear clean continue to g).
- d) Soak each vessel in a 10% v/v nitric acid bath for 10-15 minutes.
- e) Rinse each vessel thoroughly with DI water.
- f) Inspect each vessel. If visible residue remains contact TRACES Staff for additional cleaning instructions.
- g) Following the DI water rinse vessels should be left on the bench top to dry. Vessels must be placed on their side as this will allow acid vapors to dissipate while preventing dust particles from falling inside.

5.2. Cleaning Lids and Turntable

- a) Use hot water, liquid detergent, and the foam cleaning brush provided in EV216 to clean all plastic vessel parts.
- b) Rinse each vessel thoroughly with DI water.
- c) Following the DI water rinse Lids and the turntable should be left on the bench top to dry.

5.3. Cleaning Composite Sleeves:

- a) If acid gets on a sleeve, wipe it off with a damp paper towel and allow to dry. Do not soak composite sleeves in water.
- b) If sleeves get wet, allow them to air dry.

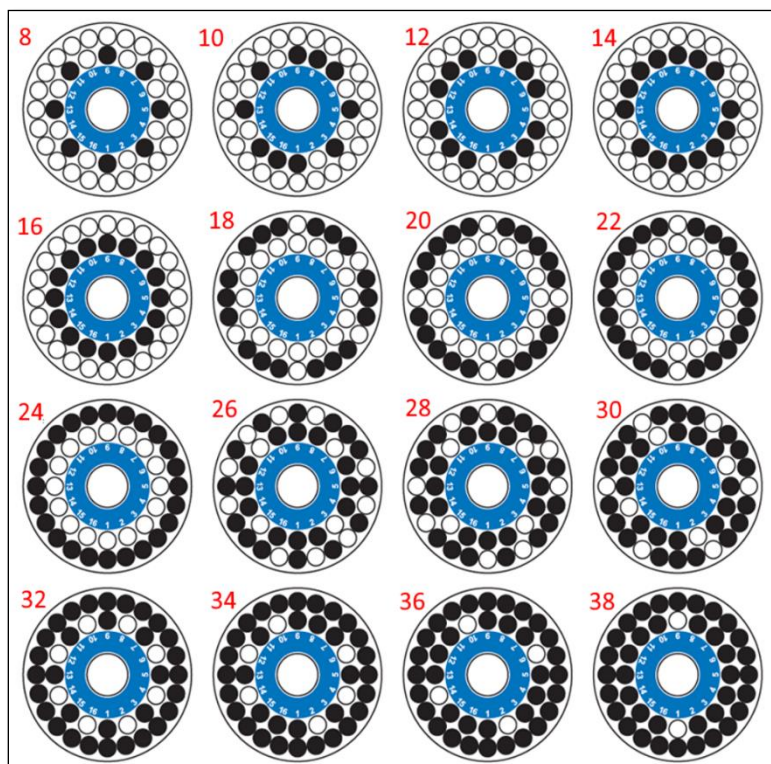
Appendix A – MARSXpress Vessel Guidelines.

- Never wash or soak the vessel sleeve directly in water. They should be wiped with a damp cloth.
- Maximum sample size of dry weight organic samples refers to well characterized samples and is not for unknowns.

Vessel Specifications

Vessel Size	55 mL
Minimum Volume	5 mL
Maximum Volume	25 mL
Max. Organic Sample Size (dry wt)	0.5 g
Max. Inorganic Sample Size (dry wt)	1.0 g
Maximum Method Temperature	210 °C
Minimum Vessels per Run	8

Correct Xpress Vessel Orientation



Incorrect Xpress Vessel Orientation

