CEM Mars 5 Microwave Accelerated Reaction System

General description and features

The microwave accelerated reaction system is designed for digesting, dissolving, hydrolyzing a wide variety of materials in a laboratory setting. The system uses microwave energy to heat samples in polar or ionic solutions rapidly and at elevated pressures. Its main purpose is for preparing samples for analysis by atomic absorption (AA), inductively coupled plasma emission spectroscopy (ICP), gas or liquid chromatography.

The system consists of:

- A microwave power system with user selectable power settings (0-1200 Watts),
- A fluoropolymer-coated microwave cavity (oven interior),
- A cavity exhaust fan and tubing to vent fumes,
- A programmable microcomputer that controls and monitors the power, temperature, and pressure within the reaction vessels, and is capable of storing 100 programs with up to five reaction stages per program,
- Explosion proof reaction vessels, and one specialized vessel for monitoring temperature and pressure,
- An alternating turntable that turns the samples 360 degrees within the microwave field,
- A door safety interlock system that prevents microwave emissions when the door is open.

Reaction vessels

- CEM has several vessel types that are designed for their ovens: (1) Closed-system vessels including the HP-500 (500 psig material design pressure and 260 °C), pictured below (fig.1), have liners are composed of PFA and are ideal for many types of samples including: soils, sediments, sludges, air filters, wipes, paint chips, metals, alloys, biological and plant tissues, waters, wastewater, and TCLP extracts. The XP-1500 vessel can handle pressures up to 1500 psig material design pressure or 300 °C. The XP-1500 vessel liners are available in either TFM® Fluoropolymer

or quartz, and are useful in digests of oils and heavy organics, hazardous wastes, refractory materials & ceramics, and ores and slags. CEM also makes a high throughput accessory set for digesting over 50 samples in an open-vessel (50 ml. polypropylene centrifuge tubes). Digestion with this system is temperature controlled and samples should be readily decomposable. Since venting is not controlled this type of system is not recommended for analyzing trace volatile compounds.

- A number of safeguards have been incorporated in the MARS 5 system to eliminate the possibility of an explosive high pressure material failure. Each reaction vessel fits within a microwave-transparent sleeve made of Kevlar, and each vessel-sleeve assembly gets clamped within a carriage or 'support module' with 5 foot-pounds of torque applied to the clamping bolt. A plastic membrane pressure rupture disc that fits between the exhaust port in the vessel lid and the threaded blue cap (shown in figure 1, and 2) ensures the digestion takes place under completely closed conditions at lower pressures. The blue cap has a small hole drilled in it. Should pressures exceed what is safe for the particular vessel the membrane will rupture, exhausting gas through the port in the vessel lid and the cap and into the oven, which exhausts into a fume hood. The microwave oven also has a rupture pressure sensor that stops the power to the microwave if it detects a sudden pressure change.

Figure 1. From left to right; control vessel in the support module with pressure sensor attached and fiber optic temperature sensor inserted, a typical reaction vessel in a support module, and an assembled reaction vessel showing the explosion-proof Kevlar sleeve and the load distributing cap piece placed on the vessel lid.

- The plastic membrane rupture disc can easily be placed inside the blue cap with the pen-sized suction cup devise supplied by the manufacturer. See figure 2.

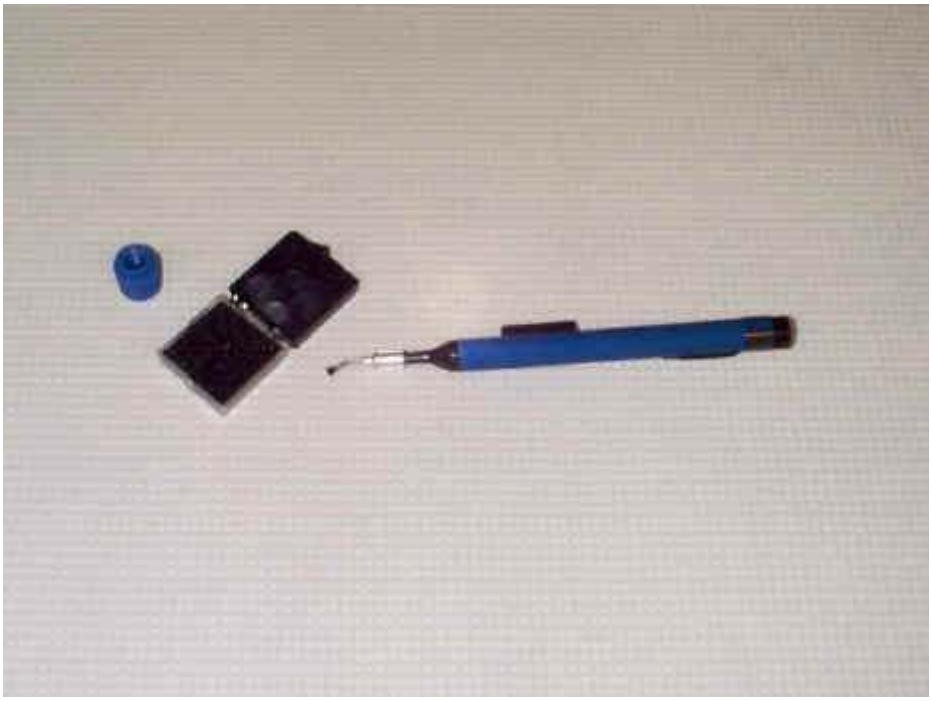

Figure 2. From left to right; threaded blue cap that fits on the reaction vessel lid and into which a pressure membrane (in the black box) is placed with the pen-sized suction devise.

Control Vessel

Only one reaction vessel of a set of 14 vessels (per carousel) is monitored for temperature and pressure. The amount of power supplied by the oven and the rate of temperature and pressure increase within the vessels is regulated by the parameters of a user-defined computer program. The power is modulated based on the temperature and pressure measured within a specialized control vessel (figures 1, 3, and 4). Since the amount of heating within a solution is dependant on the types of ions and dipolar molecules present it is important that the sample placed in the control vessel be representative of the other samples digested simultaneously.

The control vessel is made of the same material as the other reaction vessels except that it also has a pressure sealed glass well (Thermowell) that penetrates into the vessel to allow a fiber optic temperature sensor to be lowered into the vessel. A pressure sensor is also attached to the control vessel lid. See figure 3.

Thermowell and fiber optic temperature sensor

The Thermowell is a glass tube open at only one end. The open end fits into a compression fitting in a threaded cap that tightens as it screws on to the control vessel lid and allows the temperature sensor to be lowered into the pressure sealed vessel. The closed end of the Thermowell is submerged in the sample mixture.

Temperature in the sample mixture as it is heated is measured by a microwave-transparent infrared fiber optic sensor. One end of the sensor is lowered into the Thermowell and the other is connected to a snap-in port in the oven (see figure 8). The temperature sensor that can read up to 300 °C and is completely inert to all corrosive reagents.

Pressure sensor

The pressure control system can read pressures up to 1500 psig and is composed of two parts: (1) the control electronics and connector, which are built into the oven, and (2) the sensor which is connected to the control vessel lid via a flexible microwave transparent tube (the blue tubing shown below is a sheath that surrounds the pressure line). The sensor consists of a pressure sensing load-cell mechanism located within the black cylinder attached to the tubing, and gets mounted to the connector in the oven cavity. See figure 3, 4 and 8. During operation the pressures within the reaction vessels are transmitted to the load cell which sends a signal to the electronic control unit. Unlike older microwave systems that had pressure lines charged with water, the MARS5 system relies on control vessel vapor pressure directly acting on the load cell. Both the pressure sensor and temperature sensors can be calibrated and zeroed using the microwave oven's computer.

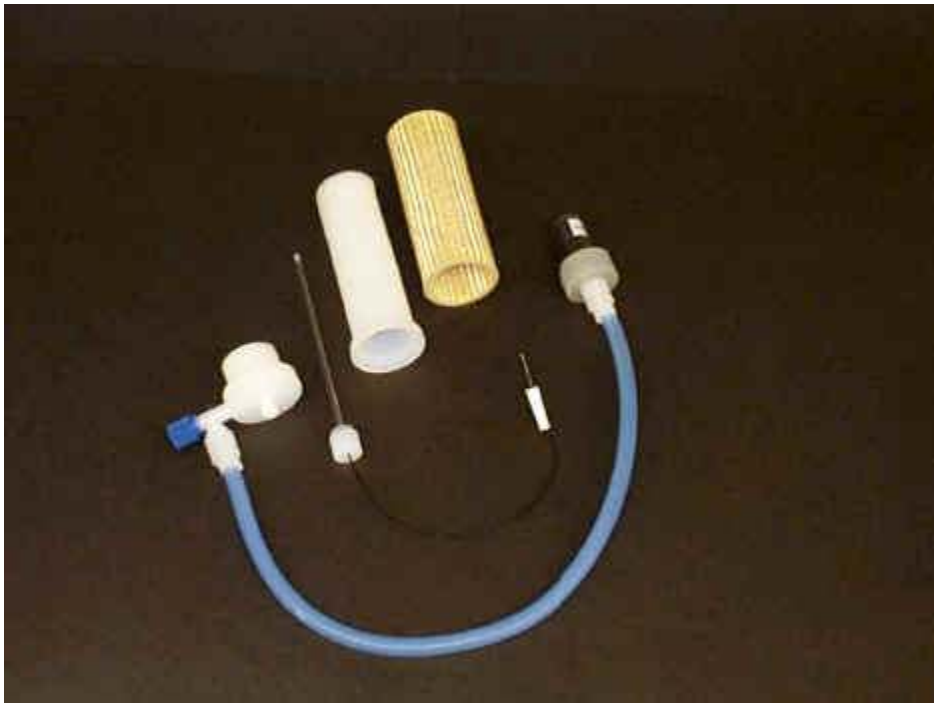

Figure 3. Control vessel parts, from left to right: Vessel lid with pressure release check valve and pressure transducer attached, glass thermal well with compression fitting and fiber optic temperature sensor, Teflon reaction vessel, Kevlar explosion proof sleeve, and pressure transducer.

Figure 4. Control vessel assembled and housed in the support module.

The Carousel

- To promote even heating among and within all the reaction vessels in the microwave oven cavity they are rotated through the microwave field using a carousel. The carousel is essentially a turntable that holds up to 14 support modules and reaction vessels. (The MARS 5 HP-500 system has 14 vessels per carousel, different systems will hold more or fewer support modules). The turntable rides on ball rollers and is turned 360 degrees per cycle, alternating the direction of rotation in each cycle. The turntable fits on a drive lug within the oven. See figures 5 and 6.
- In addition to rotating the vessels in the microwave field the oven also has the capacity to rotate small magnetic stirring bars that can be placed within the reaction vessels to ensure even mixing. This feature can be useful in an extraction protocol.
- The support modules are secured to the turntable with an interlocking key system, and an outer plastic ring is placed around the top of the support modules for additional support and for explosion containment.

Figure 5. Carousel, showing pressure sensor and fiber optic temperature sensor attached to the control vessel and outer plastic ring around all of the support modules.

Figure 6. Carousel bottom; showing the ring of ball rollers and receptacle for the drive lug.

The Oven

External features:

- Push button door opener on the top operates the safety door latch.
- On/off switch is located on the side above the air intake.
- Microcomputer keyboard and display are on the front.
- Exhaust tubing and blower are attached from the back. These remove harmful fumes from the cavity to a fume hood or approved air handling device.
- Power cord receptacle and access to fuses are on the back.
- The oven also features the *Reactiguard* system, which senses pressure changes within the oven cavity in the event of a sudden pressure change and then shuts down the power.

User programmable microcomputer

- The oven's microcomputer has a menu of preprogrammed settings. Some of these are EPA methods, and some are methods developed by CEM Corp. The user can also change a number of the ovens parameters including the percentage of power applied, the total wattage, control temperature, pressure, ramp and hold time, and the number of reaction stages or cycles.
- The measured values for time, power, pressure, and temperature can be stored and recovered within the computer or sent to a remote computer for analysis on some oven models.

Figure 7. Oven exterior

Figure 8. Inside the oven; showing the pressure and temperature sensor connections. The temperature sensor connection is located in the middle of the oven ceiling. The pressure tubing wraps around the temperature connector, and is attached to the oven ceiling with a removable plastic clip.

Accessories for the MARS 5 supplied by CEM Corp.

- **Stirring** The stirring option provides variable speed stirring in each individual vessel. A magnetic spin bar is placed into the vessel to provide sample stirring. This insures sample homogeneity and eliminates possible temperature gradients. Stirring speed is controllable from the main program software screen. Includes 14 stir bars. This option is recommended for all applications.
- **Available Stir Bars**
  Digestion
  Extraction - Egg Shaped
  Extraction - Octagon Shaped
  Extraction - Carboflon Filled (for use with non-polar solvents)
  A rotating plate containing magnets is located underneath the MARS cavity floor. The magnetic field couples with a spin bar placed into the vessel to affect sample stirring.
- **MARSLink™ Software** (Version 2.0) This powerful, yet easy-to-use, Windows®-based, multi-tasking software package connects the MARS 5 System to any PC (configured with Windows® 3.1 or higher). The software provides method storage, data collection, real-time graphic display of temperature and pressure profiles and report writing capabilities. Includes program disk (3.5" & 5.25" formats), 12 ft. null modem serial cable with RS-232 connectors and installation guide.

- **Computer requirements:** IBM PC or compatible running Microsoft Windows® Version 3.1 or higher, Windows® 95 or higher, or Windows® NT.
- **MicroVap Accessory Set** Enhance the performance of your CEM microwave digestion system with this accessory for pre- and post digestion processing. The MicroVap makes drying of samples before digestion as easy and clean as possible! It also reduces acid volume after the digestion step and concentrates acidified drinking water samples. Process up to 12 samples at a time using a variety of CEM microwave digestion liners. An optional HEPA filter can be used to control the sample environment. Samples are exposed to microwave energy while under a vacuum to accelerate the sample drying and acid volume reduction steps. An "auto-detect" feature of the MARS 5 system software determines when the sample evaporation step is complete and automatically stops operation. Acid fumes are automatically neutralized by the integrated vapor scrubber module.

A typical procedure for preparing plant material for ICP/MS

- Dried plant material (0.1 - 0.5 grams of homogeneous material) is weighed out and placed in each reaction vessel.
- Concentrated acid (10 ml 70% nitric acid) or dilute acid and oxidizer (5ml H2O + 5 ml 70% nitric acid + 2 ml 30% hydrogen peroxide) are added to each vessel. The precise recipe can vary depending on the material to be digested and who needs the information. For the sake of uniformity, specific methods have been developed that are prescribed by the EPA and ASTM for the digestion of certain material. See the links to methods section. It is recommended that the mixture allow to sit for 15 minutes before tightening the reaction vessel lids to allow any vigorous oxidation to vent.
- The vessels are placed in the support modules with the load distributing caps, and tightened with a torque wrench to 5 ft*lbs. Blue pressure caps (with pressure membranes inserted) are to be hand tight. Attach pressure sensor and Thermowell to the control vessel. Place the support modules on the carousel and attach the top ring.
- Either select pre-programmed microwave control settings from the menu installed on the oven's computer, or program in parameters for power, control temperature, pressure maximum, ramp time, hold time and stage settings that you select or are specified by a standard procedure. Zeroing the pressure sensor every so often is recommended.
- Place the carousel on the drive lug in the oven and attach the pressure sensor and temperature sensor. Close the door, press start.
- After the cool down period when the temperature sensor reads below 35 degrees C it is safe to remove the carousel from the oven. Pressure in the reaction vessels should be released slowly by unscrewing the blue cap and venting any remaining vapors in a fume hood.
- Samples should be filtered through filter paper or centrifuged and diluted to an appropriate final volume (ICP machines need to have acid concentrations be below 20%).

Sample handling and safety

A number of precautions should be observed for safe operation of the microwave system:

- Before subjecting vessels to microwave energy the vessels should be free of particulate matter or drops of liquid such as high salt solutions that can absorb microwaves, which in turn can cause localized heating and damage to the vessel.
- Pressure relief devices should always be used when heating samples.
- When working with unknown samples it is suggested that the user allow 15 minutes to predigest the samples before sealing the vessel. This will allow for easily oxidized compounds to exhaust volatile gasses and diminish the pressure inside the vessel during heating.
- Never heat high boiling point acids like concentrated sulfuric or phosphoric acid in the microwave.
- The user should consult the operation manual for the list of solutions and solvents not recommended for use in the system or in the particular reaction vessel you are using.

Figure 9. After the microwave digestion and the cool down period the pressure in the reaction vessels should be released by slowly unscrewing the blue cap and venting any remaining vapors in a fume hood.