MILESTONE

HELPING CHEMISTS

tips and techniques

for Microwave Digestion Systems

Chapter 1
Introduction

A Milestone Microwave Labstation is designed to provide many years of high performance and superior results, processing samples for a variety of applications. The design of the labstation and accessories is the result of almost fifteen years experience developing systems and vessels for microwave sample preparation. The components are selected based on their properties and suitability for the application as well as for the environment in which they will be used.

Experience gained in Milestone's worldwide applications laboratories, and in the laboratories of our many customers, continues to guide the development of future products. Also, this wealth of experience is a ready reference for method development and operator techniques for optimizing system performance.

What follows here is a useful compendium of observations, cautions, suggestions and recommendations to help you get the most out of your labstation, its related options and accessories. Some of the items address safety issues. Some of the items address care and maintenance issues. Some of the items will be more or less applicable to your specific system configuration and sample preparation routines. All of the items are worth your review and consideration.

If you follow these recommendations, you can secure the performance and the longevity of your microwave labstation for its intended purpose. Disregard these recommendations and you may find that the useful lifetime of various accessories will be limited.

Chapter2
Facilities Preparation

Like any sophisticated laboratory instrument, certain facilities must be available to support the proper function and long term performance of your Milestone Microwave Labstation.
This section identifies and addresses several concerns.

2.1 Bench space: firm, level, secure surface

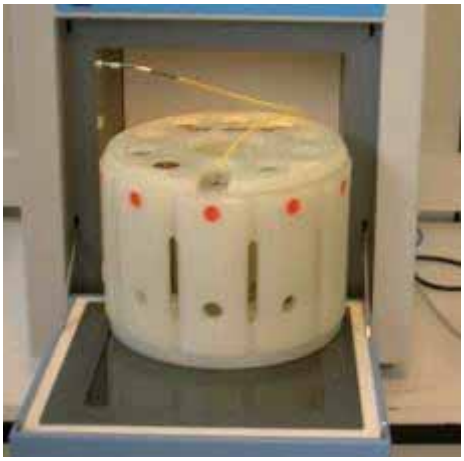
- **Labstations are Heavy**

A Milestone Microwave Labstation is heavy. A complete system, combined with a controller and a variety of vessels / rotors can exceed 100 kg total weight. The typical lab bench can easily handle the combined weight of a complete labstation. When placing the labstation on a bench or cart, care must be taken to avoid dropping the unit.

There are four rubber feet on the bottom of the Milestone Microwave labstation. The two rear feet have a smooth surface while the two forward feet are tacky. This allows the instrument to sit securely on a smooth lab bench surface, and also for easy maneuvering during installation and service. (Lifting the front feet off the bench allows the instrument to be easily turned and moved on the bench top.)

- **Door Opens Downward**

The labstation door opens downward, creating a convenient work surface for manipulating rotors and vessel components. Any placement of the labstation must not restrict the full movement of the door to its open and down position. It is entirely safe and acceptable for the fully opened door to extend beyond the edge of the lab bench.



- **Setup on a Lab Cart**

The Ethos Microwave Labstation can be setup on a movable lab cart. This approach is used when the labstation is being shared by several users or permanent bench space is not available. The cart should be designed for heavy loads, and the work surface should be firm and level when in use. A flat top cart is best so as not to restrict the full downward movement of the cavity door.

2.2 Electrical: 220-230, VAC, 16 amps

- **Ethos Labstation - Power and plug.**

A Milestone Microwave Labstation has very specific power requirements for proper operation. The labstation draws a maximum of 16 amps on a 230 VAC single phase line. The labstation must be

connected to a grounded, single phase line, with 220–230 VAC output. Each instrument is shipped with a grounded power cord with a Shuko plug attached. The laboratory should be outfitted with the corresponding Shuko receptacle. The end user, of course, is free to substitute an alternative plug / receptacle combination as appropriate to the facility.

- ✓ **WARNING Connection of the Ethos lab station to 110 VAC line will likely cause damage to the instrument.**

➢ Grounded, Not Bonded

A particular note is in order regarding the system ground. It is critical that the ground pin on the receptacle be attached to the neutral/common at the breaker box. A "Bonded" Circuit (ground pin is attached to an earth ground pipe) must not be used since this can lead to serious problems with the ATC-CE (thermocouple style temperature probe).

➢ Low Power Consequences

There are consequences to running an instrument with low line voltage (below 220 VAC). First, and most apparent, will be a reduced maximum output power. Low power (between 208 and 215 VAC) will generally reduce the magnetron output power. This may not even affect a customer's work except when running full rotors with large sample volumes. The symptom may be a failure to follow (drop below) the programmed temperature profile. Also, low power will limit the maximum number of vessels and/or the maximum total sample mass that can be processed at any particular target temperature or temperature ramp rate.

Second, low power will generally result in additional stress and train on the carousel motor(s), the exhaust fan motor and the twist board relay. This may shorten the lifetime of these components and it may prevent the normal operation of the labstation (one or more of the motors will not develop sufficient torque and may stall). The lifetime of these components may be reduced by 15% to 20%.

➢ Step Up Transformers

Milestone recognizes that it may be necessary for the facility power to be upgraded prior to delivery and installation of a Milestone Microwave Labstation. Milestone recommends that the selection and permanent installation of step-up transformers be undertaken only

by qualified individuals, in full compliance with all required local electrical codes, and with special attention to grounding (not bonding) of the input and output.

- **800 Controller (with keyboard and mouse)**

A labstation controller can be operated on either 110 or 230 VAC.
Each controller, if not auto sensing, is configured at the factory to operate on 230 VAC only and is supplied with a 230 VAC power cord. If 110 VAC operation is desired, this can usually be configured at the time of installation.

2.3 Ventilation

(process fumes, vessel opening, instrument surroundings)
There are several issues that are best addressed by proper ventilation.
The primary concern is to limit operator exposure to process chemical vapors (acids, solvents, reaction products, etc.) during routine handling of vessels.

- **Working with Vessels in the Hood**

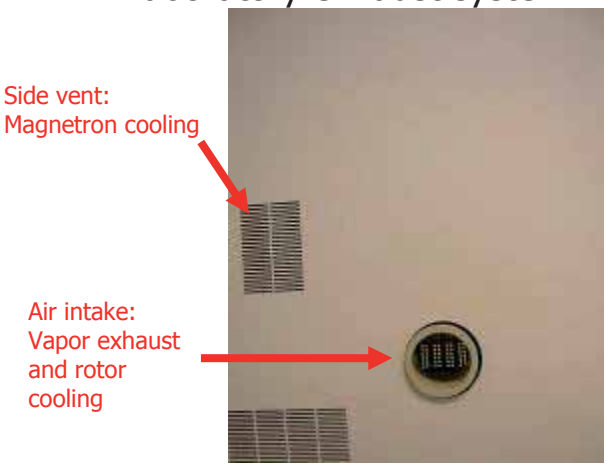
In most cases, quantitative loading of samples into vessels will involve use of an analytical balance. Milestone recommends that all addition of acid mixtures or hazardous solvents to vessels should take place inside an appropriately vented fume hood.
The microwave heating process will generate reaction products inside the vessel. Even after the vessels have been cooled, digestion products will continue to exert a positive pressure inside the closed vessel.
Milestone recommends that vessels be opened inside an appropriately vented fume hood to contain and convey reaction vapors.

➢ Air Circulation and Clearance

Milestone Microwave Labstations are designed for the laboratory environment. The instrument requires sufficient clearance (approximately 15 centimeters space) on all sides of the chassis to allow adequate movement of air. The round opening(s), on the side of the instrument draw air into the cavity for rotor cooling and vapor exhaust. The vent slits at mid level on the side(s) of the chassis, actively cool the magnetrons. In the rear, air is drawn in through a dust filter for cooling of the electronic components.

➢ Cavity Exhaust

The Milestone Microwave Labstation is delivered with a two meter section of flexible hose with an elbow. This hose is to be installed in the rear of the exhaust module. The exhaust hose must be connected to some source of active ventilation: 1) one can simply snake it into the opening of a fume hood, 2) one can arrange some mechanical connection to attach the hose through the side of a fume hood, or 3) one can connect it to the local trunk of an active laboratory exhaust system.



➢ On the Bench, Not in the Hood

Milestone specifically cautions that the Milestone Microwave Labstation should not be permanently installed inside an active fume hood due to the potential for acid vapors to attack and accelerate the corrosion of internal electronic components.

✓ **WARNING In laboratories where acids and acid vapors are not present (common to solvent extraction or most organic synthesis applications), there should be little or no consequence to installing the labstation in a dedicated (solvent free), active fume hood. As with all electronics, one should take care to never operate a microwave lab station in an explosive atmosphere.**

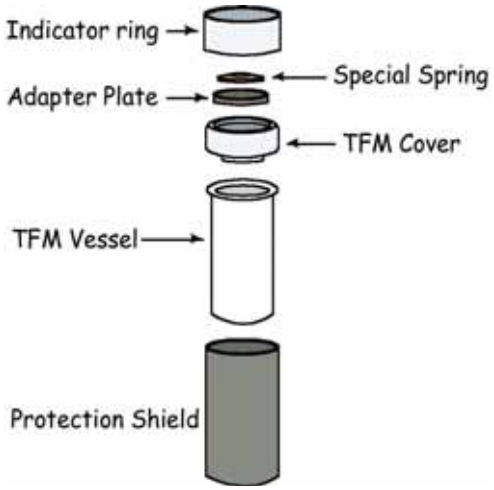
Chapter3
Vessels

Each closed, microwave sample preparation vessel is actually a system of components. They function in tandem to provide:

1. A closed, clean environment for containing the sample during processing, thereby preventing contamination,
2. A closed container that prevents loss of volatile species, even when processing materials at temperatures well above the normal boiling point of the mixture,
3. A closed container that minimizes the use of expensive acids/solvents,
4. A system that can safely release (vent) over-pressures in the vessel, then reseal the vessel and continue the microwave process to completion.

3.1 Components

Every new Milestone Microwave Labstation is accompanied by an Operator Manual. In this manual, the available spare parts and consumables are listed for easy ordering.
Each microwave sample preparation vessel assembly is a system of components that consists of:



1- *a vessel that contains the sample*.

2- *a vessel cover that seals the vessel and acts as a overpressure release valve.*

3- *a safety shield that surrounds the vessel and provides structure and safety for operation at elevated pressures.*

4- *a pressure adapter plate, that fits on top of the vessel cover.*

5- *a spring rated to the pressure performance specifications of the particular vessel/rotor combination.*

6- *a vent indicator ring.*

- **Labeling**

Milestone recommends that the components of each individual vessel assembly be segregated as a set. An effort should be made not to mix components between runs, during cleaning and/or during storage. Vessels and covers, in particular, should share a common history. This will facilitate maximum useful lifetime; maximize performance for routine operation at the extrems of vessel specifications; and ease identification, isolation and replacement of worn components when they begin to affect sample preparation performance.
Milestone recommends labeling each complete vessel assembly. A diamond-tipped scribe is recommended to mark the bottom of the TFM vessel and the top of the TFM vessel cover. Etching the sides of vessels is *not* recommended.

- **Usage Log**

A log system is an important tool to help determine the expected lifetime of the assemblies in particular application and can provide a rationale for budgeting for future consumables purchases. A usage log can also help track variable sample results by providing a complete elemental history for the vessel.

3.2 Care

Each vessel assembly requires a certain level of care and attention to its components in order to: maintain sample preparation performance, maximize the useful lifetime, and to ensure continued safe operation of the labstation. The exact lifetime of each component cannot be specified as it is a complicated function of the temperatures, pressures and chemistry of your specific application.

- **Active Cooling after every run**

After the completion of a microwave heating cycle, Milestone Cap top Vessel bottom recommends active cooling of each microwave vessel (while still in its individual rotor position or rotor segment). Active cooling is important because it reduces the time the vessel components are exposed to temperature and pressure extremes, which in turn minimizes the stress on the vessel components. Active cooling also allows faster access to prepared samples. Cooling strategies are discussed in the Rotor section, beginning on page 23.

- **Clean and dry between runs**

All external surfaces of vessel components should be clean and dry before they are reassembled and used for the next microwave sample preparation process. It is particularly important to eliminate any moisture or process residues from between component surfaces (between the vessel and safety shield, between the vessel cover and adapter plate, between the adapter plate and spring, etc.). Such trapped moisture or residues will accelerate the degradation of components during subsequent heating cycles.

- **Cleaning Vessels and Covers**

Under the influence of temperature and pressure, constituents of processed materials can adhere to or migrate into the inside surface of microwave sample preparation vessels. The chemical condition of the vessel(s) and cover(s) will directly affect one's ability to accurately and precisely prepare samples for chemical analysis. Milestone's TFM vessels and covers can be cleaned to any desired level of analytical sensitivity. There are several approaches that can be taken. One or more will best suite most applications. Backgrounds down to ppb (parts per billion) level, and lower can be obtained with reasonable effort.

Closed Vessel "Clean Blank Run" Method

Probably the simplest method for cleaning vessels and covers is to process a blank (acids or solvents) under the same process conditions (reaction parameter profile) that is used for preparing the analytical samples. How "clean"the vessels become will depend on the quality of the acids or solvents used. Successive "cleanings" may be necessary to achieve the desired low background.

Soaking Method

Another traditional method for cleaning vessels and covers is to soak them in an acid or solvent bath, at elevated temperature, for an extended period of time before they are rinsed, dried and stored. Typical conditions include 1:1 nitric acid and water in a Teflon container maintained at 80 - 90 degrees for an hour or

more. How "clean" the vessels become will depend on the quality of the acids or solvents used and the length of time at elevated temperature. Successive "cleanings" may be necessary to achieve the desired low background.
On occasion, successive cleaning may be carried out with alternative acid mixtures to target and remove specific contaminants.

TraceCLEAN Method
Unattended, automated vessel cleaning, to ppb levels, can be performed with Milestone's traceCLEAN system.
The traceCLEAN is a self-contained system that cleans vessels and covers using the condensation of continuously distilled acid vapors. The system uses nitric acid for the cleaning vapors. Detailed product information is available on the Milestone web site, at www.milestonesrl.com

3.3 Reference Vessel(s): Purpose

The reference vessel is a specialized vessel that facilitates reaction parameter monitoring (temperature and / or pressure) and allows feedback process control. To allow feedback process control, a reference vessel must be prepared to contain chemistry identical to any other sample vessel being processed during the same run.

In every Milestone rotor, the reference vessel contains a specialized cover design that is different from that of the other vessels in the rotor. The cover itself is different and, in the ATC-400 design, both the adapter plate and spring are different as well. The ATC-400 reference vessel cover specifically has a PTFE coated, closed end ceramic thermowell, for insertion of a temperature sensing probe.

In some reference vessel covers, a hollow, flexible, high pressure hose also is installed. This hose is for pressure monitoring and pressure feedback control.

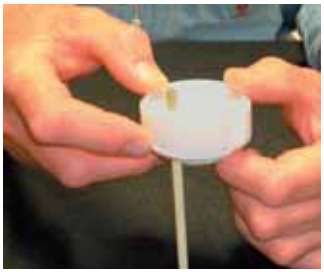
- **Assembling the ATC-400 Reference Vessel Cover**

Assembling the ATC-400 reference vessel cover involves installing a new thermowell along with its associated ferrule and, when appropriate, installation of the pressure sensor line (in reference vessel covers intended for use with pressure monitoring and feedback control).

Internal Temperature Monitoring and Control

The ceramic thermowell can be damaged by any twisting or bending motion. The reference vessel cover must be assembled following specific instructions and heeding appropriate cautions.

Whenever a ceramic thermowell is replaced, so should the Teflon ferrule be replaced.

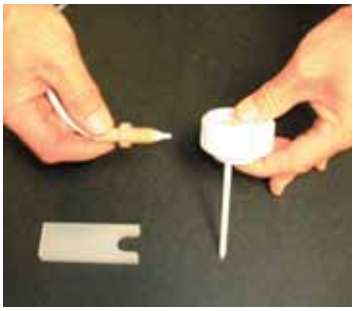


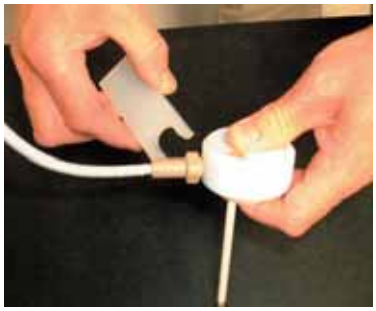
The open end of the ceramic thermowell is inserted up through the bottom of the cover by gently pushing down on the cover with the closed end of the ceramic against a hard flat surface. Downward force is gently applied until the open end of the ceramic

extends approximately one inch or more through the top side of the cover.
A new Teflon ferrule is first inserted into the threaded plug of the reference vessel cover, and then the ceramic thermowell is inserted through the ferrule into the plug, as far as it will go. Using the special cover tool, the plug and ceramic thermowell is gently pushed down until the plug meets the cover, using a counter clockwise motion.
Care must be taken not to cross-thread the soft TFM cover with the hard PEEK plug. Once the threaded plug contacts the cover, a complete counterclockwise turn followed by several clockwise turns of the tool will seat the ferrule and seal the ceramic in the cover. Care also should be taken not to over tighten the plug. Finger tight is sufficient. The adapter plate should sit perfectly flat in the cap.
After a newly installed thermowell has been used for the first time, Milestone recommends that the special cover tool be used to check the tightness of the threaded plug.

Internal Pressure Monitoring and Control

The hex nut plug of the flexible pressure tube is inserted into the tapped opening on the side of the reference vessel cover. Several clockwise turns of the hex nut will
secure the fitting, hand tight. A special slip wrench is used to complete the installation without over tightening the hex nut.





✓ **WARNING The threaded plug should never be adjusted while the cover is hot.**

- ✓ **WARNING The threaded plug should never be adjusted while the cover is hot.**

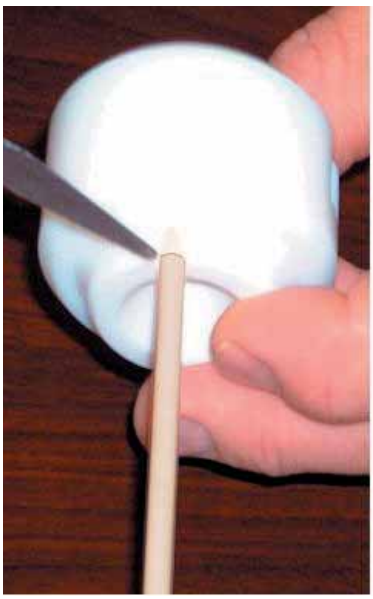
➢ Care of the Reference Vessel Cover

The reference vessel must be maintained in working order. An improperly used or maintained reference vessel may prevent correct
feedback of temperature/pressure information and interfere with control of the reaction conditions. Once the reference vessel cover is assembled, it should not be disassembled except to replace the thermowell or pressure line.

Safe storage of the reference vessel cover
Once assembled, the reference vessel cover must be protected from mechanical stresses that could damage the ceramic thermowell. By far the safest way to store the reference vessel cover is to fully assemble an empty reference vessel and secure it (without torquing) in a reference rotor segment or reference position in a monobloc rotor.

Vessel and cover as a set
A reference vessel cover will impress the sealing surface of a vessel differently from any impression made by a standard vessel cover. Standard vessel covers may not mate well to the sealing surface of a vessel once that vessel has been used with a reference vessel cover. Milestone recommends the components of the reference vessel assembly (cover, vessel, and shield) be handled as a set; the vessel should not be exchanged with any other vessel in the rotor nor used with any standard vessel cover.

Examining the Ceramic Thermowell - what to look for



Before each use of the reference vessel cover, the operator should examine the entire length of the thermowell looking for cracks. A crack will appear as a faint white
line around the circumference of the ceramic. Particular attention should be given to the area of the ceramic where it exits the bottom of the cover. The ceramic should be firm and inflexible in the cover.

Examining the Threaded Plug - what to look for

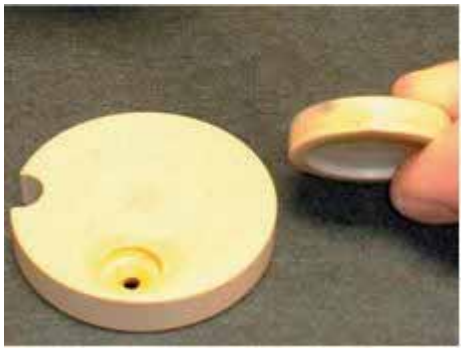
Before each use of the reference vessel cover, the operator should examine the top of the threaded plug looking for discoloration. This may indicate leakage of reaction vapors, under temperature and pressure, around the ferrule and plug. This may also indicate a cracked thermowell. The plug should be gently tightened and if the discoloration continues, the ferrule and thermowell should be replaced. In extreme cases, the cover will need to be replaced.

Examining the adapter plate

Before each use of the reference vessel cover, the operator should examine the under side of the adapter plate looking for discoloration. This discoloration may indicate leakage of reaction vapors, under temperature and pressure, around the ferrule and plug. This may also indicate a cracked thermowell. The plug should be tightened, and if the discoloration continues to advance, the ferrule should be replaced. In extreme cases, the cover will need to be replaced.

The operator should examine both sides of the adapter plate looking for stress cracks. If any cracks appear, no matter how small, the adapter plate must be replaced.

Cracks may begin to appear in—and radiate out from—the well in the plate that sits immediately above the threaded plug.



Examining the Spring

Before each use of the reference vessel cover, the operator should examine the spring for cracks and for proper shape. If any cracks appear, no matter how small, the spring must be replaced. The shape of the spring is checked for proper shape using the provided spring gauge. If the spring passes through the slot in the gauge, the spring must be replaced.

The spring should also be checked for discoloration.

Operating the reference vessel with a compromised thermowell or threaded plug can result in the spring being exposed to hot acid vapors. Marked discoloration or development of a spot on the perimeter of the spring is a clear indicator of a problem associated with the thermowell or plug.

Cleaning the reference vessel: Closed vessel "clean blank run" method

The only way to clean the reference vessel and assembled cover is to process a blank (using the same process acids or solvents) under the same process conditions (reaction parameter profile) that are used for the analytical samples.

How "clean" the reference vessel becomes will depend on the quality of the acids or solvents used. Successive cleanings may be necessary to achieve the desired low background.

Although the reference vessel can be cleaned using the soaking method or by using Milestone's traceCLEAN system, these methods cannot be used to clean the reference vessel cover. Care must always be taken to prevent any liquids or vapors from getting into the open end of the thermowell. Liquids inside the thermowell will affect the performance of the temperature probe.

Checking the pressure line for leaks: Discoloration

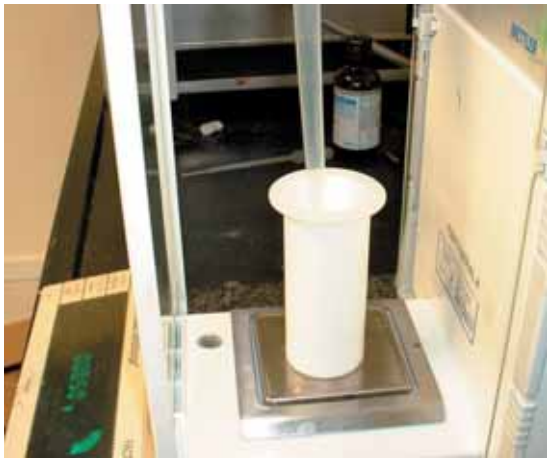
Before each use of the reference vessel cover, the operator should examine the hex nut plug of the pressure line looking for discoloration. This may indicate leakage of reaction vapors, under temperature and pressure, around the hex nut plug. The plug should be tightened using the slip wrench and if the discoloration continues, the line should be replaced. In extreme cases, the cover will need to be replaced.

Checking the pressure line for leaks: Discoloration
Before each use of the reference vessel cover, the operator should examine the hex nut plug of the pressure line looking for discoloration. This may indicate leakage of reaction vapors, under temperature and pressure, around the hex nut plug. The plug should be tightened using the slip wrench and if the discoloration continues, the line should be replaced. In extreme cases, the cover will need to be replaced.

Cleaning the pressure line
It is advisable to routinely flush out the pressure line installed in the reference vessel cover. This can be accomplished by heating water in the reference vessel without connecting the pressure line to the transducer in the ceiling of the cavity. To clean the line, one prepares the reference vessel with 20 mL of water, secures the reference vessel in its corresponding segment or position in the monobloc rotor, and executes a brief program that brings the water to the boiling point so that the generated steam will flush the line.
When this is performed with a reference segment, the single segment is placed in the cavity and the end of the pressure line is placed in a beaker to collect any materials expelled from the line during the cleaning process.
When this is performed with a monobloc rotor, the rotor adapter in the floor of the cavity should be temporarily removed and the end of the pressure line is placed in a beaker to collect any materials expelled from the line during the cleaning process.

Other Vessel Components
Information on the rest of the vessel components (vessels, covers, safety shields, springs, etc.) is found in the Performance Test section, beginning on page 39 of this manual.

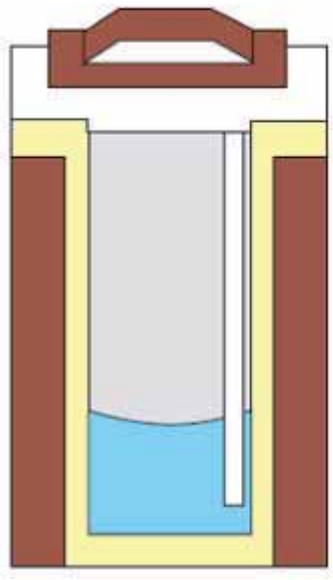
3.4 Loading samples (weighing samples and acids)



Sample materials, normally, are quantitatively introduced into a vessel using an analytical balance. Acid or solvent mixtures, normally, are quantitatively introduced into a vessel using an analytical balance or calibrated pipettes. Milestone recommends a four-decimal balance. Before introducing sample materials, an anti-static device should be used to eliminate any static electricity from the vessel. This will keep sample particles, which can cause hot spots, from sticking to the walls of the vessel.

- **Minimum Volume**

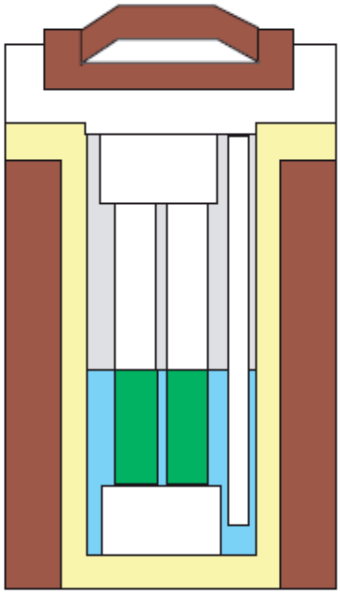
The volume of liquid in the reference vessel must be sufficient to submerge the end of the thermowell to a depth of at least one centimeter. In the standard 100 mL vessels, the minimum recommended volume is 8 mL at room temperature. More importantly, the thermowell must be submerged to a depth of at least one centimeter at the maximum temperature of the heating process.
This fact should be taken into consideration when using liquids with a high vapor pressure that will result in a substantial fraction of the liquid mass in the vapor phase at the maximum temperature of the heating process.



Small Volume Processing
Many applications require processing of small quantities / volumes. Milestone offers the SH3 micro sample vials and holder for processing olumes of 6 mL or less in a standard 100 mL vessel. Individual SH3 vessels are available in either TFM or quartz.
The SH3 vial assembly, loaded with one to three individual sample mixtures, is placed inside the standard vessel. A quantity of the same acid or solvent liquid is placed directly into the standard vessel, around the SH3 vials and holder, to a depth equal to the height of the liquid inside the vial(s). This is done for the reference vessel as well, with the thermowell sitting down in the pool of liquid, surrounding the vial(s) and holder, not with the thermowell inserted into any of the individual 6 mL vials.
Comprehensive information regarding use of the SH3 inserts is available.

- **Quartz Inserts - avoid static, hot spot samples, etc.**

Some sample types are problematic when working with TFM vessels and covers. Some samples (finely powdered materials) are notoriously prone to scatter due to static electric effects from the vessel material.



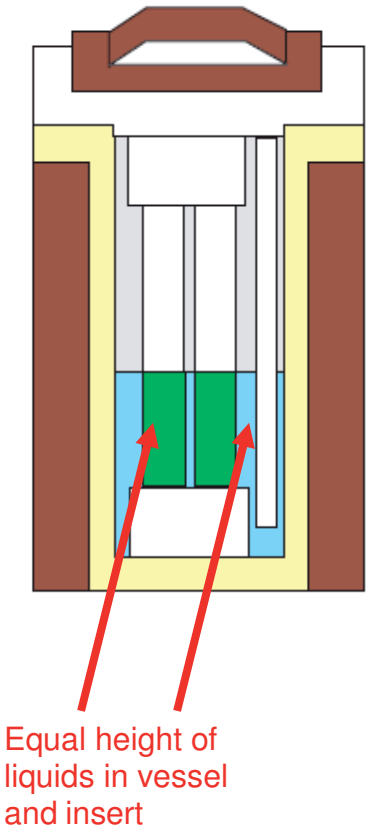
Other sample types are quite viscous and difficult to introduce into the bottom of the vessel. Some sample materials referentially absorb microwaves directly and can superheat, thereby damaging the vessels they are in contact with.

Milestone offers 30 mL quartz inserts with lids that can be used to handle problem samples.

The quartz material does not build up any static charge so they are very convenient when working with finely powdered samples. The quartz insert will not be damaged by localized superheating of any materials placed inside. As an additional advantage,

the quartz inserts have essentially no chemical memory effect.

Sample materials along with acid or solvent mixtures are introduced directly into the quartz insert and the insert is introduced into the 100 mL TFM vessel. An additional quantity of liquid is placed directly in the 100 mL TFM vessel, around the quartz insert, to a depth equal to the height of the liquid inside the quartz insert. This is done for the reference vessel as well, with the thermowell sitting down in the pool of liquid, between the microwave vessel and the insert, not with the thermowell immersed directly in the quartz insert.



Chapter 4
Rotor(s)

Every microwave sample preparation rotor works in conjunction with one or more vessel assemblies. A rotor provides: (a) the structure and support for securing the closed vessel assemblies, (b) a structure that facilitates the functioning of the vessel overpressure vent-andreseal technology, and (c) a platform for moving the vessel assemblies about inside the microwave cavity. As such, a rotor is a key component in safe and effective microwave sample processing.

4.1 Components: Monobloc Rotors

Each monobloc rotor is a system of components that consists of:

1. a machined polypropylene rotor body with positions for either six- or ten-vessel assemblies

2. six or ten large diameter pressure screws for securing vessel assemblies at each position

3. a removable PTFE disk (the insulating plate) in the floor at each vessel assembly position

4. a system of plastic ball wheels underneath the rotor body

4.2 Components: Segmented Rotors

Each segmented rotor is a system of components that consists of:

1. an assembled polypropylene rotor body with positions for either ten or twelve segments,

2. a maximum of either ten or twelve segments,

3. a large diameter pressure screw in the top of each segment for securing vessel assemblies,

4. a removable PTFE disk in the floor at each segment,

5. a system of plastic ball wheels underneath the rotor body

4.3 Care

Each monobloc or segmented rotor requires a certain level of care and attention to its components in order to: (a) maintain sample preparation performance, (b) maximize the useful lifetime of the individual components and (c) to ensure continued safe operation of the lab station. The exact lifetime of each component cannot be specified as it is a complicated function of the temperatures, pressures and chemistry of your specific application, “Your results will vary.”

- **Clean and dry: between runs**

All surfaces of rotor components should be clean and dry before they are reassembled and used for the next microwave sample preparation process. It is particularly important to eliminate any moisture or process residue between component surfaces (between the vessel and positioning disk, between the positioning disk and the rotor/segment body, between the threads of the pressure screw and the rotor/ segment body). Such trapped moisture or residues will accelerate the degradation of components.

- **Clean and dry: at the end of the day**

Opening vessels at the end of a microwave process will usually release pressurized reaction products. The released materials can contaminate the external surface of vessel components and various rotor components. These foreign materials should be removed from all surfaces to prevent accelerated degradation of the components.

The pressure screws in either style of rotor require particular attention.
A pressure screw should turn easily and without resistance using only one’s fingers. To maintain this performance, Milestone recommends that the pressure screws be removed from the rotor body or segments at the end of each work day and the screws and corresponding threads in the rotor/segment body be thoroughly rinsed and allowed to air dry over night before they are reassembled and used for the next day’s work.
No lubricants of any kind should be used on the pressure screws or in the corresponding threads of the rotor/ segment body. Lubricants will trap acid or solvent vapors that can attack the threads on subsequent runs.

Re-tapping pressure screw threads: limited lifetime extension
In the event that damage occurs to the pressure screws or corresponding rotors threads, several remedies are available:

1) Pressure screws can be replaced as consumables,
2) Milestone Service can use a special tapping tool to re-cut the threads in the rotor/segments body,
3) In extreme cases the rotor body or individual segments can be replaced as consumables.

4.4 Loading vessels into the rotor

Although it may seem obvious how a vessel assembly must be loaded into a rotor/segment body, several points should be noted.
Only fully assembled Milestone vessel sets (vessel, cover, shield, adapter plate, spring, indicator ring) should ever be used to process samples in a Milestone monobloc or segmented rotor. The use of the indicator ring (*fig. 1*) is recommended but is not required. Some choose to work without the ring to reduce assembly and cleaning time.
The indicator ring will not affect the performance of the microwave heated process.
When a vessel set is loaded into a rotor/segment body, care must be taken to ensure that the vessel set is centered on the white Teflon disk (insulating plate) in the bottom of the rotor/segment body at the particular position (*fig 2*).
This disk must be maintained in good condition. The disk must be replaced if any deformation of the disk edge occurs due to improper handling. The condition of this disk is particularly critical in the reference vessel position in the rotor/segment body.

4.5 Use of workstation

The workstation is a simple fixture for securing rotor/segment bodies while using the torque wrench. The workstation provides the necessary counter force to the leveraged force of the torque wrench.

As such, the workstation is an essential lab station tool.

The workstation has rubber feet for gripping the surface of the lab bench or fume hood. The workstation can also be effectively used on the work surface formed by the fully opened lab station door. Many operators elect to either clamp or permanently bolt the workstation in place in the fume hood.

A particular note is in order when using the workstation with rotor segments. There is a special version of the workstation or at least a special adapter to accommodate rotor segments. Each segment, when viewed from above, has a triangular shape.

✓ **WARNING** **In order to properly secure the segment for best advantage while using the torque wrench, the workstation/adapter should be positioned such that the apex of the segment (pointy end of the pie shape) is pointed directly away from the user. The apex should not be pointed directly toward the user.**

4.6 Proper Torquing

The torque wrench is an essential lab station tool. The ratcheting action of the torque wrench produces a calibrated radial force on the pressure

screws which is translated into a downward force on the vessel assemblies.
When used correctly, the torque wrench:
1) provides the necessary force to secure vessel assemblies in the rotor/segment body;
2) consistently facilitates proper deformation of the pressure relief springs, essential to the function of the vent and reseal pressure relief technology;
3) facilitates controlled opening of the vessels after the completion of the heating and cooling processes.

The torque wrench is best used with the rotor/segment body secured in the workstation on a firm level surface. The handle of the torque wrench should be grasped with one hand (near to the end) and the other hand should grip the rotor/segment body. Long rather than short movements of the wrench are best. Several long, smooth, continuous arcs of the wrench should be used to secure the vessel assembly until the wrench clicks.

4.7 Placing the rotor in the Lab Station

- **Installing a monobloc rotor**

To install a monobloc rotor, one merely places the rotor in the center of the cavity and then manipulates it back and forth until the rotor engages with the conical rotor adapter in the center of the cavity floor.
One can then activate the “Turntable” function from the software to observe that the rotor turns freely, and without binding.

- **Installing a segmented rotor**

With segmented rotors, the situation is different and somewhat easier.
It is standard procedure for the segmented rotor body platform to remain inside the cavity and for the individual segments to move in and out of the cavity as needed. One can activate the “Turntable” function from the software to advance the rotor to the corresponding position to place or remove the appropriate segment.
The polypropylene ring cover from the top of the rotor will need to be lifted, since this secures the individual segments in the rotor. Once the segment is positioned, the ring cover is again put in place.
One can process as few and as many (up to the maximum number) samples at a time as desired. When working with less than a full rotor, it is of particular importance that segments be properly secured in the segmented rotor body. If they are not, segments can: (a) tip over, (b) fall out of the rotor, or (c) cause the rotor to bind and fail to turn.

When working with less than a full rotor, it is preferred procedure to distribute the segments symmetrically about the rotor body. It is also recommended that empty segments (lacking a vessel assembly) be installed in the rotor to ensure perfect alignment of all the segments and uniform placement of the ring cover.

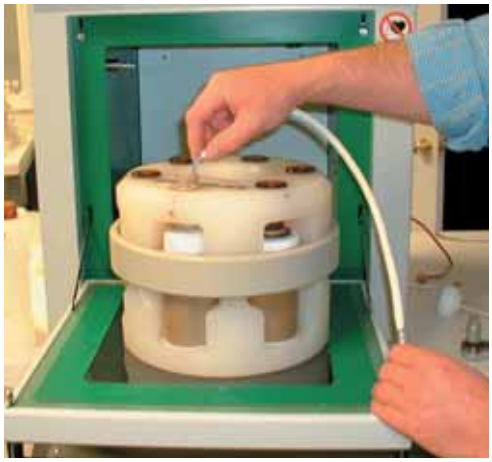
4.8 Handling the ATC-400-CE Probe

The ATC-400-CE temperature probe (thermocouple) is the monitoring and control heart of the lab station. As such, particular attention should be focused on its handling and use.
Care should be taken to avoid closing the cavity door on the probe as this may destroy the probe. When not being used, the probe should be detached from the electrical connection inside the cavity and safely stored.
One should note that the electrical connector has a definite shape and only one orientation of the connector will result in a positive connection. One should also note the black mark on the probe near the electrical connector as this will serve as a guide to the correct orientation when preparing to connect the probe.

- **Monobloc Rotors**

Milestone recommends inserting the temperature probe into the assembled reference position of the rotor *before* placing the rotor into the cavity. Failure to do this could damage the temperature probe.
One should be able to insert and withdraw the ATC-400-CE temperature probe from the assembled and torqued reference vessel position without any resistance. If there is any resistance the reference vessel should immediately be inspected for:

a) proper placement in the rotor;
b) good condition of all the vessel components;
c) good condition of the positioning disk in the reference vessel position of the rotor;
d) any deformation of the rotor body at the reference vessel position.



When the ATC-400-CE temperature probe is inserted, it should be inserted fully, giving a gentle twist of the knurled PEEK knob to snug it into the top of the rotor. Once secured in the rotor, the probe should be able to rotate freely inside the knurled knob, with the knob acting as a simple sleeve bearing.
Once the probe is secured in the rotor, the rotor can then be placed in the cavity.

- **Segmented Rotor**

Milestone recommends inserting the temperature probe into the assembled reference segment *before* the segment is placed into the cavity.
Failure to do this could result in damage to the temperature probe.
One should be able to insert and withdraw the ATC-400-CE temperature probe from the assembled and torqued reference vessel segment without any resistance. If there is any resistance the reference vessel should immediately be inspected for:

a) proper placement in the segment
b) good condition of all the vessel components
c) good condition of the positioning disk in the bottom of the segment
d) any deformation of the segment body.

When the ATC-400-CE temperature probe is inserted, it should be inserted fully, giving a gentle twist of the knurled PEEK knob to snug it into the top of the segment. Once secured in the segment, the probe should be able to rotate freely inside the knurled knob, with the knob acting as a simple sleeve bearing.
Once the probe is secured in the reference segment, the segment can then be placed in the cavity.

- **Positioning the reference vessel before starting**

Experience in the field indicates that the position of the reference vessel in the cavity at the start of a microwave process can affect the lifetime of the ATC-CE probe. It is believed that positioning the rotor such that the reference vessel is toward the back of the cavity, before plugging the free end of the probe to the electrical connector inside the cavity, (and before starting a heating cycle) can reduce mechanical stress along the length of the probe.
Once the electrical connection to the probe has been made, one should activate the Turntable function from the software and observe the smooth movement of the rotor, without hesitation or binding.

4.9 Handling the ATC-400-FO Probe

The ATC-400-FO temperature probe (fiber optic) is the monitoring and control heart of the lab station. As such, particular attention should be focused on its handling and use.
Take care to avoid closing the cavity door on the probe as this will destroy the probe. When not in use, the probe should be retracted through the top of the cavity and either stored in the top compartment or inserted into the safety holder attached to the chassis cabinet.
The fiber optic probe assembly has an integrated mechanical strain relief. Along the length of the fiber optic probe, there is an approximately one inch length of shrink wrapped black tubing. There is also a small, split conical piece of polypropylene that inserts into the top of the cavity. The split in this conical piece captures the black tubing and prevents the fiber optic probe from easily being pulled into the cavity and tangled due to the movement of the rotor. The strain relief must always be used. Failure to use it will reduce the life of the probe.

- **Monobloc Rotors**

Milestone recommends insertion of the temperature probe into the assembled reference position of the rotor before the rotor is placed into the cavity. Failure to do this could result in damage to the temperature probe.
One should be able to insert and withdraw the ATC-400-FO temperature probe from the assembled and torqued reference vessel position without any resistance. If there is any resistance the reference vessel should immediately be inspected for:

a) proper placement in the rotor
b) good condition of all the vessel components

c) good condition of the positioning disk in the reference vessel position of the rotor
d) any deformation of the rotor body at the reference vessel position.

When the ATC-400-FO temperature probe is inserted, it should be inserted fully, giving a gentle twist of the knurled PEEK knob to snug it into the top of the rotor. Once secured in the rotor, the probe should be able to rotate freely inside the knurled knob, with the knob acting as a simple sleeve bearing. Once the probe is secured in the rotor, the rotor can then be placed in the cavity.

- **Segmented Rotor**

Milestone recommends insertion of the temperature probe into the assembled reference segment before the segment is placed into the cavity. Failure to do this could result in damage to the temperature probe.
One should be able to insert and withdraw the ATC-400-FO temperature probe from the assembled and torqued reference vessel segment without any resistance. If there is any resistance the reference vessel should immediately be inspected for:

a) proper placement in the segment
b) good condition of all the vessel components
c) good condition of the positioning disk in the segment
d) any deformation of the segment body

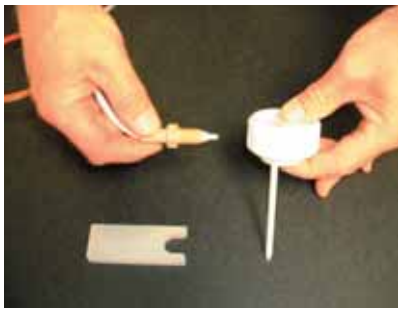
When the ATC-400-FO temperature probe is inserted, it should be inserted fully, giving a gentle twist of the knurled PEEK knob to snug it into the top of the segment. Once secured in the segment, the probe should be able to rotate freely inside the knurled knob, with the knob acting as a simple sleeve bearing. Once the probe is secured in the reference segment, the segment can then be placed in the cavity.

- **Positioning the reference vessel before starting**

Experience in the field indicates that the position of the reference vessel in the cavity at the start of a microwave process has no effect on the lifetime of the probe. It is believed that positioning the rotor such that the reference vessel is toward the back of the cavity, before starting a heating cycle, can reduce mechanical stress along the length of the probe.
Once the rotor/segment is installed in the cavity, one should activate the Turntable function from the software and observe the smooth movement of the rotor, without hesitation or binding.

4.10 Connecting APC line

Pressure monitoring and control requires that the APC option be installed in the Ethos lab station and that the appropriate reference vessel cover be available for use.
The pressure line needs to be attached between the reference vessel cover and the threaded pressure guide tube that extends through the ceiling of the cavity and connects directly to the pressure transducer.
The two ends of the pressure line are distinct.
The male end of the pressure line is installed in the reference vessel cover. Care must be taken to avoid cross-threading the hard peek fitting on the pressure line in the soft Teflon of the reference vessel cover. Finger tight is sufficient to begin with. The polypropylene slip wrench (provided by Milestone) should then be used to firmly secure the fitting without over stressing the materials.



- **Monobloc Rotor**

With a monobloc rotor, the temperature probe should be inserted into the assembled and torqued reference vessel, and the rotor should be positioned in the cavity before the female end of the pressure line is connected to the threaded pressure guide tube in the ceiling of the cavity.

- **Segmented Rotor**

With a segmented rotor, the temperature probe should be inserted into the assembled and torqued reference vessel, and the reference segment should be positioned in the cavity before the female end of the pressure line is connected to the threaded pressure guide tube in the ceiling of the cavity.

- **Precautions**

Working with a pressure monitoring reference vessel cover requires some additional attention.
The pressure line and fittings should be inspected before each run.

Care should be taken to note any is coloration of pitting of the peek connector at the reference vessel cover. This may indicate leakage and the need to adjust the fitting, replace the line, or replace the cover.
The peek fitting that connects to the pressure guide tube in the ceiling of the cavity should also be inspected for discoloration and pitting.
Similarly this may indicate leakage and the need to replace the line.
The pressure guide tube in the ceiling of the cavity must be inspected.
This threaded tube must be maintained to avoid problems with leakage and possible failure of the reference vessel. The polypropylene guide tube should be inspected for discoloration of the material and pitting on the surface of the threads. Damaged threads indicate the guide tube should be replaced.
Pressure monitoring can provide useful information to the operator, particularly during method development. Pressure monitoring can support feedback control of the microwave heating process, enhancing the operator's ability to control exothermic processes.
However, use of pressure monitoring can reduce throughput due to the requirement that the reference vessel must be cooled well below the boiling point of the reaction mixture before the pressure line can be disconnected from the pressure guide tube.
For this reason, operators will often use pressure monitoring during method development but only use temperature feedback control during routine processing of samples. This requires that some alternative to the pressure line be available.

Blind Plug

A reference vessel with temperature and pressure monitoring capability is provided with a blind threaded plug and a Teflon stopper, allowing one to remove the pressure line from the cover and to secure the opening in the cover. With the pressure monitoring port secured, the operator is free to use the reference vessel for temperature monitoring and control.

The blind plug and stopper are installed using the same slip wrench used to secure the pressure line in the cover.

Temperature Only Cover

A *far better* approach is to have on hand a separate reference vessel cover that provides only temperature monitoring capability. In this way, an operator uses one cover during method development and an entirely separate cover for routine processing. This avoids the possibility of cross threading pressure fittings in the cover, it also reduces the chance of leakage through the pressure port due to wear and tear of the cover threads.

4.11 Heating in a Microwave

The exact temperature profile used for processing a particular sample will depend on many things. For some samples, the required profile may be defined by regulation (EPA Methods). For most samples, the profile will be determined during method development.

4.12 Cooling

At the completion of every microwave heating process, the processed samples and the vessel assemblies that contain them need to be cooled down from the maximum temperature. This is done primarily to reduce internal temperatures and pressures to the point where the vessel assemblies can be safely handled, opened, and the processed samples accessed. This is also done to relieve the temperature and pressure stress on the vessel assemblies, as mentioned above.

- **End of Run Cooling**

Every microwave heating process should conclude with a period of time where the microwave cavity is dynamically ventilated prior to opening the door and removing any rotors or vessels. This is routinely accomplished by including a VENT period in the microwave program. In this regard, VENT does not mean opening the vessels, rather it means “VENTilation” of the cavity by the exhaust module.
Alternatively, one can manually turn on the exhaust module at anytime after the completion of a microwave program.

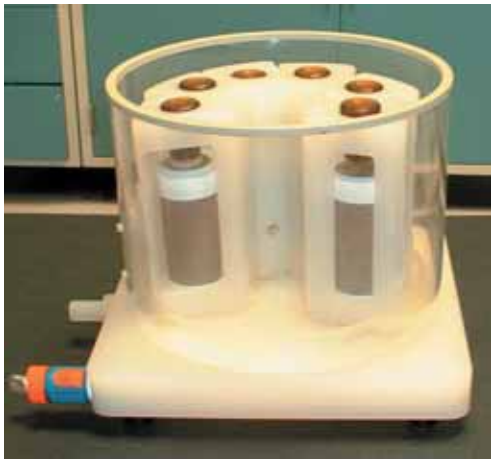
- **Enhanced Air Cooling**

Milestone has available a “Enhanced Air Cooling Kit”. This kit contains adapters for the Ethos lab station that direct the flow of air through the top of the Ethos lab station and around vessels in segmented rotors. Using this kit can provide more effective air

cooling after the completion of the microwave heating process, but it is not as effective as water cooling.

- **Water Cooling**

Milestone's Segmented Rotor segments and monobloc rotors can be effectively water cooled at the completion of the microwave heating process. Water cooling can be accomplished by using the standard cooling station (available from Milestone) or by simply placing the monobloc rotor or individual segments into any convenient container that can hold water. A lab sink filled with water, so that the vessels are immersed up to the bottom of the indicator ring (a little more than three-quarters up from the bottom of the safety shield) is appropriate for the purpose.
minutes in a water bath is sufficient to reduce the internal temperature well below the normal boiling point of acid mixtures. Longer cooling times may be necessary when working with volatile elements or low boiling solvents.



4.13 Removing Vessels/Rotors from the cavity

Immediately after the completion of a microwave heating program, rotor segments or a monobloc rotor can be removed from the cavity.
Non-reference segments can be removed from the rotor without additional concern. However, care must be exercised in handling any temperature probes or pressure lines associated with the reference segment or with the monobloc rotor.

➢ Disconnecting the Temperature Probe

The temperature probe must be carefully attended to when removing a reference segment from the cavity.
If the ATC-FO (fiber optic) probe is being used in a segmented rotor, the rotor should be advanced to the front position. The rotor cover is then lifted to allow removal of the reference segment. With the reference segment sitting on the door the probe is then detached from the reference segment and retracted up through the cavity for safe storage in the compartment above the cavity.

If the ATC-FO (fiber optic) probe is being used in a monobloc rotor, the reference position should be advanced to the front position. The entire rotor is then lifted out of the cavity and moved out onto the door. With the rotor sitting on the door, the probe is then detached from the reference position and retracted up through the cavity for safe storage in the compartment above the cavity.
If the ATC-CE (thermocouple) probe is being used in a segmented rotor, the probe should first be disconnected from the cavity wall. At this point the alarm will sound. The alarm will stop if the instrument is shut off or if the shorting plug is installed in the connector.
Advance the reference position to the front position. Next, lift the rotor cover to allow removal of the segment. With the segment sitting on the door, the probe is then detached from the segment.
If the ATC-CE (thermocouple) probe is being used in a monobloc rotor, the probe should first be disconnected from the cavity wall. At this point the alarm will sound. The alarm will stop if the instrument is shut off or if the shorting plug is installed in the connector.
The reference position should then be advanced to the front position.
The entire rotor is then lifted out of the cavity and moved out onto the door. With the rotor sitting on the door, the probe is then detached from the reference position and retracted up through the cavity for safe storage in the compartment above the cavity.

➢ Disconnecting the Pressure Line

Operating with pressure monitoring imposes additional conditions and handling requirements. At the completion of a heating cycle, the reference vessel must cool well below the boiling point of the mixture, before the pressure line can be disconnected from the pressure guide tube.
If using a segmented rotor, all other segments in the rotor can be safely removed from the cavity without delay, at the completion of the to begin water cooling while the reference segment continues to cool.

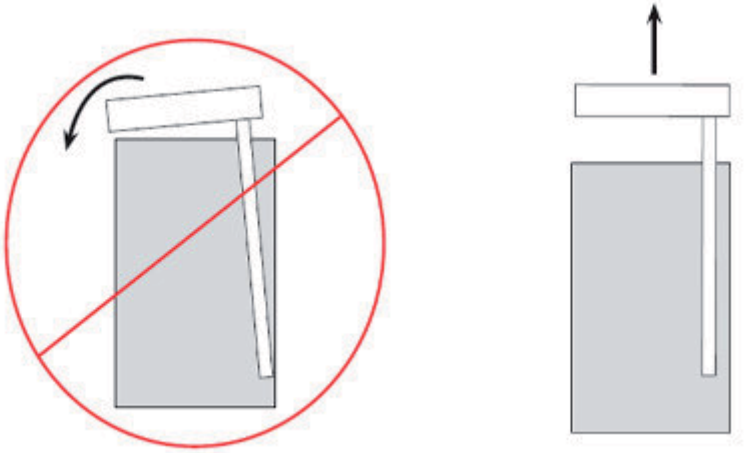
If using a monobloc rotor, none of the other vessels can be removed until the reference vessel cools in the cavity.
With some effort, one can safely move the reference segment out of the cavity and into a container of water on the door, with the pressure line still attached, and thereby accelerate the rate of cooling.

a) To begin with, one should manually turn on the exhaust module and have it running while disconnecting the pressure line. Also, one must be aware that small droplets of acid may be present at the open end of a pressure tube so protective gloves should always be worn.

b) The knurled fitting on the end of the pressure line, that
connects to the guide tube in the ceiling of the cavity, is slowly loosened and observe the pressure reading displayed on the controller is monitored until the pressure is completely released.

c) The open end of the pressure line should be covered (with a paper towel) to collect any droplets while removing either the reference segment or the entire monobloc rotor from the cavity.

4.14 Opening vessels (slow and gentle, avoid vapors)



Microwave vessels should never be opened before they have been sufficiently cooled. Doing so will not only eject hot acid vapors from the vessels but it will also reduce the useful life of the vessel components.
The rotor or segments should be secured in the workstation in a fume hood. When opening vessels, very short movements of the torque wrench are best. With each short turn of the pressure screw, one listens and looks for release of process vapors. With each release of vapors, one should pause briefly until the release stops, and only then should the pressure screws be given an additional turn. This is repeated until no additional pressure releases are noted.

Chapter 5
Periodic Performance Tests

A Milestone Microwave Labstation is designed to provide many years of high performance and superior results, processing samples for a variety of applications.

Everything that is placed inside a Milestone Microwave Labstation is considered to be consumable. The exact lifetime of each component cannot be specified as it is a complicated function of the temperatures, pressures and chemistry of your specific application. In other words, “your results will vary.” For optimum performance and continued safety of the Microwave Lab Station, consumable components must be replaced when they have reached the end of their useful life.

There are prescribed tests and examinations that can be performed to periodically evaluate the condition of most consumable parts and accessories. How often these tests are performed will depend on your usage of the system.

Milestone recommends that the components of each individual vessel assembly be segregated as a set. An effort should be made not to mix components between runs, during cleaning and/or during storage. Vessels and covers, in particular, should share a common history. This will facilitate maximum useful lifetime; maximize performance for routine operation at the extremes of vessel specifications; and ease identification, isolation and replacement of worn components when they begin to affect sample preparation performance.

5.1 Microwave Output Power Test

A simple test can be performed to determine the output power of the magnetrons. Occasions when a measurement of the output power may be performed include:

1) the initial installation of the labstation
2) sudden changes in sample processing performance

3) relocation and installation of the labstation
4) routine laboratory procedures, etc.

The output power is determined by measuring the temperature change of a 1.0 litter container of water. Use a low, wide microwave transparent container and place it in the rear of the cavity. In systems with a thermocouple temperature probe, the thermocouple should be removed and the jumper plug installed. In systems with a fiber optic temperature probe, the fiber optic probe should be withdrawn up through the top of the cavity but left connected to the electronics.
Stir the water and measure the initial temperature with an accurate thermometer. Heat the water at maximum power for 60 seconds.
Immediately after, stir the water and measure the maximum final temperature of the water.

output power = (Tf - Ti) x 70

5.2 Vessel/Cover Inspection

Milestone recommends that all vessels and covers be inspected periodically, as a routine part of labstation care. For a labstation that is used daily, inspection of vessels and covers should be performed once a month.

- **Fit and Finish**

A new vessel and cover will have a snug fit. After they have been used, even once, the fit will relax but vessel and cover will still form a seal when assembled. Poorly fitting vessels and covers will have reduced pressure handling capability.

Lateral movement of cover
To examine the vessel and cover for proper fit, the vessel is placed on a firm surface, the cover is put on the vessel and then an effort is made to move the cover back and forth in the vessel. Any lateral movement of the cover indicates a poor fit and suggests the need to replace the vessel or cover or both.

Fit inside safety shield
A vessel should fit easily, without resistance, inside a safety shield. Any vessel that does not easily pass fully into a shield should be replaced. Vessels can become misshapen, after a microwave heating process, if they are removed hot from a shield, before they have sufficiently cooled.

Cuts, gouges, or "troughing"
Any cuts, gouges or "troughing" (loss of material) on the inside surface of the vessel or on the sealing surfaces of the vessel or cover, indicates the need for replacement.
"Troughing" is usually the result of sample materials sticking to the inside surface and super heating under the application of microwave energy.

Discoloration
A new vessel and cover are pure white. Discoloration of the vessel material with age is normal. The gradual infiltration of NOx vapors into the matrix, (in digestion applications), will result in a diffuse red/brown coloration.
This will have no effect on the analytical results.

Wash and dry
Failure to wash and dry vessels and shields between uses will eventually lead to degradation of the shield material and in the worst case situations some of the shield material will become impregnated into the outer wall of the microwave vessel. Such vessels and shields should be removed from service and replaced.

5.3 Shield inspection

Milestone recommends that all shields be inspected periodically, as a routine part of labstation care. For a labstation that is used daily, inspection of shields should be performed once a month.

- **Fit and Finish**

A new shield will be light brown in color with a smooth, hard surface, inside and out. There should be no pits or cracks.

As the shield ages through routine usage, the color will gradually hange with yellow highlights appearing anywhere the shield is exposed to acid vapors. This is normal.

Pitting
Excessive heat, spillage of acid, failure to wash a shield between uses will lead to pitting of the surface, particularly near the top around the vessel opening. Provided that the surface of the shield that mates with the open end of the vessel is smooth and flat, providing a firm support for sealing the vessel against the cover, then a mildly pitted shield can usually continue to be used. Continued pitting will eventually lead to shield failure.

Wash and dry
Failure to wash and dry vessels and shields between uses will eventually lead to degradation of the shield material and in the worst case situations some of the shield material will become impregnated into the outer wall of the microwave vessel.
Such vessels and shields should be removed from service and replaced.

Chips or cracks
Chips or cracks may appear on well used or severely stressed shields. A shield is inspected for chips by holding it to the light and examining the external surface. Chips may occur when a shield is dropped on a hard surface.

Stress cracks
A shield is inspected for stress cracks by holding it to the light and examining the inside surface as well as the outside surface for hairline fissures, tracks or splits in the surface. At the first sign of any hairline fissures, tracks or splits in the surface, the shield must be taken out of service and replaced.

5.4 Spring Inspection

Milestone recommends that all springs be inspected periodically, as a routine part of labstation care. For a labstation that is used daily, inspection of springs should be performed once a week.

➢ Fit and Finish

A new spring will be light brown in colour with a smooth, hard surface, top and bottom. There should be no pits or cracks. A high pressure spring (100 bar limit) will weigh approximately 4.8 grams while a medium pressure spring (30 bar limit) will weigh approximately 3.8 grams. The weight of the spring does not change noticeably during its useful life.
In general, the lifetime of a spring will be extended by: a) never storing an assembled vessel under torque, b) always cooling vessels and rotors after completion of the microwave heating cycle and c) releasing the vessel assembly from the rotor once it has cooled.

Discoloration
As the spring ages through routine use, the color will gradually change with yellow highlights appearing anywhere the spring is exposed to acid vapors. This is normal.

Cracks
Cracks may appear on well used or severely stressed springs. A spring is inspected for cracks by holding it to the light and examining the top and bottom surface. Cracks may occur when a spring is routinely exposed to high temperatures for extended periods of time, or when the reaction chemistry results in exposure to chlorine vapors (from use of aqua regia) or fluorine vapors (from use of hydrofluoric acid). If any cracks appear, no matter how small, the spring should be removed from service and replaced.

➢ Spring Test

A spring is checked for proper shape using the provided spring gauge. If the spring does not pass through the slot in the gauge, then it is still good and can be used. Any spring that passes through the slot in the gauge must be removed from service and replaced.

5.5 Adapter Plate inspection

Milestone recommends that all adapter plates be inspected periodically, as a routine part of labstation care. For a lab station that is used daily, inspection of adapter plates should be performed once a week.

- **Fit and Finish**

A new adapter plate will be light brown in color with a smooth, hard surface, top and bottom. There should be no pits or cracks.

In general, the lifetime of an adapter plate will be extended by:

a. never storing an assembled vessel under torque
b. always cooling vessels and rotors after completion of the microwave heating cycle
c. releasing the vessel assembly from the rotor once it has cooled.

Discoloration

As the adapter plate ages through routine usage, the color will gradually change with yellow highlights appearing anywhere the adapter plate is exposed to acid vapors. This is normal.

Cracks

Cracks may appear on well used or severely stressed adapter plates. An adapter plate is inspected for cracks by holding it to the light and examining the top and bottom surface. Cracks may occur when an adapter plate is routinely exposed to high temperatures for extended period of time, or when the reaction chemistry results in exposure to chlorine vapors (from use of aqua regia) or fluorine vapors (from use of hydrofluoric acid). If any cracks appear, no matter how small, the adapter plate should be removed from service and replaced.

5.6 Rotor Inspection

Milestone recommends that the rotor body (and segments) be inspected periodically, as a routine part of lab station care. For a lab station that is used daily, inspection of the rotor body (and segments) should be performed once a month.

➢ Fit and Finish

A new rotor body is white in color with smooth, machined surfaces, inside and out. With age, the white polypropylene rotor body will take on a yellow or brown coloration due to exposure to acid vapors. This is normal and does not affect the performance.

Free movement

The rotor should turn freely, in either direction, when using the carousel motor. The roller balls beneath each rotor body should be free and easy to move by hand. Care should be taken to keep the roller balls and the tracks free from debris. Failure to keep the balls clean will lead to binding of the rotor which can cause inconsistent results.

Monobloc rotors: flat and true

A monobloc rotor should be flat and true top and bottom.
When placed top side down on a smooth hard surface, such as a lab bench, a monoblock rotor body should lie flat without any rocking back and forth. Excessive process temperatures and failure to cool the rotor immediately after a microwave heating process is complete can result in warping of the rotor.

Segmented rotors: flat and true

Individual segments should be flat and true, top and bottom. When placed top side down on a smooth hard surface, such as a lab bench, a rotor segment should lie flat without any rocking back and forth. Excessive process temperatures and failure to cool the rotor, immediately after a microwave heating process is complete, can result in warping of the segments.

Pressure screws

The pressure screws should turn freely in the rotor block (or segment) using only finger force. The pressure screws and corresponding rotor threads should be cleaned regularly to maintain this performance.

Chapter 6
Recommended Spare Parts

It is impossible to predict the useful lifetime of consumable components. Over the course of several months use of a new Milestone Microwave Labstation, the operator can expect to develop a consumables history.

Although Milestone stocks consumable components for timely shipment in response to customer orders, we can also identify and recommend appropriate spares to keep on hand.

Initially, and until they develop a consumables history, Milestone recommends new customers acquire and store one complete vessel assembly consisting of a shield, vessel, cover, adapter plate and spring.

Certain consumable components can be critical to performing a particular application or to the continued safe operation of the labstation. Milestone recommends that all customers acquire and store the following items:

For systems configured for internal temperature (only) monitoring and control:

- 1 spring for the reference vessel (P/N 050184)
- 1 adapter plate for the reference vessel (P/N 050183)
- 1 thermowell and ferrule for the reference vessel cover (P/N 055135)
- 1 temperature sensor (depending on system configuration, either thermocouple [P/N 050214] or fiber-optic [P/N FO00110])

For systems configured for internal pressure monitoring and control:

- 1 spring for the reference vessel (P/N 050184)
- 1 adapter plate for the reference vessel (P/N 050183)
- 1 thermowell and ferrule for the reference vessel cover (P/N 055135)
- 1 temperature sensor (depending on system configuration, either thermocouple [P/N 050214] or fiber-optic [P/N FO00110])
- 1 pressure line (connects to the reference vessel cover) (P/N 933635)

- 1 pressure guide tube with O-ring (threaded stem on the pressure transducer) (P/N 933636)
- 1 complete temperature-only reference vessel cover (P/N 050211)

For systems with monobloc or segmented rotors:

- 1 vessel insulating disk for the rotor (High pressure 6 or 10 position rotor: P/N 35119. Medium pressure 12 position rotor: P/N 35197).

Chapter 7
Method Development

Method development is as much a philosophy as it is a procedure. As with any laboratory instrument, how one approaches microwave method development will have an impact on success and satisfaction.

7.1 Where to Start

Several resources can be suggested where information and guidance can be obtained when developing new methods for processing (digestion or extraction) samples in the microwave labstation.

- **Existing Chemistry**

The first place to look is at existing chemistry. If chemistry has been developed to successfully process a sample by other than microwave techniques, then that essential chemistry should be the starting point for microwave method development. If the chemistry works by conventional sample processing techniques then chances are it will work as well and faster when using microwave techniques with only minor adjustments.

- **Existing Microwave Methods**

Milestone is constantly adding new methods to its growing list of real world samples successfully processed by microwave techniques. Each new customer is provided with a copy of the appropriate sample preparation “Application Report Book” which details information on a variety of samples, the chemistry used and the microwave heating process that successfully prepared the samples for analysis.

- **Literature**

The following book is the second edition of an excellent work describing both the history and state-of-the-art for this technology.
“Microwave-Enhanced Chemistry, Fundamentals, Sample Preparation, and Applications”, H.M. (Skip) Kingston, Stephen J. Haswell, American Chemical Society, Washington, DC, 1997. ISBN 0-8412-3375. This can be ordered up from Milestone, Inc. or directly from Oxford Press, which distributes ACS publications.

- **Online**

Another good source of real world information can often be obtained from the participants on the PLASMACHEM Server, operated by Syracuse University. Enrollment in the service is free, and can be obtained by sending mail to: plasmachem-l@listserv.syr.edu (that's a lower-case "L" after the hyphen, not a numeral "one"). The site hosts a lively exchange of information between chemists skilled in the art.

7.2 Key Concepts

Method development for microwave sample preparation is not unlike method development for other instruments and techniques. A few points are useful to keep in mind as one approaches developing and optimizing methods. It is important, first of all, that the language be clear.

"Total digestion" refers to processes that result in a clear, precipitate free solution that is stable on standing at room temperature. In a total digestion, the original matrix is necessarily destroyed, thereby liberating all possible elemental constituents. Every constituent of the original matrix is a potential analyte and may require post digestion adjustment of the chemistry to achieve the desired result.

Leach or extraction refers to a process that liberates all analytes of interest from the surface of the sample matrix but does not necessarily result in a clear, precipitate free solution. The original matrix is not necessarily destroyed. Full recovery of all analytes of interest is the key to determining the effectiveness of the leaching process.

- **Reasonable sample size**

A common goal is to process a sufficiently large sample to yield reliable precision in the analysis.

The maximum allowed sample size will depend on the composition of the sample and the process being used. A leach or extraction will always allow larger sample sizes than a total digestion of the same material due to the reaction products created during a digestion.

High organic content samples will generally allow digestion of a maximum of 0.5 to 1.0 grams. Special techniques (use of quartz inserts, pre-digestion in an MMR, etc.) may allow larger sample sizes to be digested successfully.

When attempting to maximize the digestion sample size, Milestone recommends that one proceed iteratively, processing a small sample (0.25 g) and repeat the process each time increasing the sample size

by an additional 0.25 g). Monitoring pressure in the reference vessel will provide insight into the reaction dynamics.

- **Chemistry that works**

Successful microwave processes require an intimate knowledge of the sample matrix and the analytes of interest. Several references are provided above where one can obtain suggested chemistry for attacking a sample. These resources are no substitute for a good chemical education and are certainly no substitute for the knowledge and insights available from others who have worked with similar samples.

- **Stoichiometric quantities**

A fundamental feature of closed vessel microwave processing is the fact there is no loss of volatile species during the process. Also, far less volume of acids or solvents is required than for open vessel techniques where some volume is always lost to the environment.
Closed vessel microwave digestion techniques allow stoichiometric quantities of reactants to be used. Of course, this requires even more knowledge of the sample composition.

- **Solubility Issues**

Stoichiometry is not the only issue to focus on. In fact, many problems with method development occur when operators fail to account for the solubility of either the matrix or the analytes of interest. One must provide sufficient volume of acids or solvents to maintain solubility at the elevated process temperature as well as at room temperature.

- **Process Temperature and Time**

As part of method development, one should endeavor to determine the lowest temperature and the shortest duration at that temperature which will result in successful processing of samples. High, sustained process temperatures result in shortened useful life of vessel and rotor components.

- **Process Pressure and Time**

As part of method development, one should endeavor to fully contain the reaction products in the vessel, without venting. One may find that the sample size needed to obtain the desired analytical sensitivity requires the use of alternative vessel types and performance ratings.
Pressure should generally be thought of as an artifact of the heating process, not as a parameter for process control. By this is meant that temperature, rather than pressure, drives the chemistry of a reaction

and it is the control of process temperature that will ensure uniform sample processing, regardless of sample size or the number of samples processed simultaneously.

- **Sample Stirring**

Stirring the sample during the microwave heating process can have benefits for particular samples. Stirring ensures a uniform reaction mixture. This will help speed the reaction, particularly for solvent extraction or for digestion of finely ground sample matrices. Stirring can effectively prevent sample materials from adhering to the vessel bottom or walls. This is important when processing sample materials that are either quite viscous or are strong absorbers of microwaves.
Stirring also can prevent “floaters” (sample materials that initially float on the surface of the reaction mixture) from adhering to the vessel wall.

Appendix

Customer Support

Milestone's role as an industry leader is commitment to providing innovative, reliable and easy-to-use laboratory instruments is surpassed only by our very knowledgeable and experienced staff. Our chemists, applications specialists, technical support team and office representatives are dedicated to making your business and chemistry successful.
If you have any questions about tips, techniques, applications, support, etc., please contact your local representative, or call or email Milestone's Customer Support Team:

tel: +39 035 570186
e-mail: marketing@milestonesrl.com

We are also in constant contact with other Milestone Applications Labs around the world, who, along with our staff, have compiled one of the largest and most diverse libraries of microwave methods available.
With over 20 years of microwave experience behind us, Milestone has the knowledge and resources to help you optimize your instrumentation and your chemistry for the best possible results.