MILESTONE
HELPING CHEMISTS


# PERFORMANCE CHARACTERISTICS OF THE MILESTONE ULTRAWAVE

**The performance and benefits of Single Reaction Chamber (SRC) technology.**

## | INTRODUCTION

The ultraWAVE with Single Reaction Chamber (SRC) technology overcomes many of the limitations of conventional microwave sample preparation systems. For this reason, it has found a place in a wide array of market segments, such as contract, production and research laboratories in the pharmaceutical, polymer, metallurgical, geological, environmental and consumer product industries.

With the ultraWAVE, there is no requirement to batch samples with a similar matrix. As a result, all sample types can be digested at the same time. Additionally, the high temperature and pressure capabilities of the technology allow even the most difficult organic matrices to be digested in the minimum amount of time. With the nitrogen-pressurized capping system, samples of widely different analyte concentrations can be positioned next to each other in the chamber without concerns of cross-contamination.

Milestone offers a wide range of racks and vials, including racks that accommodate vials with different volume types to digest different sample types and amounts in a single microwave program, enhancing the lab workflow and productivity.

As a result, the real-world benefits of the ultraWAVE microwave digestion system preparing samples for trace element analysis are truly “Any Sample at Any Time.”

## | EXPERIMENTAL

This technology report describes how widely differing sample types can be digested together without cross contamination, and complete recovery of volatile analytes such as Hg is achieved. A comparison of digestion blanks obtained from a variety of different vial materials, in non-cleanroom conditions is also made.

## INSTRUMENT

The ultraWAVE meets the requirements of modern laboratories, thanks to its unique benefits, such as:

- Superior digestion quality
- High productivity
- Ease of use
- Superb safety

*Figure 1 – Milestone's ultraWAVE*

Developed by Milestone, the ultraWAVE with Single Reaction Chamber (SRC) technology is designed with a 1 Liter reactor, capable of operating at very high temperature and pressure (300 °C and 199 bar, respectively). This capability ensures complete digestion of even the largest sample sizes as well as highly reactive and difficult-to-digest samples.

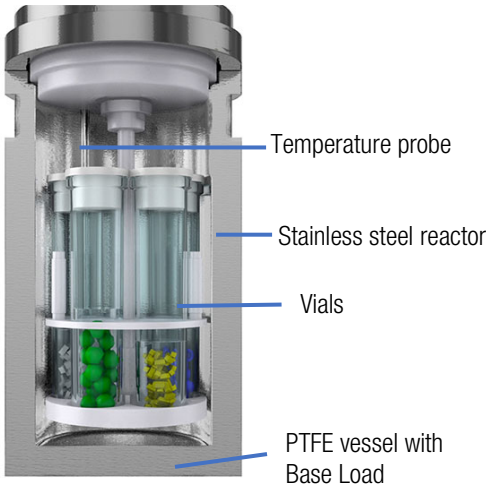


*Figure 2 – Schematic of the ultraWAVE's single reaction chamber (SRC)*

For the first time, a microwave digestion system ensures the same temperature and pressure conditions in all positions, even when different samples and/or different chemistries are used. This results in superior digestion capabilities, higher productivity and better workflow for the lab. The ultraWAVE's base load and positive pressure load prior to heating generate an equilibrium of temperature and pressure in each position, thus avoiding sample/element loss and cross contamination. Samples can be weighed directly into disposable glass vials, eliminating the cleaning steps. Sample throughput compared to closed-vessel digestion is approximately 2x higher. Labor and consumables costs are significantly reduced over traditional microwave digestion. However, since the sample prep workflow using SRC technology is quite different to the workflow with traditional

closed-vessel digestion, it is important to consider possible analytical implications.
For example, glass vials are not as clean as quartz or TFM. This technology report compares the blank contribution from glass, quartz and TFM vials, in order to assess the suitability of glass vials for ultratrace applications. Because all samples and blanks are placed into a single chamber and digested together (vials are capped, the caps are loose fitting to prevent pressure build up in the vial), this note also investigates the possibility of cross contamination between samples and loss of volatile elements during the digestion program.

## | BLANK CONTRIBUTION FROM DIFFERENT VIAL MATERIALS

To determine the digestion blank contribution from the three different vial materials, a digestion run was performed with a 15-position rack containing 15 mL vials of each material type. The quartz and TFM vials were precleaned by soaking in concentrated $HNO_3$, while the glass vials were used uncleaned. Ultrapure concentrated $HNO_3$ (4 mL), was added to each vial, and the ultraWAVE ramped to 220 °C in 15 minutes and held at 220 °C for 15 minutes. After cooling, the acid was diluted to 40 mL with DI water. TFM caps were used with all vials. Analysis was performed on an Agilent 7500cx, with a standard glass Micromist nebulizer and quartz Scott-type spray chamber and Ni cones. The analyte list comprised of 26 of the most common analytes. Both prep and analysis were performed in a regular lab environment. As expected, the glass vials were found to give the highest blanks, but only for a relatively small number of elements. The highest elements measured in glass were: Na 525 ppb, B 120 ppb, K 113 ppb, Al 60 ppb, Ca 13 ppb and Mg at 4.3 ppb. The remaining elements were ppt level. For the other vial materials, B and K were found at the single figure ppb level in both quartz and TFM, though B was likely present in the DI water, and K is an environmentally common element.
Figures 3 and 4 show the data in graphical form – Figure 3 has a maximum concentration scale of 100 ppb, while Figure 4 is the same data displayed with a maximum concentration scale of 10 ppb.

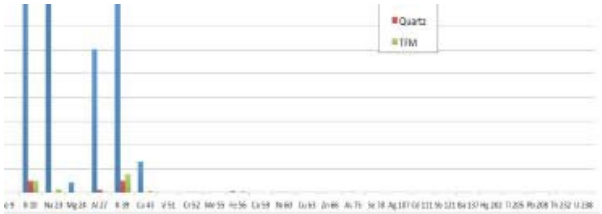


*Figure 3. Digestion blank by vial material*

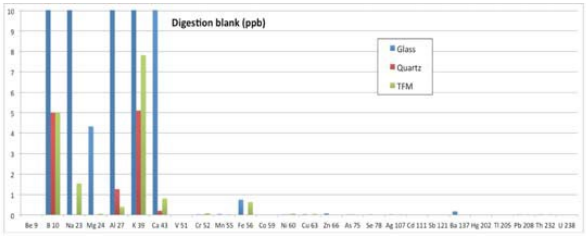


*Figure 4. Digestion blank by vial material (expanded scale)*

Clearly, if ultratrace analysis in digested samples by ICP-MS is to be performed for the above mentioned elements Na, B, K, Al, Ca and Mg, then either quartz or TFM vials should be used. However, B is not a commonly required analyte and the other analytes listed are all present at high levels in most routine sample types, so for many applications, glass vials are acceptable.

And in fact, for USP method <233>, none of the above listed analytes are required to be measured. What this study also demonstrates is the ability of the ultraWAVE – especially with quartz and TFM vials - to produce digestates with extremely low blank levels, making it equally suitable as a sample prep technique for ICP-MS as well as ICP-OES.

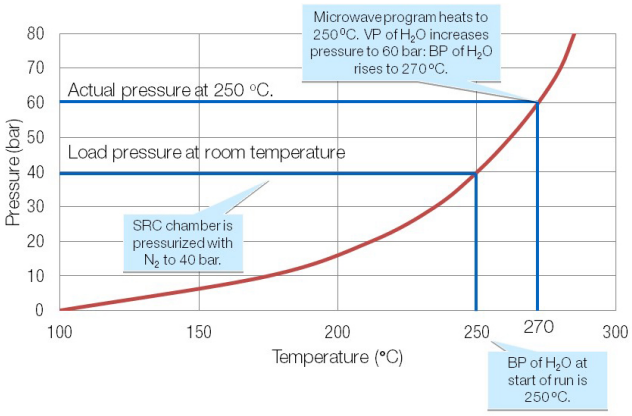


*Figure 5. Boiling point of water with increased pressure*

## | ENSURING FULL RECOVERIES

The reason cross contamination or loss of volatile analytes does not occur in the SRC is due to the effect of pressure increase in the chamber. Prior to the start of the microwave program, the chamber is pre-pressurized with $N_2$ – typically at 40 bar. This pressure acts as a cover over the samples. As the samples heat up, their vapor pressure further increases the pressure in the chamber, which increases the boiling point of the samples, so the samples never boil. Figure 5 illustrates the initial pre-pressurization of 40 bar increases the BP of water to 250 °C. Assume the microwave program heats the sample to 250 °C. As microwave energy is applied, the sample heats up, which increases its vapor pressure, subsequently increasing the pressure in the chamber. At the target run temperature of 250 °C, the increase in pressure due to the vapor pressure increase in the sample is 20 bar. Added to the original pressure of 40 bar, this gives 60 bar pressure in the chamber. However, at 60 bar, the BP of water is 270 °C – so the sample never boils. Because the sample never boils, no volatiles are lost and no physical transport of sample between vials can occur.

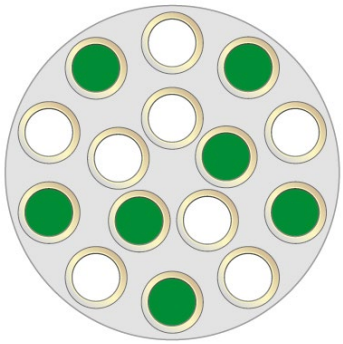

*Figure 6. Schematic of vial rack (15 positions) White vials: blanks. Green vials: 110 ppm Hg*

## | CROSS CONTAMINATION TEST AND VOLATILE ELEMENT TEST

To confirm that cross contamination and loss of volatiles does not occur, two different tests were performed. First, 8 blanks were placed in a 15-position rack with 7 vials containing 110 ppm Hg standard solution. The blanks consisted of 4 mL high purity concentrated $HNO_3$ in glass vials. The vials were not precleaned. Figure 6 shows the arrangement of blanks and standards in the rack- the blanks were spaced evenly through the rack. A digestion run to 250 °C was performed. The blanks were then diluted to 40 mL with DI water and analyzed for Hg using an Agilent 7500cx ICP-MS.

| | Hg (ppt) |
|---|---|
| Blk position 01 | 0.020 |
| Blk position 03 | 0.032 |
| Blk position 05 | 0.001 |
| Blk position 07 | 0.002 |
| Blk position 09 | -0.003 |
| Blk position 11 | -0.007 |
| Blk position 13 | -0.007 |
| Blk position 15 | -0.006 |

*Table 1 - Hg concentration in the blanks*

Table 1 shows the concentration of Hg measured in each of the blanks, showing that no Hg from the standards had been transferred to the blanks by spatter, boiling, volatilization or other mechanism. The test above demonstrates that cross contamination, even with a volatile element such as Hg, does not occur. In the second test, three different certified reference materials (CRM), certified for Hg, were digested together in the ultraWAVE along with two acid blanks. Hg analysis was then performed on the digestates using a Milestone DMA-80 *evo* direct mercury analyzer.

| Sample | Certified | ultraWAVE |
|---|---|---|
| **DORM-3 Fish protein** | 409 ± 27 µg/kg | 393 µg/kg |
| | | 383 µg/kg |
| | | 388 µg/kg |
| **ERM-EC680 Polyethylene** | 25.3 ± 1.0 mg/kg | 24.9 mg/kg |
| | | 24.4 mg/kg |
| | | 24.6 mg/kg |
| **NIST2709 San Joaquin soil** | 1.4 ± 0.08 mg/kg | 1.36 mg/kg |
| | | 1.40 mg/kg |
| | | 1.35 mg/kg |

*Table 2 - Measurement of Hg in 3 reference materials following digestion in the ultraWAVE*

The CRMs had Hg concentrations covering 2 orders of magnitude, yet excellent recoveries were obtained for all aliquots of every standard (Table 2), demonstrating that Hg is not lost during the digestion run and complete recovery is achieved.
The variety of sample types - a fish tissue, a polymer and a soil - able to be digested together under the same conditions also demonstrates the highly efficient workflow of the ultraWAVE.

## CONCLUSION

A detailed comparison of digestion blank contribution due to the choice of vial material was made. The comparison demonstrates that the ultraWAVE, used in a routine, non-cleanroom environment, can produce digestates suitable for ultratrace analysis by ICP-MS. While the higher productivity, ease of use and superior digestion power of the ultraWAVE are well accepted, this report demonstrates that the digestion of samples in a single chamber, in loosely capped vials, does not negatively impact analytical data quality. The technique of pre-pressurizing the chamber with $N_2$ effectively prevents cross contamination and loss of volatiles, since the digest solution never reach boiling point.