



PRODUCTIVITY BENEFITS OF SINGLE REACTION CHAMBER (SRC) MICROWAVE DIGESTION *THE ULTRACLAVE ADVANTAGE*

SUMMARY

Milestone's UltraCLAVE is the most productive and efficient microwave digestion system available. Instead of the traditional approach of using an individual, closed reaction vessel for each sample, the UltraCLAVE features a large, pressurized reaction chamber into which all samples are placed simultaneously for digestion by microwave.

This patented Single Reaction Chamber (SRC) technology eliminates the need for assembly/disassembly of reaction vessels, allows any combination of sample types to be digested in the same run, handles racks of 40 or even 77 samples, uses standard glass autosampler vials, and greatly reduces consumable costs. Since the reaction chamber is pressurized, there is no cross contamination of samples or loss of volatiles.

The UltraCLAVE with SRC technology removes the limitations of conventional closed vessel microwave digestion and makes sample preparation more efficient than ever before.



INTRODUCTION

Closed vessel microwave digestion is a well accepted sample preparation technique in metals labs worldwide. Digestion is safe and effective, and can be applied to almost any sample type. Closed vessel digestion, however has some limitations. With conventional closed vessel digestion the samples must be digested in individual, pressurized reaction vessels and the vessels must be acid cleaned prior to each use (normally by performing a cleaning cycle in the microwave with an acid blank). In addition, the vessels need to be assembled and sealed with a special tool and mounted in a rotor. After the microwave program is completed, the vessel must be disassembled and the sample solution transferred to another container prior to analysis, increasing sample handling and potential for contamination.

The high temperature and pressure achieved during microwave digestion give rise to a further limitation with closed vessel systems: relatively high consumables costs - since the vessels degrade over time: vessels, caps/disks and shield need to be replaced – typically annually in a busy lab. Finally, with closed vessel digestion, a sample must be selected as a reference to monitor the digestion process, and so the sample weight, matrix type and acid type and amount used for every sample in the run must match the sample in the reference vessel. So for each sample type being digested, a separate run must be performed.



PHOTO: A 40 position rack in position prior to digestion. The SRC chamber will rise up from below and be locked in position by the two clamps.

THE SINGLE REACTION CHAMBER APPROACH

Unlike conventional closed vessel digestion, the Milestone UltraCLAVE offers a new and completely unique approach to micro-

wave digestion. The UltraCLAVE features a large, pressurized reaction chamber into which all samples are placed simultaneously for microwave digestion. This patented Single Reaction Chamber (SRC) completely changes the traditional workflow technology: the microwave cavity becomes the pressurized reaction vessel, samples are digested in standard autosampler vials and loading samples is as simple as loading an autosampler rack. And since the chamber is pressurized, there is no sample cross contamination or loss of volatiles. Productivity is greatly increased, operating costs are reduced, and the limitations of conventional closed vessel digestion are removed at a stroke.

Productivity benefits of the UltraCLAVE with SRC technology:

- reaction vessel assembly and disassembly steps are eliminated
- minimal sample handling
- any combination of sample matrix can be digested in the same run
- cleaning run of reaction vessels is eliminated
- difficult samples are digested more quickly and more completely

Operating cost benefits of the UltraCLAVE with SRC technology:

- operator time is significantly reduced – by up to 4x
- reaction vessel consumables (vessel/cap/shield) costs eliminated
- less digestion acid is used – typically 4mL HNO₃ is required (10mL with closed vessel)
- standard, reusable quartz sample vials (or disposable glass vials) are used
- a single 40 position rack can handle virtually every sample type and application

For more detailed information on the technical features of the UltraCLAVE, see Milestone UltraCLAVE product brochure (pub. # UltraCLAVE/01/2010)

MIXED BATCH DIGESTION: A FIRST IN MICROWAVE DIGESTION

Consider the sample workload of a contract lab supporting the consumer goods, biomedical and pharma industries. On a given day, the labs may receive samples of toys, hair, consumer products and nutraceuticals for digestion and analysis. Conventional microwave digestion would require four separate runs – one for

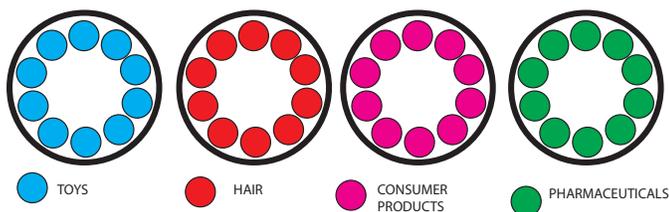


Figure 1. Configuration for closed vessel digestion

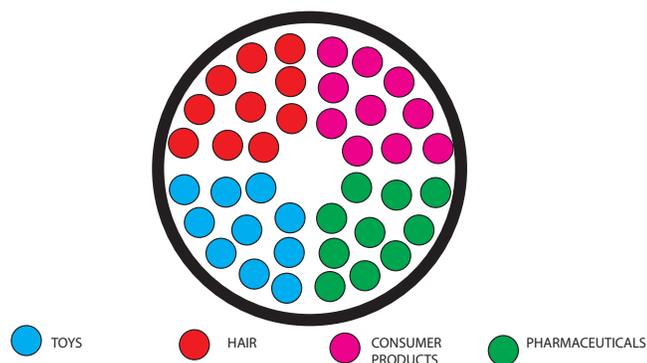


Figure 2. Configuration for AutoCLAVE digestion

each matrix type – even if there were only a single sample of one of the matrices.

A 10 position rotor is typically used for these sample types, and a graphical representation of the required 4 separate microwave runs is shown in Figure 1 above. Total sample capacity of the 4 instruments is 40 with a maximum of 10 of any given matrix.

Compare that with an UltraCLAVE digestion run in Figure 2 above: a single run using a 40 position rack holding 20mL vials digests all samples simultaneously. Capacity is 40 samples – in any combination of sample type.

Vessels	Arsenic (ug/L)	Cadmium (ug/L)	Mercury (ug/L)	Lead (ug/L)
Quartz (reusable)				
Min.	0.03	0.01	<0.01	0.29
Max.	0.05	0.02	0.02	0.48
Ave.	0.04	0.01	0.01	0.40
Glass (disposable)				
Min.	<0.01	<0.01	<0.01	0.05
Max.	0.01	0.01	0.03	0.13
Ave.	<0.01	0.01	0.02	0.09

Table 1. Blank levels in a mixed batch

The productivity benefit for a lab analyzing a variety of sample types is clear. The UltraCLAVE can even accept 77 10mL sample vials, but generally 40 x 20mL vials are preferred for more difficult samples. Of course mixed batch digestion is only an advantage provided cross contamination of samples does not occur. The SRC is pre-pressurized with nitrogen, and when heating begins pressure further increases. Since the whole chamber is pressurized, sample boiling or spitting does not occur. In addition, the vials are capped with loose fitting polypropylene caps to prevent condensed water dripping into the sample from the roof of the Teflon chamber liner. To test for cross contamination, a selection of quartz and glass vials containing the reagent blank (4mL of high purity HNO₃) were placed in a mixed batch digestion run of toys, consumer products and pharmaceutical samples. The vials were not pre-cleaned in the UltraCLAVE prior to use. Table 1 shows the concentrations of 4 key analytes in the blanks, measured by collision cell ICP-MS. In all cases the blanks are at the low to mid ppt level. Cross contamination did not occur, and for these analytes, disposable glass vials are clearly suitable for use, further saving operator time.

COST SAVINGS WITH THE ULTRACLAVE

In addition to higher sample capacity, the UltraCLAVE brings very significant labor saving too; as stated earlier, closed vessel microwave digestion is a relatively labor intensive technique.

600 Samples/wk	Closed vessel system	UltraCLAVE (reusable quartz vials)	UltraCLAVE (disposable glass vials)
Number of instruments	4	1	1
Number of rotors/racks	8	2	2
# of Technicians	4	1	1
Labor (hrs/wk)	160	40	32
Labor cost (\$/wk)	3200	800	640
Labor cost (\$/yr)	160,000	40,000	32,000
Labor savings/yr		\$120,000	\$138,000

Table 2. Labor saving - closed vessel digestion vs. UltraCLAVE

Considering labor alone, the cost savings achieved with the UltraCLAVE are substantial. Table 2 shows the labor costs savings for a busy lab digesting 600 samples/week. Such a lab would require 4 traditional closed vessel microwave instruments and 4 technicians working full time (40hr/week). By comparison, an UltraCLAVE would require only one technician working full time, or 32 hours/week if disposable vials were used.

This does not take into account the higher consumables costs of the closed vessel systems (vessels, caps, shields), which could be as much as \$30-35K more per year than the cost of the UltraCLAVE vials. Finally, the UltraCLAVE brings additional savings in high purity HNO₃, typically using 50% less than a closed vessel system.

CONCLUSION

The Milestone UltraCLAVE is revolutionizing microwave digestion due to its patented SRC technology by significantly increasing sample throughput and dramatically lowering labor costs. Virtually any type of sample can be digested using a standard quartz or glass vial, using the same operating parameters. Mixed batches of different sample types can be digested simultaneously, without cross contamination of samples or loss of volatiles. And loading the UltraCLAVE is as simple as loading an autosampler rack.

The UltraCLAVE automates the sample digestion process in order to keep pace with the high sample capacity of modern ICP-OES and ICP-MS instrumentation.



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