



## USP <232>/<233> Pharmaceutical Samples

Single reaction chamber (SRC) microwave digestion offers significant benefits over traditional microwave digestion for pharmaceutical samples: higher sample throughput, reduced labor and consumables costs, simplified workflow and superior digestion quality.

### Summary

New USP chapters <232> and <233> for the measurement of inorganic contaminants in pharmaceutical samples will be implemented on January 1, 2018. While samples can be dissolved in aqueous or organic solvents, the large majority will require digestion, and closed vessel microwave digestion is stipulated by USP. Single reaction chamber (SRC) microwave digestion is a new type of closed vessel digestion that was developed by Milestone and offers many advantages over closed vessel digestion – particularly for pharmaceutical sample types.

### Instrumentation

The Milestone UltraWAVE can digest up to 15 samples simultaneously, at up to 300 °C and 199 bar pressure. Its high temperature and pressure capability enables the complete digestion of virtually every sample type. Samples are weighed into vials and placed in the chamber: and any combination of sample types can be digested together - greatly simplifying the sample prep workflow. Raw materials, excipients, API and final product can all be digested together in the same run. And the UltraWAVE uses disposable glass vials, eliminating vessel assembly/disassembly and vessel cleaning which significantly reduces labor and consumables costs. Most pharmaceutical sample types have high organic content, including oils and fats which are difficult to

#### St. John's Wort

Parts Per Million (ug/g)

Step	Arsenic	Cadium	Lead	Mercury
Sample Result	0.184	0.109	0.24	ND
Duplicate Result	0.195	0.115	0.19	ND
Detection Limit	0.008	0.003	0.03	0.1

#### Fish Oil Gelcaps

Parts Per Million (ug/g)

Step	Arsenic	Cadium	Lead	Mercury
Sample Result	0.007	ND	0.018	0.035
Duplicate Result	0.006	ND	0.022	0.024
Detection Limit	0.002	0.0007	0.007	0.008

#### Quality Control Summary (Fish Oil Gelcaps)

Parts Per Million (ug/g)

Step	Arsenic	Cadium	Lead	Mercury
Spike Conc	15.3	5.09	10.2	15.3
Spike Result	15.6	5.15	9.96	14.2

*The data above shows excellent DLs and recovery – even for the volatile element mercury – no loss of volatiles or cross-contamination with the UltraWAVE.*



digest and generate CO<sub>2</sub> which increases pressure in the reaction vessel. The high pressure capability of the UltraWAVE enables it to digest entire fish oil gelcaps - not possible with traditional closed vessel microwave.

The UltraWAVE can digest samples in as little as 3mL acid which minimizes reagent blank levels – important since ICP-MS will be used predominantly for USP <232>/<233> when implemented. The SRC chamber is pre-pressurized with N<sub>2</sub> prior to the start of the run – this prevents sample boiling, eliminating cross-contamination and loss of volatiles.

Due to its higher sample capacity, use of disposable vials, and faster cool down time, UltraWAVE sample throughput is 2x – 3x higher than closed vessel digestion. Its lower consumables costs,

simple operation and superior digestion quality also make the Milestone UltraWAVE the perfect choice for pharmaceutical sample prep.

## About Milestone

With over 50 patents and more than 18,000 instruments installed in laboratories around the world, Milestone has been widely recognized as the global leader in metals prep technology for the past 26 years. Committed to providing safe, reliable and flexible platforms to enhance your lab's productivity, customers worldwide look to Milestone for their metals digestion, organic extractions, mercury analysis and clean chemistry processing needs.

Learn more or request an onsite demonstration:  
[info@milestonesci.com](mailto:info@milestonesci.com) or 1-866-995-5100

