



# Microwave Digestion for Specialty Polymers

Utilizing Single Reaction Chamber (SRC) Technology for Trace Metals Analysis for Trace Metals Analysis for polymer samples.

## Summary

With stricter industry regulations now in place, demand for trace metals analysis at lower detection levels has reached an all-time high. ICP, once the standard for metals analysis of polymers, is rapidly being replaced by ICP-MS, placing increased emphasis on sample preparation methodologies. Closed-vessel microwave digestion has proven to be an effective technique, offering fast, complete digestions, a clean environment, and effective recovery of volatile compounds. The single drawback has been the inability to run digestion on several matrix types simultaneously. Milestone's Single Reaction Chamber (SRC) microwave diges-

tion is a revolutionary new approach, incorporating all of the benefits of closed vessel microwave digestion with new levels of convenience and effectiveness. The Milestone UltraWAVE is a bench-top instrument that operates at very high pressures and temperatures, capable of processing large, dissimilar and difficult samples quickly, easily—all without batching. The data shown in this technical note demonstrates that the digestion of samples in the UltraWAVE results in uniformly high analytical data quality, making it the ideal solution for trace metals detection in specialty polymers.

SRC technology enables a chemist to digest up to 15 different polymer samples simultaneously at temperatures up to 300°C and pressures up to 199 bar, simplifying the workflow, while maintaining superior quality digestions.



Polymers represent a broad class of compounds with a tremendous range of physical properties. While some of these compounds are relatively easy to prepare for trace metals analysis, most polymeric and plastic materi-

als are very stable matrices and require extremely high temperatures and pressures to achieve complete digestion, which can be difficult to achieve even with conventional closed-vessel microwave systems. Since polymers are principally organic, they generate a lot of pressure during the organic decomposition of digestion processes. Today's chemist usually complements microwave technology with traditional tools like hot plates and Parr bombs to digest these highly stable matrices which in turn have their own set of limitations – large acid requirements, contamination, acid handling challenges, lengthy digestions cycles and exposure of chemists to acid fumes. Finally, although multiple samples can be digested in closed-vessel microwaves simultaneously, samples of similar matrices need to be batched to ensure complete control over the system. This limits the productivity of a lab testing wide varieties of sample matrices.

Milestone's UltraWAVE SRC benchtop digestion system can digest up to 15 different samples simultaneously at temperatures and pressures as high as 300°C and 199 bar. This high temperature and pressure capability enables a complete digestion of almost any specialty chemical that needs to be

analyzed for trace metals. Samples can be directly weighed into disposable glass, autosampler type vials with the appropriate acid mixtures- no minimum acid quantity is required. Quartz and TFM vials can also be used depending on the user's application. This minimizes acid handling and transferring steps, reduces errors due to contamination and the chemist's exposure to acids. It also completely eliminates the need to clean, assemble and disassemble vessels used in conventional microwave systems.

## Method Optimization

The reaction chamber was pre-pressurized to 40 bar to prevent the acids from boiling which subsequently prevented cross-contamination and loss of volatiles. A 15 position rack was used with disposable glass vials during digestion, which were covered by loose fitting caps. This ensures pressure equilibrium on either sides of the vial while preventing the condensation from the top of the chamber from dripping into the sample. 2 polymer excipient samples, Polypladone XL-10 and Pladone K90, were digested in one single run using 1 g in 5 mL conc HNO<sub>3</sub>. The heating profile used in the microwave is listed below:

**Table 1. Microwave program used for digestion of Polypladone and Pladone**

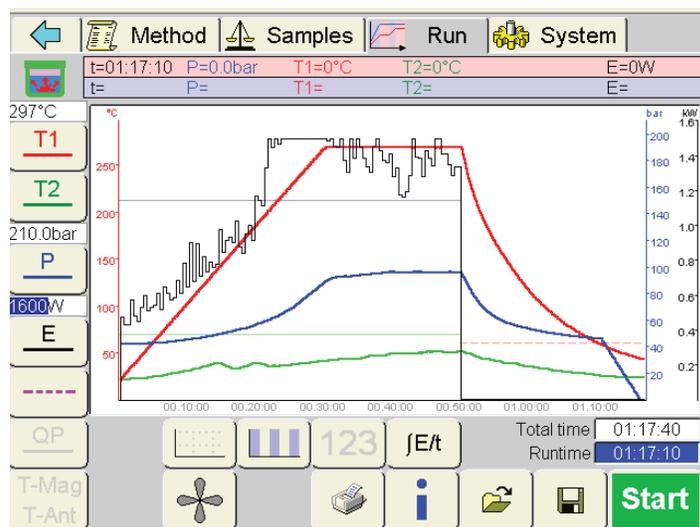
Time (min)	Power (watts)	Temp (°C)	Pressure (bar)
20	1500	260°C	150
15	1500	260°C	150

The actual microwave digestion profile shows a resultant pressure over ~90 bar (~1350 psi) was generated during the digestion cycle. As previously noted, many microwave vessel designs will not be usable for these digestion conditions. The digested samples were diluted to 30 mL with DI water and analyzed for the certified elements. The certified values for these elements and the recovery achieved after the digestion is mentioned in Table 2.



**Table 2. Recovery of various trace metal elements from Polyplasdone and Plasdone K 90 (% Recovery)**

Catalyst Element	Polyplasdone XL-10 (% Recovery)	Weight
Chromium	82.8	78.4
Copper	93.0	91.2
Manganese	91.4	76.2
Molybdenum	76.2	76.2
Nickel	89.0	78.6
Palladium	88.4	92.0
Platinum	92.0	74.0
Vanadium	91.6	77.8
Rhodium	89.8	84.6
Ruthenium	78.0	78.8
Iridium	92.4	76.8



The UltraWAVE's touch screen controller. The red line is the internal digestion temperature, while the blue line indicates the real-time pressure that is being monitored of the entire stainless steel chamber. The black line indicates the microwave power being emitted during the entire digestion cycle.

**Table 3. Microwave program used for digestion of CRMs -ERM681 and CRM8112A.**

Time (mins)	Power (watts)	Temp (°C)	Pressure limit (bar)
15	1500	225°C	150
15	1500	225°C	150

**Table 4. CRM Recovery Results Post Digestion.**

Element	CRM 8112A Cert (ppm)	CRM8112 found (ppm)	% recovery
Pb(220)	94.98	86.6	91.2
Cd	9.383	9.166	97.7
Hg	94.1	84.77	90.1

## Certified Reference Materials

Two certified polymer reference materials (ERM681- low density polyethylene and CRM8112A - ABS Resin) were also digested in the UltraWAVE in the 15 position rack using 0.2 g in 4 mL of conc HNO<sub>3</sub>. The microwave program used for digestions of these samples is listed in table 4.

Element	ERM681 Cert (ppm)	ERM681 found (ppm)	% recovery
Pb(22)	98.0	90.31	92.2
Cd	13.7.0	124.6	90.9
Hg	23.7	22.93	96.8
Pb(283)	98.0	95.09	97.0

## Conclusion

The data shows excellent DLs and recoveries. No loss of volatile elements (ex., Hg) and no cross contamination was observed. Due to higher sample capacity, use of disposable vials and faster cool down time, the UltraWAVE sample throughput is 2-3x times higher as compared to traditional closed-vessel microwave digestions. No other system is capable of handling the reported high pressures that are generated during the digestion of these challenging sample polymers. Its lower consumables costs, simple operation, and superior digestion quality makes Milestone's UltraWAVE a perfect choice for specialty polymers' sample prep for trace metals analysis.

## Acknowledgement

We would like to thank Wayne Xu of International Specialty Products for sharing data of Plasdone/ Polyplasdone.

## About Milestone

Our full suite of Microwave Sample Prep productivity tools is backed by over 50 patents and 26 years of industry expertise. Milestone is committed to providing safe, reliable and flexible platforms to enhance your productivity. Over 18,000 customers worldwide look to Milestone to improve their metals digestions, organic extractions, mercury analyzers or synthetic chemistry processes.

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

