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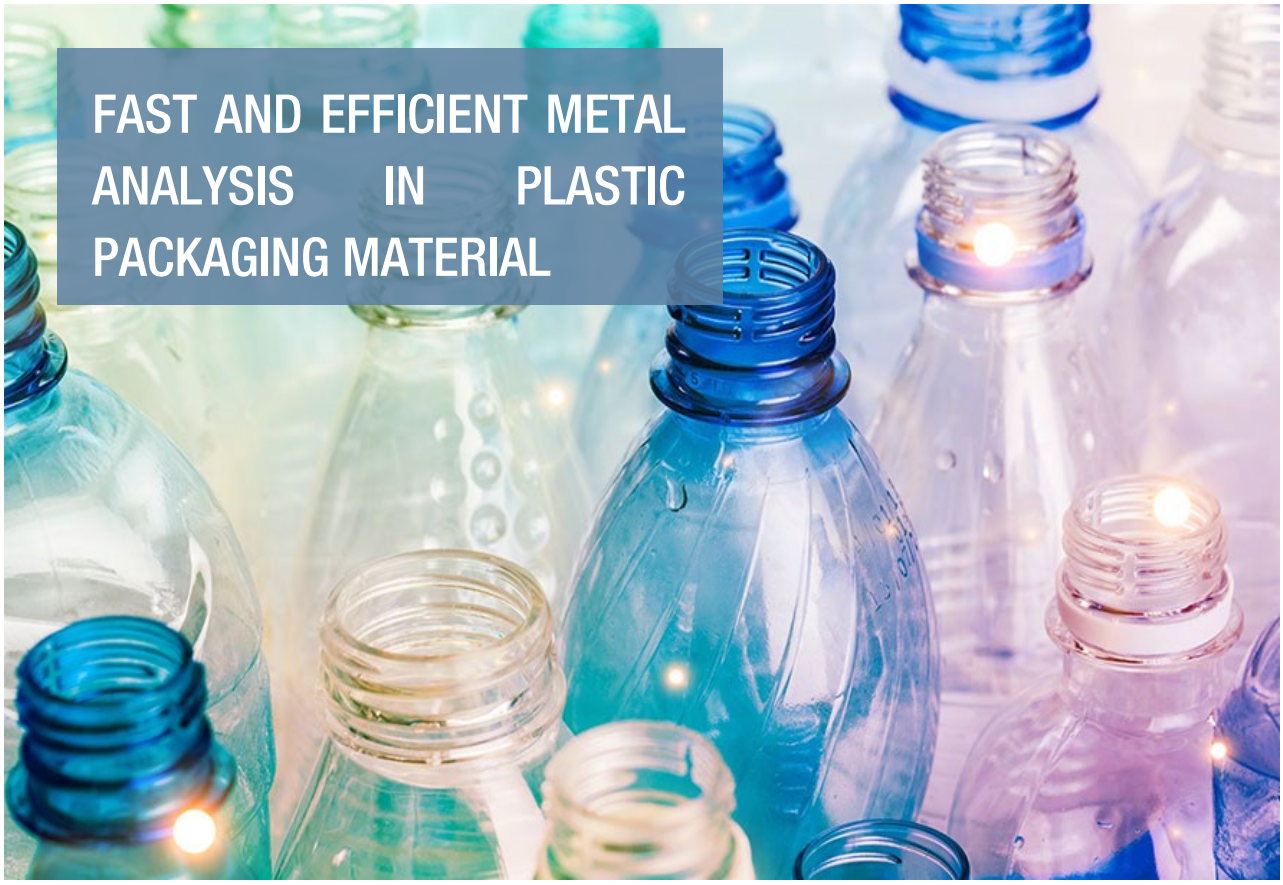

FAST AND EFFICIENT METAL ANALYSIS IN PLASTIC PACKAGING MATERIAL

Utilizing Single Reaction Chamber (SRC) Technology for trace metal analysis in polymer samples.

| INTRODUCTION

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, 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 digestion system is a revolutionary new approach, incorporating all 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 and easily—all without batching. The data shown in this technical note demonstrates that the digestion of samples in the ultraWAVE results in uniformly high-quality analytical data, making it the ideal solution for trace metals detection in specialty polymers.

| EXPERIMENTAL

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 materials 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 digestion processes.

INSTRUMENT

Today's chemist usually complements microwave technology with traditional techniques such as hot plates and Parr bomb, to digest these highly stable matrices. These, however, have their own set of limitations, such as large acid requirements, contamination, acid handling challenges, lengthy digestions cycles and exposure of chemists to acid fumes. Finally, although multiple samples can be digested with closed vessel microwaves simultaneously, samples of similar matrices need to be batched to ensure complete control of the digestion process. This limits the productivity of labs testing wide varieties of sample matrices. Developed by Milestone, the ultraWAVE SRC benchtop digestion system is designed with a 1 Liter reactor, capable of operating at very high temperature and pressure. The 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 enable 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 as is done with conventional microwave systems.

Figure 1 – Milestone's ultraWAVE

For the first time, a microwave digestion system ensures the same temperature and pressure conditions in all positions, even when different samples and/or chemistries are used.

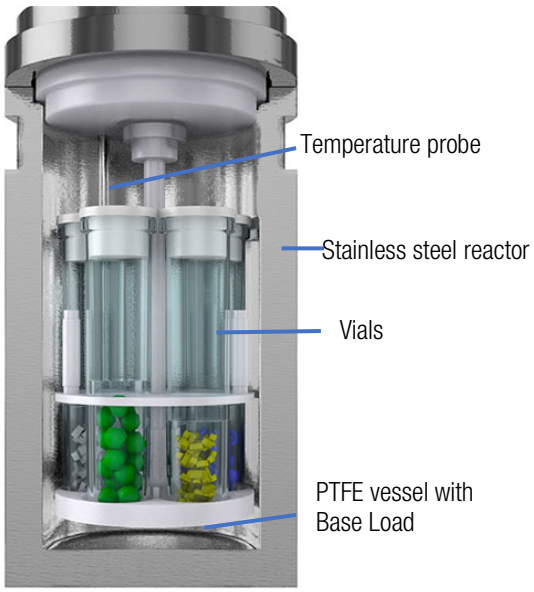

Figure 2 – Schematic of the ultraWAVE's single reaction chamber (SRC)

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. The easy handling of the utraWAVE's vials and racks greatly reduces operator time and associated labor costs.

METHOD OPTIMIZATION

The reaction chamber was pre-pressurized to 40 bar to prevent the acids from boiling, which subsequently prevents 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 side of the vial, while preventing condensation from the top of the chamber from dripping into the samples. The heating profile used in the microwave is listed below:

Time (min)	Power (watts)	Temp (°C)	Pressure (bar)
00:20:00	1500	260 °C	150
00:15:00	1500	260 °C	150

Table 1 - Microwave program used for digestion of Polyplasdone and Plasdone

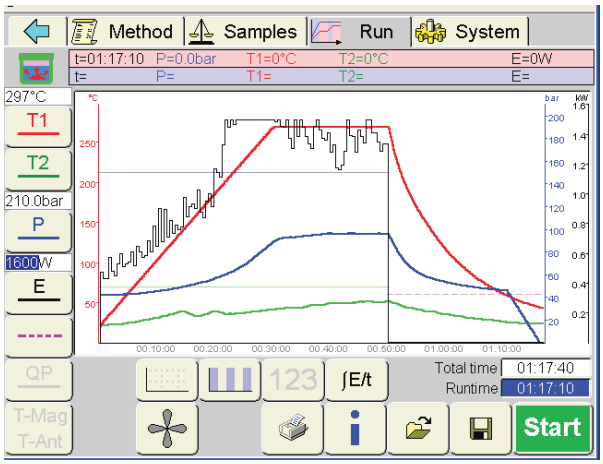

Figure 3 - The ultraWAVE's touch screen controller. The red line is the internal digestion temperature, while the blue line indicates the real-time pressure that is being monitored inside the stainless steel chamber. The black line indicates the microwave power being emitted during the entire digestion cycle.

The microwave digestion profile above 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.

RESULTS

Three separate microwave digestion runs were performed on a variety of mixed-

packaging samples from commercial sources. Each of the methods described above resulted in a clear digestion, which was diluted with deionized water to 25 mL. These samples were analyzed for Cd, Cr, and Pb using ICP-OES. Quality control (QC), laboratory blank, control spikes and duplicates were also analyzed.

Quality Control Summary

	Concentration (ppm)		
Spike Concentration	2	2	2
Spike Result	1.912	2.007	1.954
Spike Recovery %	95.6	100.4	97.7
Spike duplicate	2	2	2
Spike duplicate result	1.973	2.073	2.018
Spike duplicate recovery %	98.7	103.7	100.9
Detection Limit	0.01	0.01	0.01

Sample Summary

	Concentration (ppm)		
Product Box	<1.29	3.24	<1.29
Container Seal	<1.63	<1.63	<1.63
Outer plastic cup	<1.48	<1.48	<1.48
Inner plastic cup	<1.39	<1.39	<1.39
Inner cap cardboard	<1.48	3.25	<1.48
Label	<1.35	6.28	<1.35
Bottle	<1.36	<1.36	<1.36
Pet toy bistles (handle)	-	-	<1.44
Bristles duplicate	-	-	<1.39
Entree container	-	-	<1.36
Cardboard	<1.46	3.06	<1.46
Plastic	<1.46	<1.46	<1.46

Table 1 - ICP-OES analysis of a typical mixed packaging sample set; plastics, polymers, and printed paper following SRC digestion (0.2 g, HNO_3 25 min to 250 °C with 15 min at 250 °C)

Quantitative data, detection limits, and QC data are shown in Tables 1, 2, and 3. By examining the results, it was easy to determine samples requiring subsequent testing and flagging for the customer. Given the high throughput nature of client demand, the standardization of the SRC microwave method aligned well with the use of ICP-OES analysis for a majority of incoming samples. The use of disposable glass vessels, ACS-grade acid and small sample sizes provided a cost effective approach. A switch to TFM or quartz vessels and trace-level acid provides a path for coupling microwave digestion and ICP-MS analysis for other element panels and lower concentration ranges.

Quality Control Summary

	Concentration (ppm)		
Spike Concentration	2	2	2
Spike Result	1.936	2.047	1.954
Spike Recovery %	96.8	102.4	97.7
Spike duplicate	2	2	2
Spike duplicate result	1.875	1.979	1.893
Spike duplicate recovery %	93.8	99	94.7
Detection Limit	0.01	0.01	0.01

Sample Summary			
	Concentration (ppm)		
Clear Plastic	610.33	-	<1.28
Frosted Plastic	570.27	-	-
Clear plastic duplicate	613.43	-	-
Frosted plastic duplicate	596.22	-	-
Outer plastic wrapper	<1.44	<1.44	<1.44
Large white backing	<1.36	<1.36	<1.36
Inner plastic wrapper	<1.37	<1.37	<1.37
Small clear backing	<1.28	<1.28	<1.28
Outside package	<1.41	<1.41	<1.41
Inside package	<1.42	<1.42	<1.42
Paper back	<1.46	3.72	<1.46
Side dish with lid	-	-	<1.46

Table 2. ICP-OES analysis of mixed sample packaging, containers, and lids following SRC microwave digestion (0.2 g, 5 mL HNO_3 and 0.5 mL HCl: 25 min to 250 °C with 15 min at 250 °C)

Quality Control Summary			
	Concentration (ppm)		
Spike Concentration	2	2	2
Spike Result	1.886	1.859	1.891
Spike Recovery %	94.3	93	94.6
Spike duplicate	2	2	2
Spike duplicate result	1.849	1.826	1.853
Spike duplicate recovery %	92.5	91.3	92.7
Detection Limit	0.01	0.01	0.01

Sample Summary			
	Concentration (ppm)		
Plastic	<1.39	<1.39	<1.39
Cap	<1.37	<1.37	<1.37
Bottle	<1.34	2.95	<1.34
Label	<1.37	<1.37	<1.37
Paper	<1.44	2.6	<1.44
Plastic (2)	<1.45	<1.45	<1.45
Cap (2)	<1.42	<1.42	<1.42
Bottle (2)	<1.34	1.72	<1.34
Label (2)	<1.38	<1.38	<1.38
Outer package	<1.92	<1.92	<1.92
Wrapper	<1.50	<1.50	<1.50
Wrapper	<1.36	<1.36	<1.36

Table 3. ICP-OES analysis of mixed sample plastic cap, bottle and wrapper following SRC microwave digestion (0.2 g, 5 mL HNO_3 and 0.5 mL HCl: 25 min to 250 °C with15 min at 250 °C)

CONCLUSION

SRC microwave digestion in commercial contract environments for trace metals analysis provides an optimized sample preparation method. To achieve complete digestion for all samples simultaneously, an optimized temperature of 250 °C was used following two method development iterations at 220 °C and 240 °C. The packaging sample digestions were performed in disposable glass vials to eliminate the need for vessel cleaning in subsequent digestion runs. With a 15-position rack and a 50 min digestion time, 3 sets of 12 customer samples, blanks, control spike samples, and duplicates were digested and analyzed in a normal working day. Standardizing the SRC method with an optimized temperature, choice of vessel material and acid combination enables commercial laboratories to process any combination of customer samples without extensive method development, batching or vessel cleaning and assembly.

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