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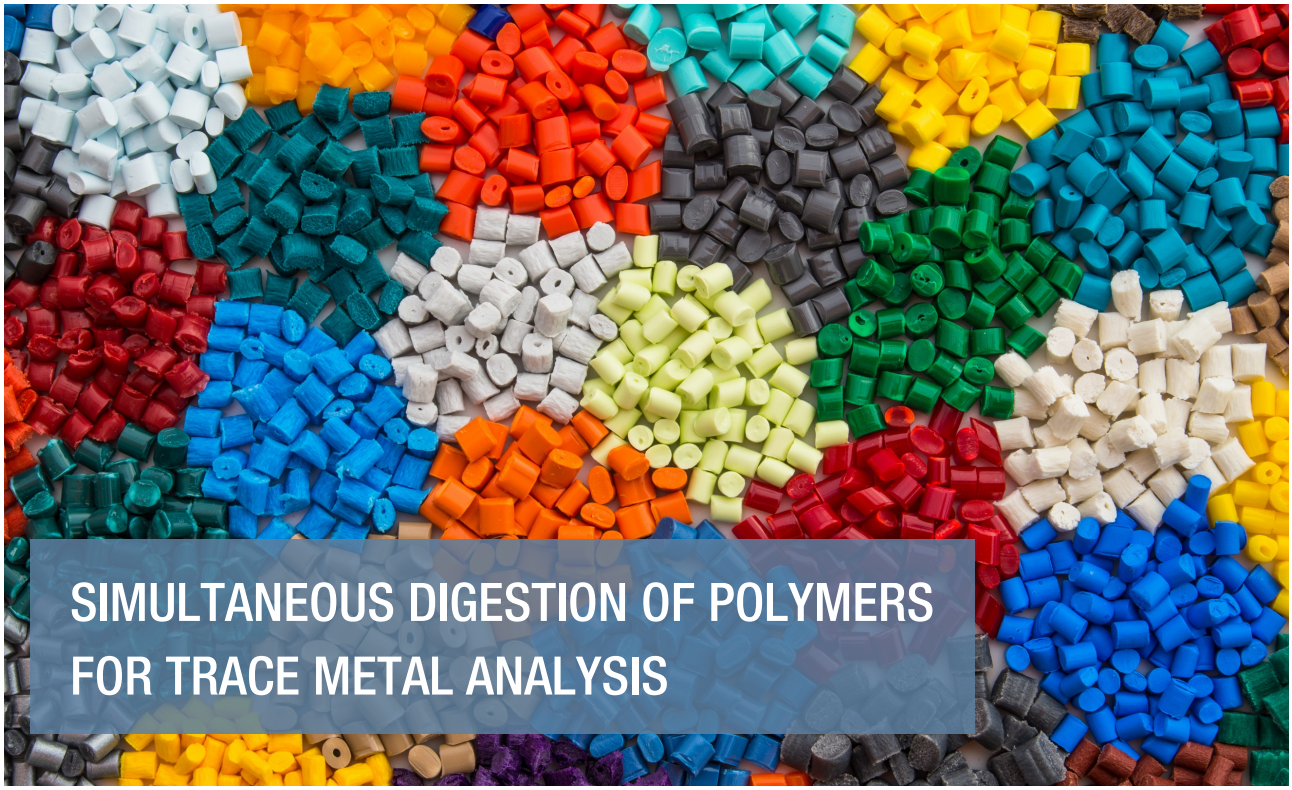


SIMULTANEOUS DIGESTION OF POLYMERS FOR TRACE METAL ANALYSIS

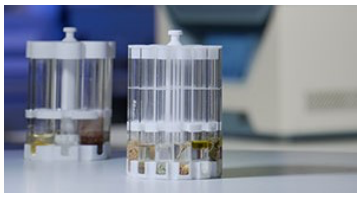
Utilizing Single Reaction Chamber (SRC) Technology for trace metal analysis in polymer samples.

I INTRODUCTION

With stricter industry regulations now in place, demand for trace metals analysis at lower detection levels has reached an all-time high, 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.

Polymers, for their part, 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 organic decomposition of digestion processes.

Milestone's Single Reaction Chamber (SRC) microwave digestion, 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 3 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 polymer samples.



| EXPERIMENTAL

INSTRUMENTATION

The ultraWAVE 3 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 (up to 0.5 g of polymers) as well as highly reactive and difficult-to-digest samples.

Figure 1 – Milestone's ultraWAVE 3

For the first time, a microwave digestion system ensures equal temperature and pressure conditions in all positions, even when different samples and/or chemistries are used. This results in superior digestion capabilities, higher productivity and better workflow for the lab.

The ultraWAVE 3 base load and positive pressure load prior to heating generates an equilibrium of temperature and pressure in each position, thus avoiding sample/ elemental loss and cross contamination.

Samples can be weighed directly into disposable glass vials, eliminating the cleaning step. The easy handling of the vials and racks greatly reduces the operator time and associated labor costs.

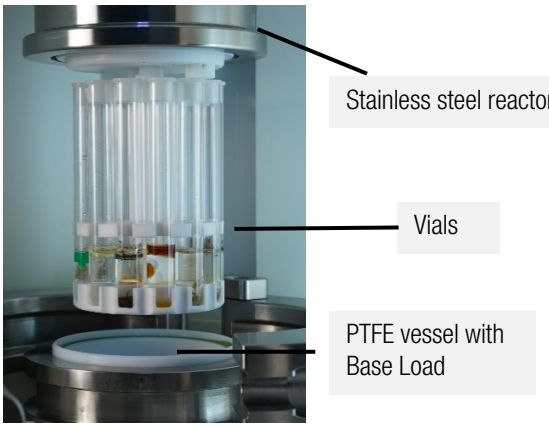


Figure 2 – Schematic of the ultraWAVE 3 Single Reaction Chamber (SRC)

SAMPLES

In this industry report, a recovery study was performed working with two different Certified Reference Materials and one polymer sample, spiked with a standard solution at known concentration of metals.

Sample ID	Sample Type	Sample matrix
ERM-EC680	Certified Reference Material	Polyethylene (Low level)
ERM-EC680k	Certified Reference Material	Polyethylene
ABS	Spiked	Acrylnitrile-Butadiene-Styrene-Copolymer

Table 1: Samples used for the study

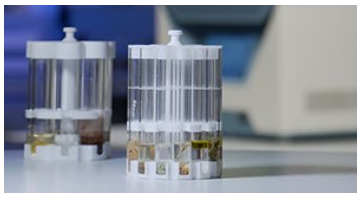
PROCEDURE AND METHOD

0.2 g of each sample were accurately weighed into disposable glass vials. Quartz vials are also available. PTFE vials are usually not recommended for polymer digestions.

Three replicates of each CRM and six replicates of ABS sample were prepared to evaluate the repeatability of the digestion step.

Prior to acid addition, three ABS samples out of six were spiked with 800 µL of a 10 mg/L Multi-element Standard solution. Considering a final volume of 50 mL, it resulted in a spiked concentration of 160 µg/L.

3 mL of HNO_3 67% and 0.5 mL of HCl 37% (ACS reagent grade) were added to the samples. Two



blanks with the same amount of acids were included. A base load of 120 mL DI H_2O and 5 mL HNO_3 67% was added into the 1 Liter PTFE vessel. The analysis was performed with an Optical Emission Spectometer ICP (ICP-OES).

Step	Time (hh:mm:ss)	Power (W)	Temp T1 (°C)	Temp T2 (°C)	Pressure (bar)
1	00:10:00	800	130	60	60
2	00:10:00	1200	180	60	80
3	00:10:00	1500	250	60	110
4	00:10:00	1500	250	60	110

Table 2: UltraWAVE digestion heating program for the simultaneous digestion of polymer samples.

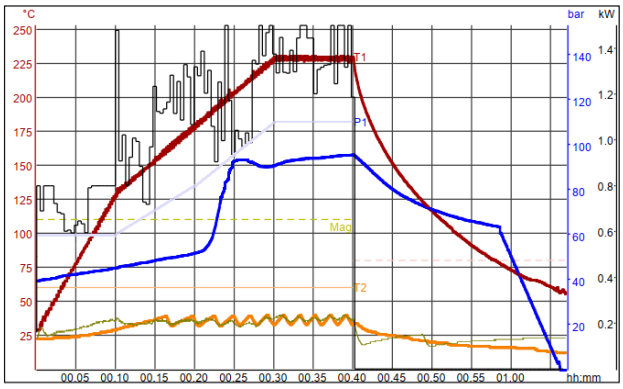


Figure 2: Internal temperature (red), external temperature (orange), pressure (blue) and power (black) graphs.

Parameter	Setting
RF applied power (kW)	1.3
Plasma gas flow rate (L/min)	15
Auxiliary gas flow rate (L/min)	1.5
Nebulizer gas flow rate (L/min)	0.75
Replicate read time (s)	5
Stabilization delay (s)	30
Sample uptake delay (s)	30
Pump rate (rpm)	15
Rinse time (s)	15
Replicates (n°)	3

Table 3: ICP-OES operating conditions

RESULTS AND DISCUSSION

The analytical results are shown in Tables 4, 5 and 6 with good recoveries of all analytes and RSDs below 10%. This demonstrates the robustness and reproducibility of microwave digestion using the ultraWAVE 3 with SRC technology.

Element	*Measured Concentration (mg/kg)*	*RSD% (n=3)*	*Certified Concentration (mg/kg)*			*Recovery (%)*
As 188.980	*29.2*	*8.4*	*30.9*	*± 0.7*		*95*
Cd 214.439	*136.2*	*1.9*	*140.8*	*± 2.5*		*97*
Cr 267.716	*110.2*	*1.7*	*114.6*	*± 2.6*		*96*
Pb 220.353	*101.5*	*1.7*	*107.6*	*± 2.8*		*94*

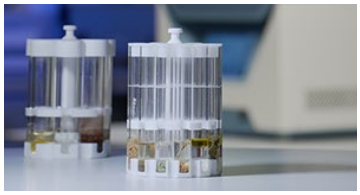


Table 4: Results for ERM-EC680, Polyethylene (Low level)

Element	Measured Concentration (mg/kg)	RSD% (n=3)	Certified Concentration (mg/kg)		Recovery (%)
As 188.980	3.65	8.9	4.1	± 0.5	90
Cd 214.439	20.1	0.9	19.6	± 1.4	103
Cr 267.716	7.62	7.8	20.2	± 1.1	38 [a]
Pb 220.353	13.1	3.8	13.6	± 0.5	97
Zn 213.857	140	1.7	137	± 20	102

[a] *Relatively low recovery is due to the presence of a significant fraction of Chromium in the form of Cr_2O_3. Refer to EC680k Certificate for detailed information.*

Table 5: Results for ERM-EC680k, Polyethylene

Element	Measured Concentration (µg/L)		RSD% (n=3)	Spike Recovery (%)
	ABS	ABS + Spike		
Ag 328.068	<20	150	2.31	94
Al 396.152	<20	169	3.60	106
As 188.980	<20	167	3.74	104
Cd 214.439	<20	152	1.74	95
Co 238.892	<20	155	1.97	97
Cr 267.716	<20	164	5.49	103
Cu 327.395	<20	164	1.54	102
Fe 238.204	<20	179	1.41	112
Li 670.783	<20	148	3.36	93
Mn 257.610	<20	153	1.31	96
Pb 220.353	<20	160	0.36	100
Zn 213.857	<20	161	2.72	101

Table 6: Results for ABS, Acrylnitrile-Butadiene-Styrene-Copolymer

| CONCLUSION

The data illustrated in this industry report demonstrates the ultraWAVE 3 ability to provide full recovery of all elements, while avoiding cross contamination even when different samples are digested in the same run. The ultraWAVE 3 ability to simultaneously digest different sample types, easy sample handling and superior throughput surpass the capabilities of hot blocks and traditional rotor-based microwave digestion systems. Its superior capabilities in terms of processing mixed samples, large sample amounts and ease of use provide unmatched productivity. The superior digestion quality achieved at high temperature and pressure maximizes the performance of the ICP-MS by reducing interferences, blanks and overall maintenance.

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