



Using Microwave Sample Prep to Determine Trace Metals Analysis in Polymer Matrices

Digestion of Polymer Samples Using the Milestone Ethos UP

Summary

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 that require extremely high temperatures and pressures to achieve complete digestion, which can be difficult to reach. Since polymers are principally organic, they generate a lot of pressure during the organic decomposition of the digestion process.

There are many challenges when using traditional methods such as hot plates and Parr bombs to digest these highly stable matrices. These challenges include large acid requirements, contamination, acid handling challenges, lengthy digestions cycles and exposure to acid fumes. Closed-vessel microwave technology is a proven alternative to these methods that speeds up the sample preparation process, improves the recovery of all the elements (including volatiles) and reduces possible sources of contamination.

The Milestone Ethos UP microwave digestion system incorporates all of the benefits of closed vessel digestion – speed, data quality, ease-of-use- in a safe and compact bench-top system.

This technical note evaluates the digestion quality of two certified reference materials :

- ECR680 Polyethylene (High level)
- ECR680K Polyethylene (Low level)

The analysis has been performed on: Mercury, Arsenic, Cadmium, Chromium, Lead and Zinc elements (Cd, Pb, Hg, Cr) are mentioned in the "Restriction of Hazardous Substances Directive. (Rohs).



Figure 1. The Milestone Ethos UP

Instrumentation

The Milestone Ethos UP matches the main requirements of many laboratories, thanks to its unique benefits:

- High throughput to increase productivity
- Flexibility to digest a variety of matrices
- Intuitive software
- Industry leading safety

The Milestone Ethos UP is a very flexible and high-performing platform used for trace elements and routine analysis. The Ethos UP is available with multiple configurations. The most suitable configuration for polymer samples is the SK-15 high pressure rotor. The SK-15 works with the Milestone "vent-and-reseal" technology for controlling and limiting the internal pressure of each vessel.

SK-15 High Pressure Rotor

The SK-15 rotor perfectly matches the lab's needs to determine trace elements, thanks to its capability to digest large sample amounts and its high temperature (300°C) and pressure (100 bar) capabilities.

The 15 position high-pressure rotor is safely controlled by a direct temperature sensor that consistently controls the digestion temperature during the run, ensuring complete and reproducible digestions of even the most difficult and reactive samples.



Analytical Procedure

The SK-15 is a high-pressure rotor that can perform complete digestions of very reactive samples without any venting of vessels and it has been used to digest the two certified samples simultaneously. Two aliquots from each sample type have been collected and digested in 2 different vessels. Table 1 shows the conditions used for the test.

Table 1. Digestion of Polyethylene ECR680 & Polyethylene ECR680K in the SK-15 Rotor

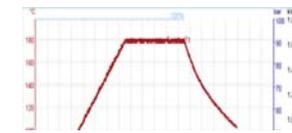
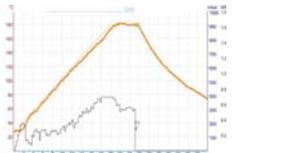
Sample Name	Sample Weight	Reagents
Polyethylene ECR680	300 mg	10 mL of HNO ₃ 65%
Polyethylene ECR680K	300 mg	10 mL of HNO ₃ 65%



All samples have been digested in the same batch since both CRM have exactly the same matrices. The Ethos UP is equipped with pre-installed libraries of methods with hundreds of applications. The EasyCONTROL software in combination with the direct and contactless temperature/pressure sensors allows the operator to fully control and monitor the entire digestion process.

The Ethos UP comes equipped with the Milestone Connect, the unique web application that allows the operator to remotely monitor the entire digestion process, through any Wi-Fi connected device.

Table 2. Method & Temperature Profile for SK-15 and Maxi-44 Rotors

	Method	Temperature Profile
SK-15 EPA 3051		
Maxi-44 3051		

Results

Table 3 shows the ICP-OES results, expressed in mg/Kg. All results have been obtained using Agilent ICP-OES (710 series).

Conclusion

The Milestone Ethos UP with SK-15 high-pressure rotor offers multiple benefits for sample preparation of difficult sample matrices, such as polymers. Thanks to the elevated temperature and pressure performances, the SK-15 rotor enables digestion quality, making ICP-MS analysis more accurate; while the closed-vessel technology guarantees a complete recovery for all elements including the volatiles.

Due to its higher sample capacity, the SK-15 rotor offers from 30 to 90% higher productivity compared to any other high-pressure rotor available on the market. The data in this technical note demonstrates that the better digestion quality achieved at higher temperatures and pressure makes analysis by ICP-OES more accurate.

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.

Table 3. ICP-OES Results

		As	Cd	Cr	Hg	Pb	Zn
Polyethylene (EC680)	Average	32.82	136.98	115.80	23.15	106.04	_*
	Certified Value	30.9	140.8	114.6	25.3	107.6	_*
	RECOVERY	106.20%	97.28%	101.05%	91.50%	98.55%	_*
Polyethylene (EC680-K)	Average	3.81	18.754	_*	4.01	13.17	132.24
	Certified Value	4.1	19.6		4.64	13.6	137
	RECOVERY	92.96%	95.68%		86.42%	96.84%	96.52%

