

SAMPLE PREPARATION OF POLYMERS FOR TRACE METAL ANALYSIS

Ensuring high-quality and productivity in elemental analysis of polymer samples using the Milestone ETHOS UP

| INTRODUCTION

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 reach. Since polymers are principally organic, they generate a lot of pressure during the digestion processes.

There are many challenges using traditional methods such as hot plates and Parr bombs to digest these highly stable matrices. These challenges include acid requirements, contamination, acid handling, lengthy digestions

cycles and exposure of chemists to acid fumes. Closed vessel microwave technology is a proven alternative to these methods which speeds up the sample preparation process, improves the recovery of all the elements (including volatiles) and reduces possible sources of contamination. Two certified reference materials were used in this study to evaluate the efficacy of the ETHOS UP in the digestion of polymer samples: ECR680 polyethylene (High level) and ECR680K low density polyethylene (Low level). The analysis was performed on Mercury, Arsenic, Cadmium, Chromium, Lead and Zinc elements, as mentioned in the “Restriction of Hazardous Substances Directive,” also known with the acronym “RoHS.”



EXPERIMENTAL

In this technical note, a recovery study on certified reference polymer materials was performed in order to prove the efficacy of the ETHOS UP in sample preparation for metal analysis.

INSTRUMENT

The ETHOS UP meets the requirements of analytical labs. It offers several unique benefits including:

- Increased ease of use and productivity
- Enhanced control in all vessels
- Fast, accurate and traceable
- Superior safety and digestion quality

The ETHOS UP is a flexible and high performing platform used for elemental analysis and routine determinations in many applications. Its construction of stainless steel coated with five PTFE layers and accommodates both high-pressure and high-throughput rotors.



Figure 1 – Milestone's ETHOS UP

easyTEMP

Milestone's easyTEMP contact-less sensor directly controls the temperature of all samples and solutions, providing accurate temperature feedback to ensure complete digestion in all vessels and high safety.

The superior temperature measurement of easyTEMP allows the processing of different samples of similar reactivities, thus reducing labor time and increasing overall throughput.

This technology combines the fast and accurate

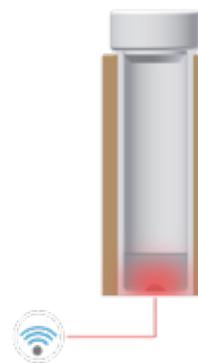


Figure 2 – easyTEMP contactless direct temperature sensor

reading of an in-situ temperature sensor with the flexibility of an infrared sensor. The ETHOS UP software provides digestion history traceability and temperature measurement for every sample. The temperature diagram and profiles are displayed real-time, and can be subsequently saved on the ETHOS UP terminal.

SK-15 HIGH PRESSURE ROTOR

The SK-15 rotor perfectly matches the needs of a modern analytical lab to determine trace elements, thanks to its capability for digesting large sample amounts at high temperature (up to 300 °C) and pressure (up to 100 bar).



Figure 3 – SK-15 easyTEMP High Pressure Rotor

The 15-position rotor is controlled by a contactless direct temperature sensor that controls the internal temperature of all vessels throughout the digestion cycle. This ensures complete and reproducible digestions of even the most difficult and reactive samples. The SK-15 also features Milestone’s patented “vent-and-reseal” technology for controlling the internal pressure of each vessel.

USER INTERFACE

The ETHOS UP comes with a dedicated touch screen terminal and easyCONTROL software which incorporates our expertise and know-how in microwave sample preparation. The ETHOS UP user-interface fully control all the digestion parameters, provides complete ocumentation and expedites the overall digestion procedure. The terminal is equipped

with multiple USB and ethernet ports for interfacing the instrument to external devices and the laboratory network. The ETHOS UP controller is user-friendly, icon-driven, multi-language and 21 CFR Part 11 compliant. To find the method which best suits your application simply select from the vast library of pre-stored methods. Included with the ETHOS UP is a unique web-based application: Milestone Connect. This app allows you to become a part of the Milestone community and gain exclusive access to a robust library of information: lists of parts, technical notes, user manuals, video tutorials, continuously updated application notes and all relevant scientific articles.



Figure 4 – easyCONTROL built-in library

ANALYTICAL PROCEDURE

ETHOS UP – SK-15 easyTEMP

SAMPLE	SAMPLE AMOUNT	ACID MIXTURE
Polyethylene (ERM EC680)	0.5 g	5 mL of HNO ₃ 65%
Low density polyethylene (ERM EC680K)	0.5 g	5 mL of HNO ₃ 65%

Table 1 – Sample amount and acid mixture used for the microwave digestion run



STEP	TIME	T2	POWER
1	00:20:00	210 °C	1800 W
2	00:10:00	210 °C	1800 W

Table 2 – Microwave program used for digestion of samples

- Final dilution: 50 mL with dionized water

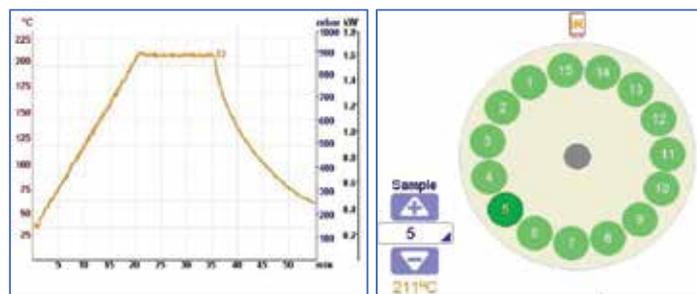


Figure 5 – SK-15 Microwave Run Report and multiple temperature traceability

QUANTIFICATION

ICP-OES Instrumental Parameters: RF power (W): 1300; Plasma flow (L/min): 15.0; Auxiliary Flow (L/min): 1.5; Nebulizer Flow (L/min): 0.75; Replicate read time (s): 10; Instrument stabilization delay (s): 15; Sample Uptake Delay (s): 30; Pump Rate (rpm): 15; Rinse Time (s): 10; Replicates: 3.

RESULTS AND DISCUSSION

The performance of the Milestone ETHOS UP equipped with SK-15 rotor and easyTEMP was evaluated through a recovery study on polyethylene and low density polyethylene (ERM EC680 and ERM EC680K respectively). The samples were digested with Milestone’s ETHOS UP and subsequently analyzed via ICP-OES.

	Certified value (mg/Kg)	Recovery % (n=3)	RSD (%)
As	30.9 ± 0.7	102.3	1.3
Cd	140.8 ± 2.5	98.6	2.6
Cr	114.6 ± 2.6	101.8	1.1
Hg	25.3 ± 1.0	93.5	0.8
Pb	107.6 ± 2.8	96.7	1.4

Table 3 – Data of the recovery study on ERM EC 680

	Certified value (mg/Kg)	Recovery % (n=3)	RSD (%)
As	4.1 ± 0.5	101.4	1.4
Cd	19.6 ± 1.4	94.9	1.6
Cr	20.2 ± 1.1	- ^a	- ^a

Hg ^b	4.64 ± 0.2	96.7	1.0
Pb	13.6 ± 0.5	97.3	2.7
Zn ^c	137.0 ± 20.0	98	1.6

Table 4- Data of the recovery study on ERM EC 680K

^a Average of 5.2 mg/Kg (RSD 2.6%) (to be compared with the Acid digestable Cr: 2.9- 16.2 mg/Kg)

^b Analyzed with ICP cold vapor generator module

^c Indicative values as reported in the certificate.

The analytical results are shown in tables 3 and 4 with good recoveries of all elements and RSDs below 3%. This demonstrates the robustness and reproducibility of digestion microwave digestion using the ETHOS UP equipped with SK-15 easyTEMP technology.

CONCLUSION

The data shown in this technical note demonstrates full recovery of the element reported in the certificates of the reference material.

Highly reactive samples such as polymers can be completely digested, even in large sample amounts along with samples of similar reactivities. The digestion process was accurately controlled by the easyTEMP sensor, ensuring superior digestion quality and reliable results. In addition to full analyte recovery, microwave digestion using the Milestone ETHOS UP provides the highest level of reproducibility, great ease of use and high productivity.

