

## DIGESTION OF POLYMERS SAMPLES USING ETHOS UP

Sample preparation for trace metal analysis of polymer matrices, using microwave digestion system

### 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 organic decomposition of digestion processes.



Traditional tools like hot plates and Parr bombs, used to digest these highly stable matrices, have their own set of limitations – large acid requirements, contamination, acid handling challenges, lengthy digestions cycles and exposure of chemists to acid fumes.

Closed-vessel microwaves technology allows speeding up the sample preparation process, improving the recovery of all the elements (including volatiles) and reducing possible sources of contamination.

Milestone's Ethos UP, microwave digestion system, incorporates all of the benefits of closed vessel microwave digestion while making sample preparation fast, easy, effective, and the highest quality.

This application report evaluates the digestion quality of two certified reference materials (CRM):

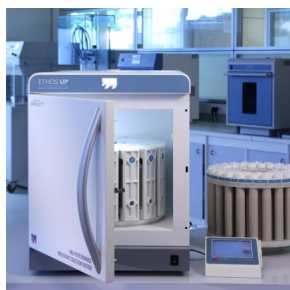
- ECR680 Polyethylene (High level)
- ECR680K Polyethylene (Low level)

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**”, also known with the acronym “**Rohs**”).



# APPLICATION REPORT

## Instrumentations



The ETHOS UP matches the main requirements of many laboratories, thanks to its unique benefits, such as:

- High productivity
- Ease of use
- High safety
- High flexibility

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, and most suitable one for polymers samples is the SK-15 high pressure.

The SK-15 works with the Milestone “vent-and-reseal” technology for controlling and limiting the internal pressure of each vessel.

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### SK-15 High Pressure rotor

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The SK-15 perfectly matches the laboratories needs to determine trace elements, thanks to its capability to digest large sample amount and its high temperature/pressure capabilities.

The 15 positions high pressure rotor is safely controlled via direct temperature sensor that constantly controls the digestion temperature during the run, ensuring perfect digestion of even the most difficult and reactive samples.

# APPLICATION REPORT

## Analytical Procedure

The SK-15 has been used to digest the two Certified samples simultaneously. The SK-15 is a high-pressure rotor, so it can perform digestion of very reactive samples without venting of vessels.

Two aliquots from each sample type have been collected and digested in two different vessels.

Here are the conditions used for the test:

| Sample Name          | Sample weight | Reagents                      |
|----------------------|---------------|-------------------------------|
| Polyethylene ECR680  | 300 mg        | 10 mL of HNO <sub>3</sub> 65% |
| Polyethylene ECR680K | 300 mg        | 10 mL of HNO <sub>3</sub> 65% |

All samples have been digested in the same batch since both CRM have exactly the same matrices.

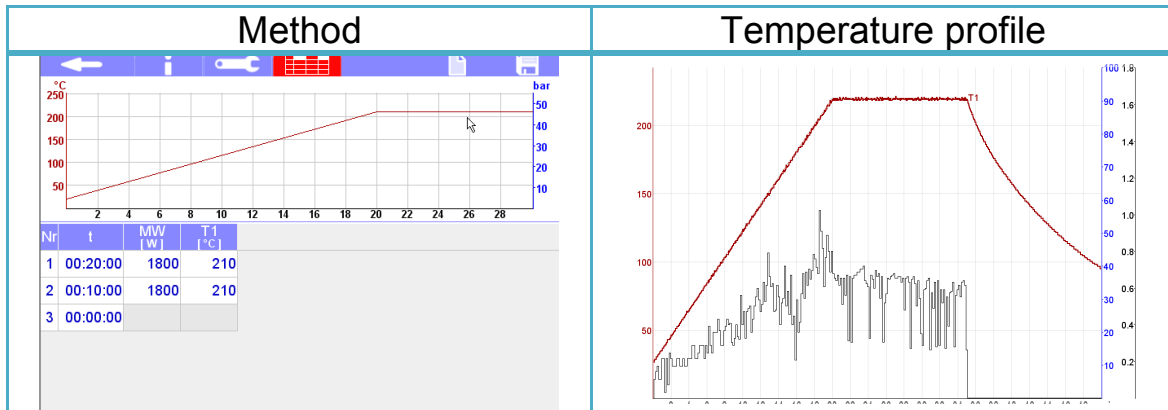
The Ethos UP is equipped with pre-installed libraries of methods with hundreds of applications.

The EasyCONTROL software in combination with the direct and contactless temperature/pressure sensors allows the operator to fully control and monitor all the digestion process.

The Ethos UP is provided with the Milestone Connect, the unique application that allows the operator to remotely monitor the digestion process, through any PC, tablets or smartphones.



# APPLICATION REPORT



## ICP-OES Results

All results are expressed in mg/Kg

|                        |                        | As             | Cd            | Cr             | Hg            | Pb            | Zn            |
|------------------------|------------------------|----------------|---------------|----------------|---------------|---------------|---------------|
| Polyethylene (EC680)   | <b>Average</b>         | 32,82          | 136,98        | 115,80         | 23,15         | 106,04        | -*            |
|                        | <b>Certified value</b> | 30,9           | 140,8         | 114,6          | 25,3          | 107,6         | -*            |
|                        | <b>RECOVERY</b>        | <b>106,20%</b> | <b>97,28%</b> | <b>101,05%</b> | <b>91,50%</b> | <b>98,55%</b> | -*            |
| Polyethylene (EC680-K) | <b>Average</b>         | 3,81           | 18,754        | -*             | 4,01          | 13,17         | 132,24        |
|                        | <b>Certified value</b> | 4,1            | 19,6          | -*             | 4,64          | 13,6          | 137           |
|                        | <b>RECOVERY</b>        | <b>92,96%</b>  | <b>95,68%</b> | -*             | <b>86,42%</b> | <b>96,84%</b> | <b>96,52%</b> |

\*Results below the ICP detection limit.

All results have been obtained using Agilent ICP-OES (710 series)

## Conclusions

Milestone Ethos UP with SK-15 high-pressure rotor offers multiple benefits for sample preparation for metals analysis of such difficult sample matrices as polymers.

Thanks to the elevated temperature and pressure performances, SK-15 rotor allows to get high digestion quality, making the analysis by ICP-MS more accurate; while the closed vessels 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 high-pressure rotor available in the market. The data showed in this technical note demonstrates that the better digestion quality achieved at higher temperatures (and pressure) makes analysis by ICP-OES more accurate.

