



DIGESTION OF POLYMER SAMPLES FOR METAL ANALYSIS

Acid digestion for trace metal analysis with ultrapure quartz inserts with low acid volumes

| INTRODUCTION

Polymer-related industries covers a very wide range of applications based on composition and nature of the polymer, which are critical to the manufacturing of the end product. Labs supporting the polymer industry need accurate and precise analytical data from a very wide range of sample matrices.

The complexity of tests performed implicates the need of technological advanced instrumentation. Laboratories often need to perform analysis of low-level trace metal samples (ICP-OES, ICP-MS, etc...).

In these conditions, sample preparation becomes a crucial operation before analysis.

Many laboratories need to reduce the amount of acid required for digestion to near stoichiometric quantities, which reduces the dilution factor and decrease the detection limit especially for some elements, in particular those at trace metal level.

This is why Milestone developed new micro-inserts for SK-15 rotor, capable to prepare samples employing small volume of acids.

These micro-inserts, based on so-called Vessel-Inside-Vessel Technology, are smaller secondary vials that can be placed inside the primary high-pressure microwave vessel of SK-15 rotor.

A variety of micro-inserts are available from Milestone in different materials (Quartz or TFM) and with different sizes and shapes, to accomplish all application requirements.



| EXPERIMENTAL

The objective of this test is completing a digestion of a reactive sample (CRM polymer material) with a maximum of 5mL of nitric acid and verifying the recovery of all certified metals with ICP-OES.

INSTRUMENT

The ETHOS UP (Figure 1) matches the main requirements of many laboratories, thanks to its unique benefits, such as:

1. High productivity
2. Ease of use
3. High safety
4. 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 micro sampling is the SK-15 high-pressure rotor (Figure 2). The SK-15 works with the Milestone “vent-and-reseal” technology for controlling and limiting the internal pressure of each vessel.



Figure 1 – Milestone's ETHOS UP

SK-15 HIGH PRESSURE ROTOR

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.

Figure 2 – SK15 easyTEMP High Pressure Rotor



MICRO-INSERTS TECHNOLOGY (VESSEL-INSIDE-VESSEL TECHNOLOGY)

Here below the inserts configuration used for the test.

Description	Picture	Working volume
QS-50 quartz vial with cap (34042)		15 mL ≤ Volume ≥ 5 mL

INDUSTRY REPORT

ETHOS UP | POLYMER



SAMPLE PREPARATION AND ICP-OES PARAMETERS

QS-50 quartz vial is the microinsert being used to digest two Polyethylene Certified Samples.

Quartz is a very resistant material, easy to clean, with a very low memory effect. The QS-50 quartz vial is therefore the right choice when working with samples that produce high exothermic reactions during

the digestion process (solvents, hydrocarbons, polymers, others...).

The SK-15 is a high-pressure rotor, which can perform digestion of very reactive samples without venting of vessels.

Here the list of samples that we performed in both micro-insert configurations:

Sample Name	Sample weight	Reagents into the vials	Reagents into the SK15 vessel	Micro-sampling config.
Polyethylene ECR-680	200 mg	HNO ₃ - 5 ml	H ₂ O dist. - 5 ml	QS-50 quartz vial
Polyethylene ECR-680K	200 mg	HNO ₃ - 5 ml	H ₂ O dist. - 5 ml	QS-50 quartz vial

Sample	Method	Temperature profile																								
Polyethylene	<table><thead><tr><th>Nr</th><th>t</th><th>MW [W]</th><th>T1 [°C]</th><th>T2 [°C]</th><th></th></tr></thead><tbody><tr><td>1</td><td>00:15:00</td><td>1800</td><td>200</td><td>0</td><td></td></tr><tr><td>2</td><td>00:10:00</td><td>1800</td><td>200</td><td>0</td><td></td></tr><tr><td>3</td><td>00:00:00</td><td></td><td></td><td></td><td></td></tr></tbody></table>	Nr	t	MW [W]	T1 [°C]	T2 [°C]		1	00:15:00	1800	200	0		2	00:10:00	1800	200	0		3	00:00:00					
Nr	t	MW [W]	T1 [°C]	T2 [°C]																						
1	00:15:00	1800	200	0																						
2	00:10:00	1800	200	0																						
3	00:00:00																									

ICP-OES PARAMETERS, AGILENT ICP-OES (710 SERIES)

Power	1.30 kW
Plasma Flow	15.0 L/min
Auxiliary Flow	1.50 L/min
Nebulizer Flow	0.75 L/min
Replicate read time	10 s
Instrument stabilization delay	15 s
Sample Uptake Delay	30 s
Pump Rate	15 rpm
Rinse Time	10 s
Replicates	3

INDUSTRY REPORT

ETHOS UP | POLYMER



| ANALYTICAL RESULTS

ICP-OES RESULTS

Polyethylene (EC680) – QS 50 Quartz inserts (All results are expressed in mg/Kg)

Replicate	As	Cd	Cr	Pb
1	30.0	144.6	119.8	113.3
2	28.80	141.5	116.7	110.1
3	28.72	141.2	115.9	110.8
Average	29.17 ± 0.72	142.4 ± 1.88	117.5 ± 2.06	111.4 ± 1.68

	Average value	Certified value	Recovery
As	29.17 ± 0.72	30.9 ± 0.7	94.40 %
Cd	142.4 ± 1.88	140.8 ± 2.5	101.1 %
Cr	117.5 ± 2.06	114.6 ± 2.6	102.5 %
Pb	111.4 ± 1.68	107.6 ± 2.8	103.5 %

Polyethylene (EC680K) – QS 50 Quartz inserts (All results are expressed in mg/Kg)

Replicate	As	Cd	Pb	Sb	Zn
1	3.75	19.60	13.18	9.22	135.3
2	3.87	19.50	13.20	10.18	134.0
3	4.05	19.45	13.85	9.33	135.6
Average	3.89 ± 0.2	19.5 ± 0.1	13.4 ± 0.4	9.58 ± 0.5	135.0 ± 0.9

	Average value	Certified value	Recovery
As	3.89 ± 0.2	4.1 ± 0.5	94,88 %
Cd	19.5 ± 0.1	19.6 ± 1.4	99,49 %
Pb	13.4 ± 0.4	13.6 ± 0.5	98,53 %
Sb	9.58 ± 0.5	10.1 ± 1.6	94,85 %
Zn	135 ± 0.9	137 ± 20	98,54 %

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

| CONCLUSION

Polyethylene samples were prepared for elemental analysis using quartz micro-inserts configuration (vessel-inside-vessel technology). They were digested and then analysed for heavy metals. Micro-inserts demonstrate to provide a robust and reproducible way to prepare reactive polymer samples for trace metal analysis with low acid volumes. The data reported in this industry report shows that the “Vessel-Inside-Vessel Technology” is a great solution for laboratories that need to examine and analyze different polymers material employing small volume of acids.



MILESTONE
H E L P I N G
C H E M I S T S

MILESTONE Srl - Via Fatebenefratelli, 1/5 - 24010 Sorisole (BG) - Italy
Tel: +39 035 573857 - Fax: +39 035 575498
www.milestonesrl.com - email: analytical@milestonesrl.com