



## Enabling high throughput in elemental analysis of several food matrices using the Milestone ETHOS UP with MAXI-24 HP

### INTRODUCTION

Demand for trace metals analysis in the food industry is growing strongly due to stricter food regulations such as the recent Food Safety Modernization Act. ICP has been the standard for metals analysis for food, but as demand for lower levels of detection grows, the industry is experiencing a significant transition to ICP-MS. This transition is placing increased emphasis on the sample preparation method. Traditional sample preparation techniques for food include hot block digestion, closed vessel microwave digestion, and ashing; each of them posing different challenges.

Hot block digestions suffer from long digestions, airborne contamination, poor

digestion quality, and poor recovery of volatile compounds. Closed vessel microwave digestion has proven to be an effective technique with fast, complete digestions, a clean environment, and full recovery of volatile compounds.

Due to variability of the food samples in term of reactivity, most often the analysts have to compromise either performance or throughput.

Today, thanks to the latest Milestone development, a new option is available on: the MAXI-24 High Performance (HP). This innovative solution perfectly integrates with the powerfull ETHOS UP, matching both performance and throughput requirements of food elemental analysis.

## INDUSTRY REPORT ETHOS UP – MAXI-24 HP | FOOD



### I EXPERIMENTAL

In this industry report, a recovery study on certified reference food materials has been performed to validate the ETHOS UP with MAXI-24 HP for sample preparation and consequent metal analysis.

#### INSTRUMENT

The ETHOS UP is the most advanced microwave sample preparation equipment, it meets the requirements of modern analytical labs.



Figure 1 – Milestone's ETHOS UP

The ETHOS UP used in this study was equipped with MAXI-24 HP rotor controlled via Milestone's easyTEMP contactless temperature. The superior temperature measurement of easyTEMP allows the processing of different samples of similar reactivities, thus reducing labor time and increasing the overall throughput.

#### MAXI-24 HP ROTOR

The latest Milestone's development is the MAXI-24 HP, which combines performance and throughput within a single rotor-based platform. It completely innovates the rotor-base solutions providing high throughput without sacrificing the performance. Thanks

to its 24 positions, it is the first high pressure and throughput rotor available in the market. The completely new design of its vessels allows to achieve conditions never seen for high throughput rotors. Thicker high purity PTFE-TFM vessels and caps, along with rugged PEEK shields are key ingredients to handle the conditions required to completely digest these samples.



Figure 2 – MAXI-24 HP Rotor

#### PROCEDURE

ETHOS UP – MAXI-24 HP	
SAMPLE	SAMPLE AMOUNT*
Oyster (IAEA-470)	0.6 g
Milk powder (ERM-BD150)	0.6 g
Fish protein (DORM 4)	0.6 g
Lobster hepatopancreas (TORT-3)	0.6 g

Table 1 - Sample list and acid mixture

\*Acid Mixture: 5 mL of HNO<sub>3</sub> (65%), 1 mL H<sub>2</sub>O<sub>2</sub> (30%)

Approximately 0.6 g of each sample was weighted into the MAXI-24 HP vessels (as reported in table 1). The acid mixture (trace metal grade) was added according to the data reported in table 1 and the proper microwave method has been used as reported in table 2.

# INDUSTRY REPORT

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STEP	TIME	T2	POWER
1	00:10:00	160°C	1800 W
2	00:15:00	200°C	1800 W
3	00:10:00	200°C	1800E W

Table 2 – MAXI-24 HP microwave program

After microwave digestion, the samples were diluted to 50 mL with DI water and then, analyzed in ICP-OES.

### 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's ETHOS UP powered by MAXI-24 HP rotor was evaluated through a recovery study on Oyster (IAEA-470), milk powder (ERM-BD150), fish protein (DORM 4) and lobster hepatopancreas (TORT-3). The samples were digested with Milestone's ETHOS UP and subsequently analyzed via ICP-OES.

	Certified value (mg/Kg)	Recovery % (n=3)	RSD (%) (n=3)
Ag	1.29 ± 0.10	93.4	2.4
As	11.9 ± 0.9	101.0	2.7
Ca	2430 ± 280	98.2	0.4
Cd	3.14 ± 0.24	96.2	1.1
Co	0.201 ± 0.025	<LOQ	-
Cr	0.97 ± 0.11	<LOQ	-
Cu	146 ± 13	89.2	2.3
Fe	131 ± 12	93.0	2.9

Hg	0.0211 ± 0.0021	<LOQ	-
Mg	3080 ± 3.90	92.8	0.9
Mn	66.7 ± 5.3	94.4	1.4
Na	19700 ± 2300	95.3	2.3
Pb	0.361 ± 0.053	<LOQ	-
Se	3.06 ± 0.33	91.3	1.8
Sr	20.6 ± 1.6	91.3	1.2
V	0.90 ± 0.13	<LOQ	-
Zn	727 ± 48	100.4	2.4

Table 3- Recovery study on Oyster (IAEA-470) digested in MAXI-24 HP.

	Certified value (mg/Kg)	Recovery % (n=3)	RSD (%) (n=3)
Ca	13900 ± 800	91.4	2.1
Cd	0.0114 ± 0.0029	<LOQ	-
Cu	1.08 ± 0.06	109.3	2.9
Fe	4.6 ± 0.5	98.2	1.3
Hg	0.060 ± 0.007	89.8 <sup>a</sup>	2.7
K	17000 ± 700	90.6	1.7
Mg	1260 ± 100	94.8	2.6
Mn	0.289 ± 0.018	91.3	0.9
Na	4180 ± 190	92.6	1.8
P	11000 ± 600	99.5	2.1
Pb	0.019 ± 0.004	<LOQ	-
Se	0.188 ± 0.014	<LOQ	-
Zn	44.8 ± 2.0	102.1	2.1

Table 4- Recovery study on milk powder (ERM-BD150) digested in MAXI-24 HP.

	Certified value (mg/Kg)	Recovery % (n=3)	RSD (%) (n=3)
As	6.80 ± 0.64	91.0	1.8
Cd	0.306 ± 0.015	<LOQ	-
Cr	1.87 ± 0.16	91.9	2.1
Cu	15.9 ± 0.9	90.7	1.7
Fe	341 ± 27	94.2	2.3
Hg	0.410 ± 0.055	94.1 <sup>a</sup>	2.6
Ni	1.36 ± 0.22	96.7	1.3
Pb	0.416 ± 0.053	<LOQ	-
Se	3.56 ± 0.34	91.5	2.8
Zn	52.2 ± 3.2	92.0	2.4

Table 5- Recovery study on fish protein (DORM-4) digested in MAXI-24 HP.

<sup>a</sup> Analyzed with ICP cold vapor generator module.

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	Certified value (mg/Kg)	Recovery % (n=3)	RSD (%) (n=3)
As	59.5 ± 3.8	104.5	2.2
Cd	42.3 ± 1.8	89.5	1.9
Cr	1.95 ± 0.24	90.5	2.7
Cu	497 ± 22	88.7	1.4
Fe	179 ± 8	92.2	1.1
Hg	0.292 ± 0.022	93.5	1.9
Mn	15.6 ± 1.0	94.4	2.6
Mo	3.44 ± 0.12	101.1	2.7
Ni	5.30 ± 0.24	92.6	1.6
Pb	0.225 ± 0.018	<LOQ	-
Se	10.9 ± 1.0	96.1	3.0
Sr	36.5 ± 1.6	98.3	1.9
V	9.1 ± 0.4	107.2	2.4
Zn	136 ± 6	94.2	1.3

Table 6- Recovery study on lobster hepatopancreas (TORT-3) digested in MAXI-24 HP.

The analytical results were shown in Tables 3 to 6 with good recoveries of all elements and RSDs below 3%. This demonstrates the robustness and reproducibility of microwave digestion process using the ETHOS UP equipped with MAXI-24 HP and easyTEMP.

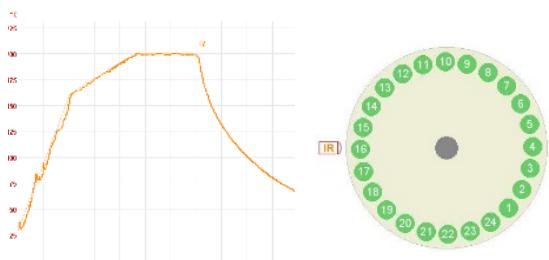


Figure 3 – MAXI-24 HP Microwave Run Report and Multiple temperature traceability

Figure 3 shown the temperature profile of the digestion as well as the multiple temperature visualization and recording for all the samples digested in the run.

## CONCLUSION

The data shown in this industry report demonstrates full recovery of the elements reported in the certificates of the reference material.

The ETHOS UP with MAXI-24 HP was successfully applied in digesting several food matrices, even highly reactive, ensuring superior digestion quality and reliable results.

This configuration provides a complete solution for food testing laboratories, enabling, at the same time and on a wide variety of matrices, the processing of high sample mass and high throughput.

In addition, microwave digestion using the Milestone ETHOS UP with easyTEMP control, provides the highest level of reproducibility and great ease of use, ensuring high quality digestion run after run.

## ABOUT MILESTONE

At Milestone we help chemists by providing the most innovative technology for metals analysis, direct mercury analysis and the application of microwave technology to extraction, ashing and synthesis. Since 1988 Milestone has helped chemists in their work to enhance food, pharmaceutical and consumer product safety, and to improve our world by controlling pollutants in the environment.



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