



**ADVANCED MICROWAVE DIGESTION
PERFORMANCE FOR HIGH-VOLUME FOOD
LABORATORIES**

Enabling high-throughput, quality metals testing of various food samples using the Milestone ETHOS UP with MAXI-24 HP

| INTRODUCTION

Demand for trace metals analysis in the food industry is rapidly increasing due to stricter food regulations that are now in place, including the 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 seismic shift to ICP-MS. This transition is placing increased emphasis on the sample preparation method employed. Traditional sample preparation techniques for food include hot block digestion, closed vessel microwave digestion, and ashing, each of which poses 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.

The reactivity of the samples that food laboratories encounter varies greatly, as do the temperatures required for complete digestion. Historically, analysts have had to choose between rotors that either delivered high throughput or high performance.

Milestone's ETHOS UP equipped with MAXI-24 High Performance (HP) rotor delivers both the digestion performance and throughput requirements that food testing and related industry labs require.



EXPERIMENTAL

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

not possible with traditional high-throughput rotors. To provide these enhanced capabilities, the MAXI-24 HP was designed with thicker high-purity TFM vessels and caps, and rugged PEEK shields.

INSTRUMENT



Figure 1 – Milestone's ETHOS UP



Figure 2 – MAXI-24 HP Rotor

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

MAXI-24 HP ROTOR

The Milestone ETHOS UP equipped with MAXI-24 High Performance (HP) rotor delivers both high-throughput and high temperature/pressure capability in a single, easy-to-use solution. This results in high-quality data and more profitable runs, all without compromising performance. The design of the MAXI-24 HP allows for up to 24 samples to be processed simultaneously and achieve temperatures and pressures

PROCEDURE

SAMPLE	SAMPLE AMOUNT	ACID MIXTURE
Oyster (IAEA-470)	0.6 g	5 mL of HNO ₃ + 1 mL of H ₂ O ₂
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

First, approximately 0.6 g of each sample was weighted into separate MAXI-24 HP vessels and acid mixtures (trace metals grade) added, as reported in Table 1. Next, a microwave method suitable for all samples was used as outlined in Table 2 below.

STEP	TIME	TEMP	POWER
1	00:10:00	160°C	1800 W
2	00:15:00	210°C	1800 W
3	00:10:00	210°C	1800 W

Table 2 – MAXI-24 HP microwave program used to digest samples

INDUSTRY REPORT

ETHOS UP – MAXI-24 HP | FOOD



After microwave digestion, the samples were diluted to 50 mL with deionized (DI) water and subsequently analyzed via ICP-OES.

QUANTIFICATION

ICP-OES 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

Table 3- ICP-OES Parameters

RESULTS AND DISCUSSION

The performance of the 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).

	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 4- Recovery study on Oyster (IAEA-470) digested with the 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 ^a	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 5- Recovery study on milk powder (ERM-BD150) digested with the 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 ^a	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 6- Recovery study on fish protein (DORM-4) digested with the MAXI-24 HP.

^a Analyzed with ICP cold vapor generator module.

	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 7- Recovery study on lobster hepatopancreas (TORT-3) digested with the MAXI-24 HP.

The analytical results shown in Tables 4 to 7 demonstrate good recoveries of all elements of interest and RSDs below 3%. These results demonstrate the robustness and reproducibility of microwave digestion process using the ETHOS UP equipped with MAXI-24 HP and easyTEMP technology.

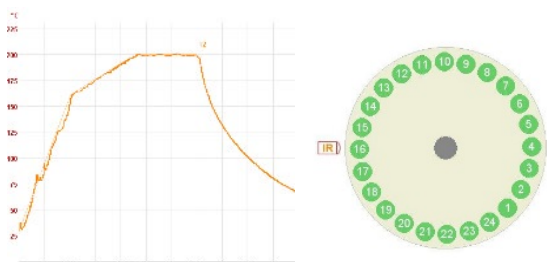


Figure 3 – MAXI-24 HP Microwave Run Report and Multiple temperature traceability

Figure 3 shows the temperature profile of the digestion, the multiple temperature visualization, and a record of all samples in the run.

CONCLUSION

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

The ETHOS UP with MAXI-24 HP successfully digested a variety of food matrices, ensuring superior digestion quality and reliable results.

This configuration provides a complete solution for food testing and related industry laboratories, delivering the high performance and throughput these labs require.

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.