

THE BENEFITS OF **MICROWAVE DIGESTION** APPLIED ON SEVERAL **INDUSTRIES**

ETHOS UP microwave digestion system is the most powerful microwave digestion system on the market today. Featuring the highest throughput rotors, stainless steel construction and patented vent-and-reseal technology, ETHOS UP ensures market-leading safety and productivity.

- **SAFETY AND RELIABILITY**
- **PERFORMANCE AND THROUGHPUT**
- **EASE OF USE AND CONTROL**
- **FLEXIBILITY**



ETHOS UP

High Performance
Microwave Digestion System

USING **ETHOS UP** FOR TRACE METAL ANALYSIS



SAMPLE PREPARATION OF
HIGH-VOLUME FOOD
SAMPLES



SAMPLE PREPARATION OF
ENVIRONMENTAL
SAMPLES



SAMPLE PREPARATION OF
LITHIUM ION BATTERIES
SAMPLES



SAMPLE PREPARATION OF
PHARMACEUTICAL
SAMPLES



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 powerful ETHOS UP, matching both performance and throughput requirements of food elemental analysis.



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.

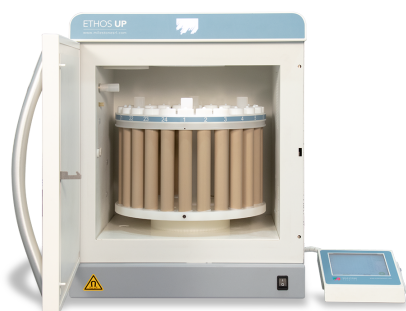


Figure 1 – Milestone's ETHOS UP

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.

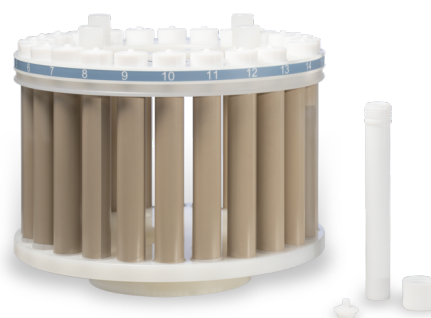


Figure 2 – MAXI-24 HP Rotor

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 HNO₃ (65%), 1 mL H₂O₂ (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

ETHOS UP – MAXI-24 HP | FOOD



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

^a Analyzed with ICP cold vapor generator module.

INDUSTRY REPORT

ETHOS UP – MAXI-24 HP | FOOD



	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.

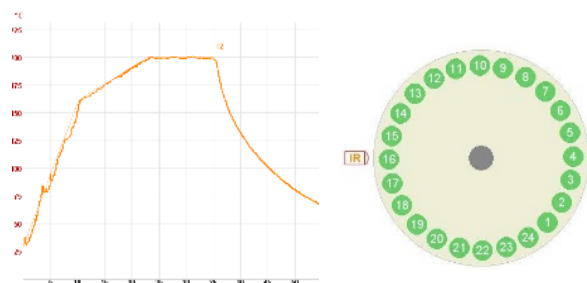


Figure 3 – MAXI-24 HP Microwave Run Report and Multiple temperature traceability

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.



SAMPLE PREPARATION OF ENVIROMENTAL SAMPLES FOR TRACE METAL ANALYSIS

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

| INTRODUCTION

Demand for trace metals analysis in environmental laboratories is growing strongly due to stricter environmental regulations. ICP has been the standard for metals analysis, but as demand for lower levels of detection grows, the laboratories are experiencing a significant transition to ICP-MS. This transition is placing increased emphasis on the sample preparation method. Traditional sample preparation techniques for environmental matrices include hot block digestion, closed vessel microwave digestion and ashing; all of which include different challenges. Hot block digestions suffer from long run times, 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.

Closed vessel microwave digestion is now included in the US EPA official sample preparation methods for most environmental samples.

ETHOS technology is perfectly designed for the three US EPA methods:

- EPA 3015A: Microwave assisted acid digestion of aqueous samples and extracts.
- EPA 3051A: Microwave assisted acid digestion of sediments, sludges, soils and oils.
- EPA 3052: Microwave assisted acid digestion of siliceous and organically based matrices.



The Milestone 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.

| EXPERIMENTAL

In this technical note, a recovery study on certified reference environmental materials has been 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 modern 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 determination in many applications. Its construction of stainless steel coated with five PTFE layers accommodates both high-pressure and high-throughput rotors.



Figure 1 – Milestone's ETHOS UP

easyTEMP

Milestone's easyTEMP contactless 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 reactivity, thus reducing labor time and increasing overall throughput.

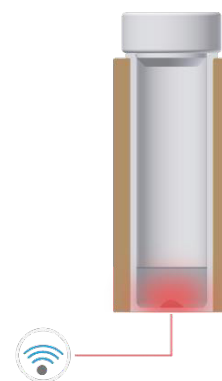


Figure 2 – easyTEMP contactless direct temperature sensor.

This technology combines the fast and accurate 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 ability to digest 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.

MAXI-44 HIGH THROUGHPUT ROTOR

The MAXI-44 is a high throughput rotor capable of digesting a large variety of environmental samples, improving throughput in the lab.

The MAXI-44 is fully controlled by contactless temperature/pressure sensors that directly control each vessel. This assures maximum safety and digestion quality.



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 provides full control of all digestion parameters, provides complete documentation 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, Multilanguage 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 5 - easyCONTROL built-in library



ANALYTICAL PROCEDURE

ETHOS UP

ROTOR	SAMPLE	SAMPLE AMOUNT	ACID MIXTURE	Reference official method
SK-15 easyTEMP	Sandy Loam soil (CRM 027)	0.5 g	9 mL of HNO ₃ 65%, 3 mL HCl 37%	EPA 3051A
	Lake sediment (BCR 280R)	0.5 g	9 mL of HNO ₃ 65%, 3 mL HCl 37%	EPA 3051A
	Fly ash (BCR 176R)	0.5 g	8 mL of HNO ₃ 65%, 1 mL HCl 37%, 1 mL HF 48%	UNI EN 14385
MAXI-44 easyTEMP	Sandy Loam soil (CRM 027)	0.5 g	9 mL of HNO ₃ 65%, 3 mL HCl 37%	EPA 3051A
	Lake sediment (BCR 280R)	0.5 g	9 mL of HNO ₃ 65%, 3 mL HCl 37%	EPA 3051A

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

SK-15 eT method and microwave run report:

STEP	TIME	T2	POWER
1	00:20:00	210 °C	1800 W
2	00:15:00	210 °C	1800 W

Table 2 – SK 15 microwave program used for digestion of samples

- Final dilution: 50 mL with deionized water

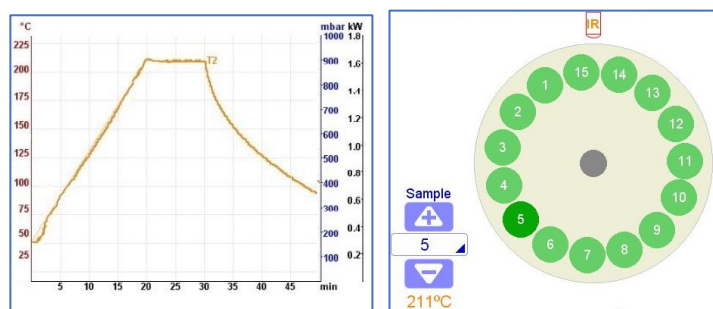


Figure 6 – SK-15 Microwave Run Report and Multiple temperature traceability

MAXI-44 eT method and microwave run report:

STEP	TIME	T2	POWER
1	00:10:00	150 °C	1800 W
2	00:10:00	180 °C	1800 W
3	00:10:00	180 °C	1800 W

Table 3 – MAXI-44 microwave program used for digestion of Samples

- Final dilution: 50 mL with deionized water

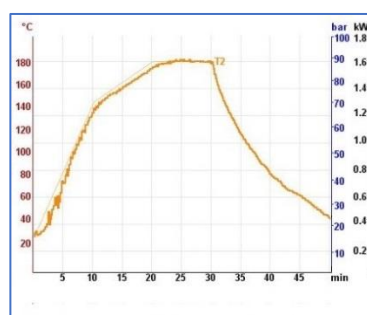


Figure 7 – MAXI-44 Microwave Run Report and Multiple temperature traceability

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.



I RESULTS AND DISCUSSION

The performance of the Milestone ETHOS UP equipped with SK-15 easyTEMP rotor was evaluated through a recovery study on sandy loam soil (CRM027), lake sediment (BCR280R), fly ash (BCR 176R). The samples were digested with Milestone's ETHOS UP and subsequently analyzed via ICP-OES.

	Certified value (mg/Kg)	SK-15 eT		MAXI-44 eT	
		Recovery % (n=3)	RSD (%)	Recovery % (n=3)	RSD (%)
As	59.0 ± 0.939	93.1	1.9	90.5	1.8
Ba	233 ± 4.27	107.3	1.5	97.9	1.9
Be	59.5 ± 1.06	88.9	0.8	87.9	2.0
B	79.6 ± 3.18	102.6	2.6	99.0	2.1
Cd	98.7 ± 1.64	93.6	0.5	93.0	1.7
Co	153 ± 2.67	94.8	2.4	98.0	1.4
Cr	240 ± 3.82	103.8	2.9	99.6	1.9
Hg ^a	16.0 ± 0.327	81.9	1.1	83.1	2.3
Mo	56.4 ± 1.36	101.1	1.1	99.5	2.1
Ni	298 ± 5.20	94.3	1.0	93.0	1.4
Pb	276 ± 4.59	91.3	2.5	87.0	2.6
Cu	89.6 ± 1.66	111.6	2.4	106.4	2.7
Se	100.00 ± 1.59	99.1	1.1	97.6	1.6
Sn	90.7 ± 2.63	88.	1.6	88.6	1.2
Tl	128 ± 2.96	93.0	1.6	92.2	1.1
V	201 ± 2.70	103.0	2.8	99.0	2.1
Zn	590 ± 9.75	94.7	1.0	96.1	1.8

Table 4-- Data of the recovery study on sandy loam soil CRM027.

^a Analyzed with ICP cold vapor generator module.

	Certified value (mg/Kg)	SK-15 eT		MAXI-44 eT	
		Recovery % (n=3)	RSD (%)	Recovery % (n=3)	RSD (%)
As	33.4 ± 2.9	102.7	1.7	98.4	2.1
Co	16.8 ± 0.9	94.4	2.2	91.3	2.4
Cr	126 ± 7	98.5	2.9	95.2	2.0
Cu	53 ± 6	89.9	2.2	90.3	1.7
Ni	69 ± 5	75.9	1.8	71.6	1.8
Zn	224 ± 25	91.8	1.9	93.4	2.1

Table 5- Data of the recovery study on lake sediment BCR280R.



SK-15 easyTEMP ^a			
	Certified value (mg/Kg)	Recovery % (n=3)	RSD (%)
As	54 ± 5	103.1	1.0
Cd	226 ± 19	91.2	2.8
Co	26.7 ± 1.6	97.0	0.7
Cr	810 ± 70	101.2	1.5
Cu	1050 ± 70	97.1	2.7
Fe	13100 ± 500	100.0	2.3
Ni	117 ± 6	98.3	1.4
Pb	5000 ± 500	98.8	1.4
Se	18.3 ± 1.9	105.7	1.2
Zn	16800 ± 400	107.1	3.0

Table 6 - Data of the recovery study on fly ash BCR 176R.

^a SK-15 easyTEMP is the recommended rotor for the sample preparation of fly ash samples

The analytical results are shown in tables 4, 5 and 6 with good recoveries of all elements and RSDs below 3%. This demonstrates the robustness and reproducibility of microwave digestion using the ETHOS UP equipped with SK-15 easyTEMP technology.

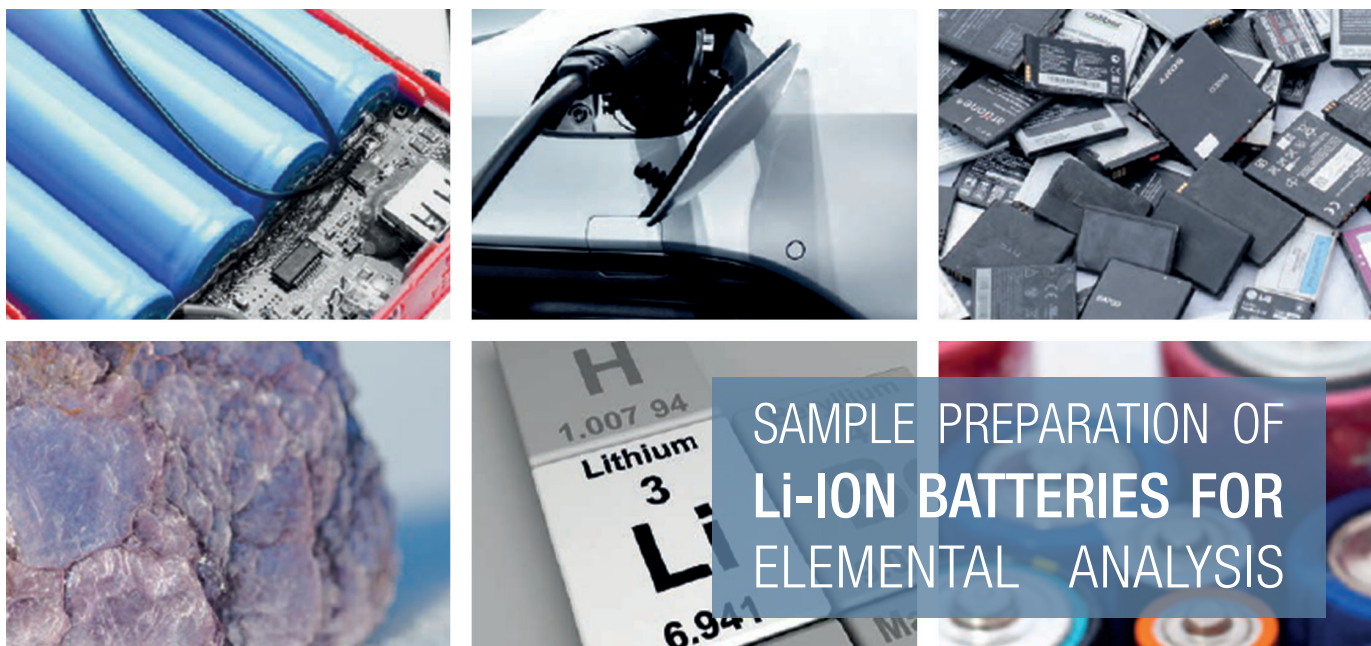
CONCLUSION

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

Milestone's ETHOS UP with SK-15 and MAXI-44 easyTEMP rotors demonstrates full compatibility with official EPA environmental methods with accurate and full control of the digestion process. The easyTEMP sensor ensures superior digestion quality and reliable results even for large amounts of different samples with similar reactivities. 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.

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.



High throughput, elemental analysis of Li-ion battery samples using Milestone's ETHOS UP with SK-15 easyTEMP (eT) rotor.

| INTRODUCTION

In recent years, efforts to combat climate change have intensified, leading to new research and innovations that are paving the way for the eventual transition away from combustion engines to cleaner electric vehicles. A central challenge of this transition is in addressing the current technological limitations within lithium-ion batteries. Before a broad shift from combustion, gasoline-powered vehicles can occur, battery performance, efficiency, and lifespan must dramatically improve. The chemical analysis of battery components is a necessary step in this process. More specifically, thorough qualitative and quantitative elemental analyses of the anode, cathode, and electrolyte materials present in batteries are required. These types of analyses are performed using ICP-based analyzers, which require solid samples to be decomposed and dissolved in an acid solution through a digestion process before the digested solutions are then injected into the analyzer.

The materials present in batteries are primarily of inorganic origin, which are very often stable and resistant materials that make acid dissolution complicated. For each sample matrix there is a specific acid mixture required (depending on the composition of the material) and that mixture must be used concurrently with high temperature and pressure conditions to achieve complete digestion of the materials before analysis. Closed-vessel microwave digestion is a proven technique, capable of achieving rapid sample digestions. More importantly, microwave digestion enables superior analytical accuracy when compared to other techniques through its higher temperature and pressure capabilities that ensure complete digestions, along with built-in safeguards that prevent analyte losses and contamination inherent to open-vessel techniques. Milestone's ETHOS UP microwave digestion system incorporates all of the benefits of closed-vessel

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ETHOS UP | Li-ION BATTERIES



microwave digestion while making sample preparation fast, easy, and effective.

In the following test, we demonstrate the system's performance for mineralizing two commonly-employed types of cathodes within the battery industry.

EXPERIMENTAL

INSTRUMENT

ETHOS UP is the most advanced rotor-based microwave sample preparation system available today, meeting the requirements for a wide variety of modern analytical labs.



Figure 1: Milestone's ETHOS UP

The ETHOS UP used in this study was equipped with an SK-15 eT rotor, controlled by Milestone's easyTEMP contactless temperature sensor. The superior temperature measurement of easyTEMP allows different samples of similar reactivities to be processed concurrently, reducing lab labor while increasing overall throughput.

SK-15 eT HIGH PRESSURE ROTOR

The SK-15 eT suits the analytical needs for determining trace elements in challenging samples, thanks to its

capability to digest large sample amounts and its ability to attain higher temperature and pressure conditions.

The 15-position, high-pressure rotor is safely controlled via the easyTEMP sensor that consistently monitors and controls digestion temperature within each vessel, ensuring complete digestion of even the most difficult and reactive samples.



Figure 2: SK-15 easyTEMP (eT) High Pressure Rotor

REAGENTS

- HNO_3 , nitric acid, 65%, ACS reagent (Sigma-Aldrich)
- HCl , hydrochloric acid, 37%, ACS reagent (Sigma-Aldrich)
- Periodic table mix 1 for ICP, 10 mg/L (TraceCERT, Sigma-Aldrich): Al, As, Ba, Be, Bi, B, Ca, Cd, Cs, Cr, Co, Cu, Ga, In, Fe, Pb, Li, Mg, Mn, Ni, P, K, Rb, Se, Si, Ag, Na, Sr, S, Te, Tl, V, and Zn in 10% V/V nitric acid (contains HF traces)
- Periodic table mix 2 for ICP, 10 mg/L (TraceCERT, Sigma-Aldrich): Au, Ge, Hf, Ir, Mo, Nb, Pd, Pt, Re, Rh, Ru, Sb, Sn, Ta, Ti, W, and Zr in 5% V/V hydrochloric acid and 1% V/V hydrofluoric acid (contains HNO_3 traces)
- Yttrium standard for ICP, 10000 mg/L (TraceCERT, Sigma-Aldrich)

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ETHOS UP | Li-ION BATTERIES



ANALYTICAL PROCEDURE

ETHOS UP with SK-15 eT rotor	
SAMPLE	SAMPLE AMOUNT*
NCA—Lithium Nickel Cobalt Aluminum Oxide	0.5 g
NMC—Lithium Nickel Manganese Cobalt Oxide	0.5 g

* Acid Mixture: 2 mL of HNO_3 (65%), 6 mL HCl (37%).
Table 1 - Sample list and amount digested.

Approximately 0.5 g of each sample was placed into individual SK-15 eT vessels (as reported in table 1). The acid mixture (trace metal grade) was then added to each vessel (according to the data reported in Table 1). Three replicates out of six were spiked with 250 μL of the periodic table mix 1 (solution c) and mix 2 (solution d) ICP standards respectively, immediately after sample weighing and prior to reagent addition. These samples were used for the recovery studies performed to validate the method.

The vessels were sealed and loaded into the rotor, the rotor was placed in the microwave cavity, and microwave method (reported in Table 2) was run.

Step	Time	T2	Power
1	00:25:00	230 °C	1800 W
2	00:15:00	230 °C	1800 W

Table 2: SK-15 eT microwave program.

After microwave digestion, the sample solutions were spiked with Yttrium internal standard solution*, diluted with DI water, and subsequently analyzed by ICP-OES.

*10 $\mu\text{g}/\text{mL}$ of Yttrium standard (e) was added to calibration standards, blanks, and digested/diluted sample solutions as an internal standard to correct for matrix effects.

The instrument setup and operating conditions are reported in the following table:

Parameter	Setting
RF applied power (kW)	1.3
Plasma gas flow rate (L/min)	15
Auxiliary gas flow rate (L/min)	1.5
Nebulizer gas flow rate (L/min)	0.75
Replicate read time (s)	5
Stabilization delay (s)	30
Sample uptake delay (s)	30
Pump rate (rpm)	15
Rinse time (s)	15
Replicates	3
Emission lines (nm)	See Tables 4 and 5

Table 3 - ICP-OES settings and operating conditions.

RESULTS AND DISCUSSION

The performance of Milestone's ETHOS UP, powered by the SK-15 eT rotor, was evaluated through a recovery study on spiked samples.

After the digestion run, we obtained transparent solutions that contained no visible solid particles, indicating complete digestion of the samples.

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ETHOS UP | Li-ION BATTERIES



The recoveries below were obtained via ICP-OES analysis:

Element and wavelength (nm)	Determined concentration (µg/L)	RSD % (n=3)	Spiked sample concentration (µg/L)	RSD % (n=3)	Spike recovery (%)
Ag 328.068	<MDL	-	81.5	8.11	102
As 193.696	<MDL	-	81.8	2.15	102
Ba 455.403	<MDL	-	75.03	2.39	94
Be 313.107	<MDL	-	75.8	3.72	95
Cd 214.439	<MDL	-	71.7	2.67	90
Cr 267.716	<MDL	-	73.8	3.88	92
Cu 324.754	<MDL	-	74.3	7.41	93
Fe 238.204	65.9	1.93	138	1.82	91
Mn 257.610	<MDL	-	77.0	3.48	96
Mo 202.032	<MDL	-	72.5	3.77	91
Na 588.995	13.5	15.2	95.7	3.9	103
Nb 309.417	71.9	0.29	143	3.51	89
Pb 220.353	<MDL	-	91.9	4.44	115
Se 196.026	15.5	3.77	89.5	6.55	93
Sn 189.925	<MDL	-	75.9	5.26	95
Sr 407.771	12.6	0.56	86.2	3.18	92
Ti 336.122	<MDL	-	71.7	2.39	90
V 292.401	25.3	1.96	97.1	2.68	90
Zn 213.857	11.4	265	86.3	1.54	94

Table 4– Data of the recovery of NCA sample (The digested solutions were further diluted by 1:2 V/V with water to lower their acid concentration prior to ICP-OES analysis. The final spiked concentration was 80 µg/L)

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ETHOS UP | Li-ION BATTERIES



Element and wavelength (nm)	Determined concentration (µg/L)	RSD % (n=3)	Spiked sample concentration (µg/L)	RSD % (n=3)	Spike recovery (%)
Ag 338.289	145	1.44	191	1.60	93
As 188.980	121	3.35	178	2.03	115
Ba 455.403	<MDL	-	49.9	1.48	100
Be 234.861	<MDL	-	45.3	2.11	91
Cd 214.439	40.9	8.43	98.4	2.81	115
Cu 324.754	<MDL	-	44.8	0.93	90
K 766.491	106	2.59	156	0.64	100
Mo 202.032	<MDL	-	44.9	5.35	90
Pb 220.353	32.5	3.41	80.1	2.88	95
Ru 267.876	50.8	6.21	97.6	0.58	94
Sn 189.925	<MDL	-	47.1	2.10	94
Sr 407.771	5.72	2.14	50.5	2.29	90
Ti 336.122	53.6	4.73	98.8	1.94	90
V 292.401	<MDL	-	52.8	2.08	106

Table 5— Data of the recovery of NMC sample (The digested solutions were further diluted by 1:2 V/V with water to lower their acid concentration prior to ICP OES analysis. The final spiked concentration was 80 µg/L)

CONCLUSION

The data shown in this industry report demonstrates full recovery of the elements of spiked solutions, validating the methods of sample preparation and analysis. The low RSD's indicate that the methods are reproducible. The ETHOS UP with SK-15 eT rotor was successfully applied in digesting several cathode samples, ensuring superior digestion quality and reliable results. This configuration provides a complete solution for digestion of a wide variety of Li-ion battery samples. 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

Milestone has been innovating microwave sample preparation for over 30 years and offers an array of solutions that are perfectly tailored for modern testing labs within the battery industry. With over 25,000 instruments installed worldwide, we are the acknowledged industry leader in microwave technology and offer the most technologically advanced instrumentation for research and quality control. For more information, please visit www.milestone srl.com.



SAMPLE PREPARATION OF PHARMACEUTICAL SAMPLES FOR TRACE METAL ANALYSIS

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

| INTRODUCTION

New USP chapters <232> and <233> for the measurement of inorganic contaminants in pharmaceutical samples have been implemented. While samples that are soluble in aqueous and organic solvents may be analyzed directly, a large proportion of samples will require digestion, and in fact digestion may be preferred for ICP-MS analysis even if the sample is soluble in organic solvent.

Closed vessel digestion is stipulated by USP and it is expected that microwave digestion will be the predominant digestion technique used. Microwave digestion can achieve high temperatures and pressures and offers many benefits when compared to

traditional sample preparation techniques such as hot plate.

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 of the highest quality.

| EXPERIMENTAL

In this industry report, a recovery study on spiked Avicel PH-101, magnesium stearate, capsules and dietary materials has been performed to prove the efficacy of the ETHOS UP the sample preparation for metals analysis.

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ETHOS UP | PHARMACEUTICAL



INSTRUMENT

The ETHOS UP meets the requirements of modern 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 determination in many applications. Its construction of stainless steel coated with five PTFE layers accommodates both high-pressure and high-throughput rotors.



Figure 1 – Milestone's ETHOS UP

easyTEMP

Milestone's easyTEMP contactless 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 reactivity, thus

reducing labor time and increasing overall throughput.

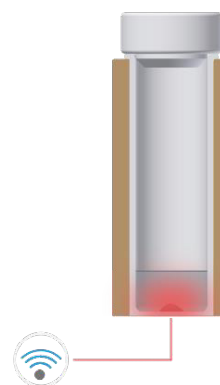


Figure 2 – easyTEMP contactless direct temperature sensor.

This technology combines the fast and accurate 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 ability to digest large sample amounts at high temperature (up to 300 °C) and pressure (up to 100 bar).



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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 provides full control of all digestion parameters, provides complete documentation 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
Avicel PH-101 (Cellulose microcrystalline)	0.5 g	4 mL of HNO ₃ 65%, 1 mL of H ₂ O ₂ 30%
Magnesium Stearate	0.5 g	10 mL of HNO ₃ 65%
Capsules	0.7 g	4 mL of HNO ₃ 65%, 1 mL of H ₂ O ₂ 30%
Dietary supplement	0.9 g	4 mL of HNO ₃ 65%, 1 mL of H ₂ O ₂ 30%

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

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STEP	TIME	T2	POWER
1	00:20:00	210 °C	1800 W
2	00:15:00	210 °C	1800 W

Table 2 – SK 15 microwave program used for digestion of samples

- Final dilution: 50 mL with deionized water

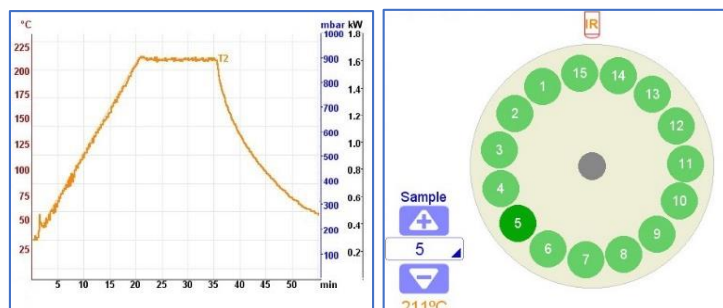


Figure 5 – SK-15 Microwave Run Report and Multiple temperature traceability

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 easyTEMP rotor

was evaluated through a recovery study on samples of interest for the pharma industry. All samples were fortified with a spike solution (Elemental Impurities according to ICH Q3D parenteral, Standard 1, ICP standard. Sigma-Aldrich code 89118-100ML) containing As, Cd, Co, Pb, Hg, Ni, Se, Ag, Tl and V (see spike concentration in Table 3). The sample were digested with Milestone's Ethos UP and subsequently analyzed via ICP-OES.

	As	Cd	Co	Pb	Hg ^a	Ni	Se	Ag	Tl	V
Spike value (mg/L)	15	2	5	5	3	20	80	10	8	10
Avicel PH-101										
Recovery % (n=3)	97.3	90.1	92.7	95.6	98.2	101.3	102.4	91.8	89.2	98.9
RSD (%)	0.6	1.8	0.8	2.5	1.3	1.6	0.4	1.7	2.4	2.6
Magnesium Stearate										
Recovery % (n=3)	107.2	94.2	93.6	97.6	95.3	98.4	103.8	97.5	91.4	97.3
RSD (%)	1.0	0.6	1.8	0.4	0.9	1.3	0.8	1.6	2.1	1.6
Capsules										
Recovery % (n=3)	91.1	92.3	95.3	102.3	97.6	93.4	104.3	96.2	92.2	98.3
RSD (%)	1.4	1.4	2.0	1.4	0.3	1.1	2.4	1.5	1.9	0.5
Dietary Supplement										
Recovery % (n=3)	100.2	95.4	94.3	95.2	97.5	96.7	101.6	93.4	92.3	101.2
RSD (%)	0.6	1.6	0.9	1.3	1.5	1.2	2.1	1.9	1.7	1.4

Table 3- Data of the recovery study on spiked samples of interest for Pharma

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

| CONCLUSION

The data shown in this industry report demonstrates full recovery of the elements spiked in the samples.

Samples, such as those from the pharmaceutical industry, 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 in alignment with the new USP requirements.

| 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.

WATCH VIDEO



QUALITY ANALYSIS
STARTS WITH
GREAT SAMPLE PREP



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

ETHOS UP
High Performance
Microwave Digestion System

Sample preparation is one of the most impactful steps in trace metals determination.

The quality of analytical data obtained by ICP, ICP-MS is directly reliant on digestion quality.

Milestone's ETHOS UP microwave digestion system maximizes the AA/ ICP/ ICP-MS performance, and a lab's overall analytical capability.