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Sample Preparation of Food Samples for Trace Metal Analysis

This report was extracted from a Milestone Industry Report on Ethos UP/Food

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.

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 certified reference food materials has been performed to prove the efficacy of ETHOS UP in the sample preparation for metal analysis.

Instrument

ETHOS UP

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 determinations in many applications. Its construction of stainless steel coated with five PTFE layers and accommodates both high-pressure and high-throughput rotors.

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 reactivities, thus reducing labor time and increasing overall throughput.

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.



Figure 1. Milestone's ETHOS UP.

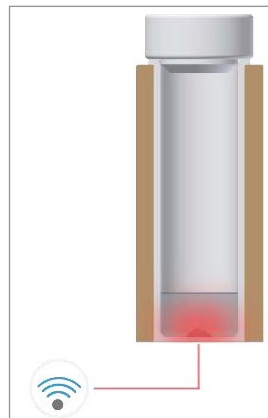


Figure 2. easyTEMP contactless direct temperature sensor.



Figure 3. SK-15 easyTEMP High Pressure Rotor.

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 capability for digesting large sample amounts at high temperature (up to 300 °C) and pressure (up to 100 bar).

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 digest a large variety of food samples, improving throughput in the lab.

The MAXI-44 is fully controlled by a contactless temperature/pressure sensor 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 fully control all the 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.



Figure 4. MAXI-44 easyTEMP High Throughput Rotor

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

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

ETHOS UP			
Rotor	Sample	Sample amount	Acid mixture
SK 15 easyTEMP	Lobster hepatopancreas (TORT-3)	1 g	5 mL of HNO ₃ 65%
	Bovine Liver (NIST 1577c)	1 g	5 mL of HNO ₃ 65%
	Diary Feed (BCR 708)	1 g	5 mL of HNO ₃ 65%
Maxi 44 easyTEMP	Lobster hepatopancreas (TORT-3)	0.5 g	5 mL of HNO ₃ 65%
	Bovine Liver (NIST 1577c)	0.5 g	5 mL of HNO ₃ 65%
	Diary Feed (BCR 708)	0.5 g	5 mL of HNO ₃ 65%

SK-15 eT method and microwave run report

Table 2. SK-15 eT microwave program used for digestion of samples

Step	Time	T2	Power
1	00:20:00	210 °C	1800 W
2	00:15:00	210 °C	1800 W

- Final dilution: 50 mL with deionized water

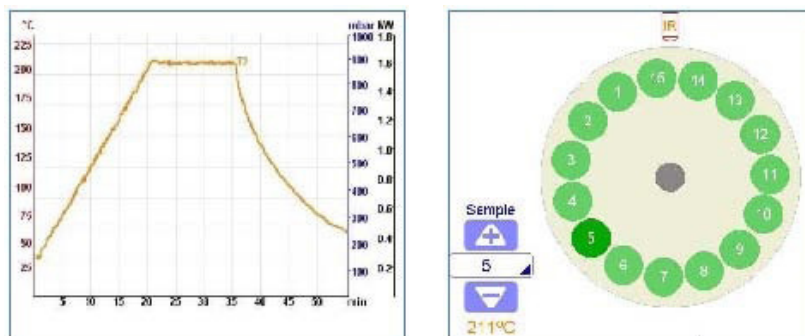


Figure 6. SK-15 Microwave Run Report and Multiple temperature traceability.

MAXI-44 eT method and microwave run report

Table 3. MAXI-44 eT microwave program used for digestion of samples

Step	Time	T2	Power
1	00:20:00	200 °C	1800 W
2	00:15:00	200 °C	1800 W

- Final dilution: 50 mL with deionized water

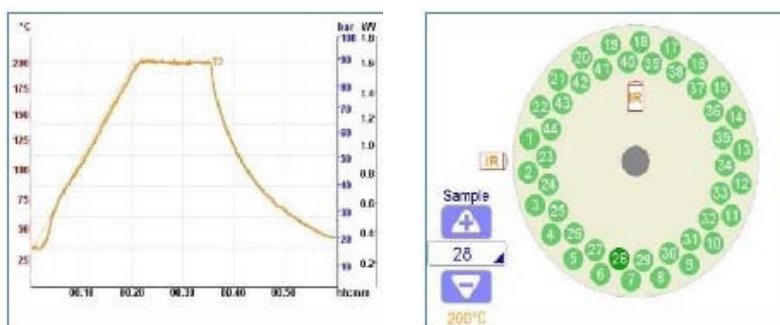


Figure 7. MAXI-44 Microwave Run Report and Multiple temperature traceability

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.

RESULTS AND DISCUSSION

The performance of the Milestone's ETHOS UP equipped with SK-15 easyTEMP rotor was evaluated through a recovery study on Lobster hepatopancreas (TORT-3), Bovine liver NIST (1577c), Dairy Feed (BCR 708). The sample were digested with Milestone's ETHOS UP and subsequently analyzed via ICP-OES.

The analytical results are shown in Table 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 and MAXI-44 easyTEMP technologies.

Table 4. Data of the recovery study on lobster hepatopancreas TORT-3

	Certified value (mg/Kg)	SK-15 eT		MAXI-44 eT	
		Recovery % (n=3)	RSD (%)	Recovery % (n=3)	RSD (%)
As	59.5 ± 3.8	102.8	2.5	108.2	2.9
Cd	42.3 ± 1.8	89.1	2.2	84.2	2.6
Cr	1.95 ± 0.24	96.5	2.9	95.4	2.5
Cu	497 ± 22	103.3	2.2	96.2	1.8
Fe	179 ± 8	93.5	2.9	94.1	2.3
Mn	15.6 ± 1.0	98.5	1.1	96.3	1.9
Ni	5.30 ± 0.24	102.1	1.0	103.2	2.1
Se	10.9 ± 1.0	94.1	1.9	97.8	1.8
Sr	36.5 ± 1.6	100.7	1.5	93.4	2.2
V	9.1 ± 0.4	83.7	2.3	84.5	2.3
Zn	136 ± 6	90.3	2.1	90.2	2.5

Table 5. Data of the recovery study on bovine liver NIST 1577c

	Certified value (mg/Kg)	SK-15 eT		MAXI-44 eT	
		Recovery % (n=3)	RSD (%)	Recovery % (n=3)	RSD (%)
Ca	131 ± 10	92.8	1.2	91.9	1.8
Cu	275.2 ± 4.6	91.6	1.4	91.8	2.3
Fe	197.94 ± 0.65	98.2	2.1	94.3	2.7
Mg	620 ± 42	101.2	1.6	96.2	1.2
Mn	10.46 ± 0.47	95.3	2.3	92.6	2.2
Mo	3.30 ± 0.13	104.6	1.4	99.4	2.3
Zn	181.1 ± 1.0	92.3	1.1	95.1	2.8

Table 6. Data of the recovery study on dairy feed BCR 708

	Certified value	SK-15 eT		MAXI-44 eT	
		Recovery % (n=3)	RSD (%)	Recovery % (n=3)	RSD (%)
Ca	4.8 ± 0.5 g/Kg	96.4	1.3	92.7	2.9
Cu	37 ± 4 mg/Kg	97.8	1.8	93.2	1.6
Mg	1.47 ± 0.22 g/Kg	99.6	1.2	95.4	2.4
P	4.7 ± 0.4 g/Kg	95.4	1.6	96.5	3.0

CONCLUSION

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

Highly reactive samples such as food can be completely digested even in large sample amounts along with different samples of similar reactivities. The digestion process was accurately controlled by the easyTEMP sensor, ensuring superior digestion quality and reliable results.

The ETHOS UP provides a complete solution for food laboratories, enabling the processing of high sample amount with SK-15 easyTEMP rotor as well as unmatched throughput with the MAXI-44 easyTEMP rotor.

In addition, 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.

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