



# Using Microwave Sample Prep to Determine Trace Metals Analysis in Food Matrices

## Digestion of Food Samples Using the Milestone Ethos UP

### Summary

The testing of foods is one of the fastest growing areas of analytical measurement. Due to stricter food regulations such as the recent Food Safety Modernization Act, there is greater emphasis on food quality and safety than ever before. Today's food labs are required to measure more analytes, at lower levels. ICP has always been the standard for metals analysis of food samples, but as demand for lower detection levels grows, the industry is experiencing a significant transition to ICP-MS, which is placing increased emphasis on the sample preparation

method and it is expected that microwave digestion will be the predominate sample preparation method used. Traditional sample prep techniques for food samples such as hot block pose various challenges. Hot block techniques 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 alternative to these methods with fast, complete digestions, clean environment, and full recovery of volatile compounds.

The Milestone Ethos UP microwave digestion system incorporates all of the benefits of closed vessel microwave digestion - speed, data quality, and ease-of-use, - all in a safe and compact benchtop system.

This technical note evaluates the digestion quality of the following certified reference materials:

- NIST 1577c: Bovine Liver
- TORT-3: Lobster Hepatopancreas

## Instrumentation

The ETHOS UP meets many of the requirements of today's food laboratory. It offers several unique benefits including:

- High throughput to increase productivity
- Flexibility to digest a variety of matrices
- Intuitive software
- Industry leading safety

The Ethos UP is a flexible and high performing platform used for trace elements and routine analysis in food laboratories. It uses 18/8 stainless steel construction, features a built-in camera and can accommodate both high-pressure and high-throughput rotors.



Figure 1. The Milestone Ethos UP

The Ethos UP includes 300 built-in digestion methods, which virtually eliminates method development. Additionally, the UP features Milestone Connect, which enables remote system control, 24/7 technical support and access to a comprehensive library of content developed especially for the analytical lab.

## SK-15 High Pressure Rotor

The SK-15 rotor is most suitable for food labs where a wide variety of matrices need to be digested. The SK-15 perfectly matches the food lab's needs to determine trace elements, thanks to its high temperature (300°C) and pressure (100 bar capabilities) and its ability to digest large sample amounts.

The 15-position rotor is controlled by a direct temperature sensor that continuously measures the internal temperature throughout the run. 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.

A wide selection of high purity quartz and TFM inserts are available for the SK-15 rotor to accommodate smaller sample amounts or minimize the dilution factor of the analytical solution.



Figure 2. The SK-15 High Pressure Rotor

## MAXI-44 High Throughput Rotor

The MAXI-44 rotor can digest a variety of food samples, greatly improving lab throughput. It is controlled by contact-less sensors that directly control the temperature and pressure of each vessel, assuring maximum safety and digestion quality.

The SK-15 and MAXI-44 rotors have been used to digest both reference materials using different sample amounts. The results are shown in Table 1.





Figure 3. The MAXI-44 High Throughput Rotor

## ICP-OES Results

**Table 1. Digestion of NIST 1577c and Bovine Liver in the SK-15 and Maxi-44 Rotor**

Sample Name	SK-15 Procedure	Maxi-44 Procedure
NIST 1577c	1 g	0.5 g
Bovine Liver	10 mL of HNO <sub>3</sub> 65%	10 mL of HNO <sub>3</sub> 65%
Tort-3 (Lobster Hepatopancreas)	1 g	0.5 g
	10 mL of HNO <sub>3</sub> 65%	10 mL of HNO <sub>3</sub> 65%

**Table 2. TORT-3 Lobster Hepatopancreas (Results are expressed in µg/Kg)**

	Cert Value	Uncert	SK-15	Recovery	MAXi-44	Recovery
As	59.5	3.8	68.6	115.3%	67.4	113.3%
Cd	42.3	1.8	38.5	91.0%	38.3	90.5%
Cr	1.95	0.24	1.73	88.7%	1.72	88.2%
Cu	49	22	442	88.9%	439	88.3%
Hg	0.292	0.022	0.286	97.9%	0.275	94.2%
Mo	3.44	0.12	3.12	90.7%	3.23	93.9%
Se	10.9	1	9.98	91.6%	9.89	90.7%
Zn	136	6	123	90.4%	121	89.0%

**Table 3. NIST 1577c Bovine Liver (Results are expressed in µg/Kg)**

	Cert Value	Uncert	SK-15	Recovery	MAXi-44 0.5 g	Recovery
As	19.6	1.40	<	*	<	*
Cd	97	1.40	91.5	94.36%	90.1	92.84%
Cr	53	14	<	*-	<	*-
Cu	275200	4600	241432.0	87.73%	243387.5	88.44%
Mo	3300	130	3664.0	111.03%	3815.5	115.62%
Pb	62.8	1	<	*-	<	*-

\*Result below detection limit of ICP-OES

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

## Conclusion

Milestone's ETHOS UP with SK-15 and MAXI-44 rotors offers multiple benefits for sample preparation. The Ethos UP is a great solution for food labs requiring high throughput and digestion of large samples. Due to higher sample capacity, the SK-15 rotor offers 30-90% higher productivity than any other high pressure rotor.

The data shown in this technical note demonstrates that the better digestion quality achieved at higher temperatures makes analysis by ICP-OES more accurate.

## About Milestone

With over 50 patents and more than 18,000 instruments installed in laboratories around the world, Milestone has been widely recognized as the global leader in metals prep technology for the past 26 years. Committed to providing safe, reliable and flexible platforms to enhance your lab's productivity, customers worldwide look to Milestone for their metals digestion, organic extractions, mercury analysis and clean chemistry processing needs.