

APPROACHING MICROWAVE DIGESTION FOR AGRICULTURE SAMPLES WITH ETHOS LEAN



Nutrient composition analysis is an important parameter to evaluate for proper cultivation. Closed-vessel microwave digestion has proven to be an effective technique for sample preparation as it is fast, ensure complete digestion in a cleaner environment, and provides full recovery of all elements including volatiles. The ETHOS LEAN is the right starting point for whom approaching microwave digestion of agriculture matrices.

| INTRODUCTION

Nutrients composition is a fundamental parameter for the growth of plants. However, too much of the wrong nutrient can have adverse effects such as burning the roots. Therefore the analysis of soils, plant materials, fruits and vegetables is used to evaluate the nutrients compositions. The sample preparation of these samples is a fundamental step to ensure reliable analysis. Traditional sample preparation techniques include hot block digestion, teflon bomb and ashing; they pose different challenges. All these techniques suffer from long run times, airborne contamination, poor digestion quality, and poor recovery of volatile compounds. Closed vessel microwave digestion has proven to be the most efficient approach to elemental analysis and to be a perfect complement to ICP-OES or other atomic spectroscopy instruments. In this industry report, Milestone ETHOS LEAN compact microwave

digestion was tested to prove its efficiency in sample preparation for agriculture samples, followed by ICP-OES analysis on several elements. The ETHOS LEAN incorporates all the benefits of closed vessel microwave digestion while making sample preparation fast, easy, effective, and of high quality.

| EXPERIMENTAL

In this industry report, ETHOS LEAN, equipped with easyTEMP temperature sensor and MAXI-14, was used to digest agriculture samples.

INSTRUMENT

- ETHOS LEAN
- easyTEMP
- MAXI-14
- ICP-OES



Figure 1 – ETHOS LEAN with MAXI-14 rotor and easyTEMP sensor

ANALYTICAL PROCEDURE

Sample is directly weighted into the MAXI-14 PTFE-TFM vessel then acid is added. After closing the PTFE-TFM vessel, the MAXI-14 rotor is loaded into the ETHOS LEAN and the run starts.

SAMPLE	SAMPLE AMOUNT	ACID MIXTURE
San Joaquin soil (NIST 2709a)*	0.5 g	9 mL of HNO ₃ (65%), 3 mL of HCl (37%)
Tomato leaves (NIST 1573a)	0.3 g	5 mL of HNO ₃ (65%) + 1 mL of H ₂ O ₂ (30%)
Cabbage (IAEA-359)	0.3 g	5 mL of HNO ₃ (65%) + 1 mL of H ₂ O ₂ (30%)

* EPA 3051A was applied

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

STEP	TIME	T2
1	00:20:00	200°C
2	00:15:00	200°C

Table 2 – ETHOS LEAN microwave program used for digestion of samples

- Final dilution: 50 mL with deionized water

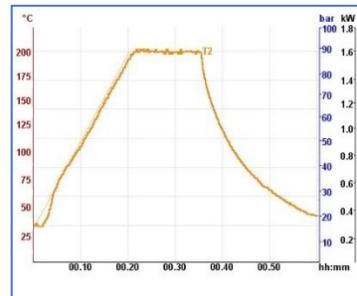


Figure 2 – ETHOS LEAN, MAXI-14 Microwave Run Report

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 Milestone's ETHOS LEAN equipped with MAXI-14 rotor was evaluated through a recovery study on San Joaquin soil (NIST 2709a), tomato leaves (NIST 1573a) and cabbage (IAEA 359) samples. The samples were digested with Milestone's ETHOS LEAN and subsequently analyzed via ICP-OES.

	Certified value	Recovery % (n=3)	RSD (%)
Al	7.37 ± 0.16 %	96.1	1.9
Ba	979 ± 28 mg/Kg	89.3	2.1
Ca	1.91 ± 0.09 %	92.1	1.48
Cd	0.371 ± 0.002 mg/Kg	<LOQ	-
Co	12.8 ± 0.2 mg/Kg	96.3	2.5
Cr	130 ± 9 mg/Kg	93.4	2.1
Fe	3.36 ± 0.07 %	86.4	1.6
K	2.11 ± 0.06 %	93.2	2.0
Mg	1.46 ± 0.02 %	99.2	2.1
Mn	529 ± 18 mg/Kg	100.1	1.6
Na	1.22 ± 0.03 %	94.2	1.8
P	0.0688 ± 0.0013 %	109.1	2.7
Pb	17.3 ± 0.1 mg/kg	95.3	1.2
Sb	1.55 ± 0.06 mg/Kg	<LOQ	-

INDUSTRY REPORT

ETHOS LEAN | AGRICULTURE



Si	30.3 ± 0.4 %	97.2	2.2
Sr	239 ± 6 mg/Kg	92.3	2.5
Ti	0.336 ± 0.007 %	93.2	2.1
V	110 ± 11 mg/Kg	103.2	1.8
Zr	195 ± 46 mg/Kg	96.2	2.7

Table 3 - Data of the recovery study on San Joaquin soil (NIST 2709a) sample

	Certified value	Recovery % (n=3)	RSD (%)
Al	598.4 ± 7.1 mg/Kg	95.6	1.3
As	0.1126 ± 0.0032 mg/Kg	<LOQ	-
Ca	50450 ± 550 mg/Kg	95.4	1.9
Cd	1.517 ± 0.027 mg/Kg	98.3	2.4
Co	0.5773 ± 0.0071 mg/Kg	<LOQ	-
Cr	1.988 ± 0.034 mg/Kg	93.5	2.2
Cu	4.70 ± 0.14 mg/Kg	98.0	2.8
Fe	367.5 ± 4.3 mg/Kg	95.4	2.7
Hg	0.0341 ± 0.0015 mg/Kg	93.5	2.5
K	26760 ± 480 mg/kg	98.5	2.2
Mn	246.3 ± 7.1 mg/Kg	102.1	1.9
Na	136.1 ± 3.7 mg/Kg	98.2	1.4
Ni	1.582 ± 0.041 mg/Kg	94.6	1.8
P	2161 ± 28 mg/Kg	92.8	1.9
Rb	14.83 ± 0.31 mg/Kg	98.3	2.4
Sb	0.0619 ± 0.0032 mg/kg	<LOQ	-
Se	0.0543 ± 0.0020 mg/Kg	<LOQ	-
V	0.835 ± 0.034 mg/Kg	<LOQ	-
Zn	30.94 ± 0.55 mg/Kg	96.1	2.3

Table 4 - Data of the recovery study on tomato leaves (NIST 1573A) sample

	Certified value	Recovery % (n=3)	RSD (%)
As	0.10 ± 0.004 mg/Kg	<LOQ	-
Ba	11.0 ± 0.5 mg/Kg	94.5	2.4
Ca	18500 ± 510 mg/Kg	93.4	2.2
Cd	0.12 ± 0.005 mg/Kg	<LOQ	-
Cr	1.30 ± 0.06 mg/Kg	91.4	1.9
Cu	5.67 ± 0.18 mg/Kg	95.1	1.1
Fe	148 ± 3.9 mg/Kg	95.2	1.6

Hg	0.013 ± 0.002 mg/Kg	96.2	1.7
K	32500 ± 690 mg/Kg	94.4	2.6
Mg	2160 ± 50 mg/Kg	93.2	2.7
Mn	31.9 ± 0.6 mg/Kg	97.2	1.9
Na	580 ± 21 mg/Kg	99.4	1.2
Ni	1.05 ± 0.05 mg/kg	99.3	2.4
Se	0.12 ± 0.011 mg/Kg	<LOQ	-
Sr	49.2 ± 1.4 mg/Kg	92.1	2.9
Zn	38.6 ± 0.7 mg/Kg	99.9	1.1

Table 5 - Data of the recovery study on cabbage (IAEA-359) sample

The tables 3-5 collects the analytical results, shows good recoveries of all elements and RSDs below 3%. This demonstrates the robustness and reproducibility of digestion process with the ETHOS LEAN equipped with MAXI-14 and easyTEMP technologies.

CONCLUSION

The data shown in this industry report demonstrate full recovery of all the target elements, according to the reference material certificates.

All the samples were successfully digested with MAXI-14 making ETHOS LEAN a perfect fit for elemental analysis of agriculture samples. The performance of the ETHOS LEAN provides high level of reproducibility and great ease of use.

ABOUT MILESTONE

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