Brazilian Journal of Analytical Chemistry
2025, Volume 12, Issue 49, 178-184

SPONSOR REPORT

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New Performance for High Volume Agriculture Laboratories

Enabling high throughput in elemental analysis of several agriculture matrices using Milestone's ETHOS UP with MAXI 24 HP

This report was extracted from the Milestone Industry Report Ethos UP – Maxi-24 HP | Agriculture

INTRODUCTION

Fertilizer is a fundamental component for the growing of plants. However, too much of the wrong nutrient can have adverse effects such as burning the roots. Characterizing the fertilizer content will indicate the formula's macronutrient content, as well as other nutrients such as calcium, magnesium, potassium, etc. Analysis on a fertilizer product gives the ratio of each macronutrient, which must be correct to ensure optimal efficacy. ICP analysis can provide a lot of information on fertilizer composition but the choice of the correct sample preparation technique is fundamental. Traditional sample preparation techniques include hot block digestion, closed vessel microwave digestion and ashing; each of them posing 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. Milestone's ETHOS UP equipped with the MAXI 24 High Performance (HP) rotor incorporates all of the benefits of closed vessel microwave digestion while making sample preparation fast, easy, effective, and of the highest quality. This innovative solution perfectly integrates with the powerful ETHOS UP, matching both performance and throughput requirements of agricultural elemental analysis.

EXPERIMENTAL

In this industry report, a recovery study on certified reference materials has been performed to prove the efficacy of the ETHOS UP in sample preparation for 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.

Figure 2. MAXI-24 HP Rotor.

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.

Procedure

Table 1 reports the conditions used to prepare the sample.

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

SAMPLE	SAMPLE AMOUNT	ACID MIXTURE
Multi-nutrient	0.2 g	8 mL of HNO_3 (65%), 0.5 mL of H_2SO_4 (96%), 2 mL of HF (48%)
Marine sediment (IAEA-457)*	0.5 g	9 mL of HNO_3 (65%), 3 mL of HCl (37%)
San Joaquin soil (NIST 2709a)*	0.5 g	9 mL of HNO_3 (65%), 3 mL of HCl (37%)
Tomato leaves (NIST 1573a)	0.5 g	5 mL of HNO_3 (65%) + 1 mL of H_2O_2 (30%)
Cabbage (IAEA-359)	0.5 g	5 mL of HNO_3 (65%) + 1 mL of H_2O_2 (30%)

* EPA 3051A was applied

All samples were weighted into the MAXI–24 HP vessel, approximately 0.5 g (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.

Table 2. Microwave program

STEP	TIME	T2	POWER
1	00:10:00	160 °C	1500 W
2	00:15:00	210 °C	1800 W
3	00:10:00	210 °C	1800 W

After microwave digestion, the samples were diluted to 50 mL with deionized water and analyzed by 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 ETHOS UP equipped with MAXI-24 HP rotor and easyTEMP technology was evaluated through a recovery study on multi-nutrient fertilizer (NIST SRM695), marine sediment (IAEA 457), San Joaquin soil (NIST 2709a), tomato leaves (NIST 1573a) and cabbage (IAEA 359) samples. The samples were digested with Milestone's ETHOS UP and subsequently analyzed via ICP-OES.

Table 3. Data of the recovery study on multi-nutrient fertilizer (NIST SRM695) sample

	Certified value	Recovery % (n=3)	RSD (%)
Al	0.61 ± 0.03%	89.3	2.3
As	200 ± 5 mg/Kg	96.7	2.6
Ca	2.26 ± 0.04%	103.5	2.8
Cd	16.9 ± 0.2 mg/Kg	93.5	1.7
Co	65.3 ± 2.4 mg/Kg	94.1	2.3
Cr	244 ± 6 mg/Kg	88.9	2.6
Cu	1225 ± 9 mg/Kg	91.7	1.7
Fe	3.99 ± 0.08%	89.7	2.3
Hg	1955 ± 0.036 mg/Kg	95.6	2.6
K	11.65 ± 0.13%	92.6	1.9
Mg	1.79 ± 0.05%	105.9	2.7
Mn	0.305 ± 0.005%	101.5	2.4
Mo	20.0 ± 0.3 mg/Kg	93.3	1.4
Na	0.405 ± 0.007%	95.6	2.2
Ni	135 ± 2 mg/Kg	90.0	2.3
Pb	273 ± 17 mg/Kg	102.3	2.6
V	122 ± 3 mg/Kg	99.6	1.0
Zn	0.325 ± 0.005 mg/Kg	101.3	2.1

Table 4. Data of the recovery study on marine sediment (IAEA-457) sample

	Certified value	Recovery % (n=3)	RSD (%)
Ag	1.93 ± 0.38 mg/Kg	94.1	1.4
Al	82660 ± 3430 mg/Kg	96.3	1.3
As	10.2 ± 1.0 mg/Kg	109.4	2.5
Cd	1.09 ± 0.08 mg/Kg	102.9	1.9
Co	14.7 ± 1.0 mg/Kg	90.0	2.1

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Table 4. Data of the recovery study on marine sediment (IAEA-457) sample (continuation)

	Certified value	Recovery % (n=3)	RSD (%)
Cr	144 ± 8 mg/Kg	89.9	2.0
Cu	365 ± 19 mg/Kg	91.3	1.4
Fe	41450 ± 2240 mg/Kg	91.6	0.9
Hg	0.143 ± 0.012 mg/Kg	93.2	1.3
Li	64.2 ± 5.5 mg/Kg	94.7	1.8
Mn	427 ± 30 mg/Kg	93.1	2.8
Ni	53.1 ± 2.7 mg/Kg	93.0	1.4
Pb	105 ± 7 mg/kg	91.4	1.2
Sn	27.40 ± 0.75 mg/Kg	93.7	2.1
Sr	137 ± 10 mg/Kg	94.1	1.1
V	87.4 ± 8.1 mg/Kg	101.3	2.0
Zn	425 ± 25.8 mg/Kg	96.8	2.5

Table 5. Data of the recovery study on San Joaquin soil (NIST 2709a) sample

	Certified value	Recovery % (n=3)	RSD (%)
Al	7.37 ± 0.16%	93.4	2.3
Ba	979 ± 28 mg/Kg	91.6	2.6
Ca	1.91 ± 0.09%	89.9	2.8
Cd	0.371 ± 0.002 mg/Kg	<LOQ	—
Co	12.8 ± 0.2 mg/Kg	94.2	2.7
Cr	130 ± 9 mg/Kg	90.7	2.4
Fe	3.36 ± 0.07%	83.3	1.4
K	2.11 ± 0.06%	92.8	2.2
Mg	1.46 ± 0.02%	102.8	2.3
Mn	529 ± 18 mg/Kg	98.1	2.6
Na	1.22 ± 0.03 %	93.6	2.8
P	0.0688 ± 0.0013%	113.1	1.7
Pb	17.3 ± 0.1 mg/kg	92.4	1.4
Sb	1.55 ± 0.06 mg/Kg	<LOQ	1.9
Si	30.3 ± 0.4%	96.3	1.2
Sr	239 ± 6 mg/Kg	95.7	2.3

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Table 5. Data of the recovery study on San Joaquin soil (NIST 2709a) sample (continuation)

	Certified value	Recovery % (n=3)	RSD (%)
Ti	0.336 ± 0.007%	91.3	2.6
V	110 ± 11 mg/Kg	102.0	2.8
Zr	195 ± 46 mg/Kg	94.4	1.7

Table 6. Data of the recovery study on tomato leaves (NIST 1573A) sample

	Certified value	Recovery % (n=3)	RSD (%)
Al	598.4 ± 7.1 mg/Kg	94.1	2.3
As	0.1126 ± 0.0032 mg/Kg	<LOQ	—
Ca	50450 ± 550 mg/Kg	96.7	1.0
Cd	1.517 ± 0.027 mg/Kg	92.1	2.1
Co	0.5773 ± 0.0071 mg/Kg	<LOQ	—
Cr	1.988 ± 0.034 mg/Kg	90.9	2.6
Cu	4.70 ± 0.14 mg/Kg	96.0	2.8
Fe	367.5 ± 4.3 mg/Kg	96.8	1.7
Hg	0.0341 ± 0.0015 mg/Kg	92.3	2.3
K	26760 ± 480 mg/kg	99.7	2.6
Mn	246.3 ± 7.1 mg/Kg	101.0	1.7
Na	136.1 ± 3.7 mg/Kg	97.1	2.3
Ni	1.582 ± 0.041 mg/Kg	93.5	2.6
P	2161 ± 28 mg/Kg	90.6	2.2
Rb	14.83 ± 0.31 mg/K.g	90.2	1.4
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	94.5	2.4

Table 7. Data of the recovery study on cabbage (IAEA-359) sample

	Certified value	Recovery % (n=3)	RSD (%)
As	0.10 ± 0.004 mg/Kg	<LOQ	—
Ba	11.0 ± 0.5 mg/Kg	91.6	1.2
Ca	18500 ± 510 mg/Kg	92.5	2.6

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Table 7. Data of the recovery study on cabbage (IAEA-359) sample (continuation)

	Certified value	Recovery % (n=3)	RSD (%)
Cd	0.12 ± 0.005 mg/Kg	<LOQ	—
Cr	1.30 ± 0.06 mg/Kg	90.3	2.0
Cu	5.67 ± 0.18 mg/Kg	94.8	2.1
Fe	148 ± 3.9 mg/Kg	95.2	1.6
Hg	0.013 ± 0.002 mg/Kg	94.1	0.7
K	32500 ± 690 mg/Kg	96.3	1.6
Mg	2160 ± 50 mg/Kg	91.4	1.7
Mn	31.9 ± 0.6 mg/Kg	98.9	2.9
Na	580 ± 21 mg/Kg	103.4	1.1
Ni	1.05 ± 0.05 mg/kg	95.5	1.4
Se	0.12 ± 0.011 mg/Kg	<LOQ	—
Sr	49.2 ± 1.4 mg/Kg	93.1	1.9
Zn	38.6 ± 0.7 mg/Kg	91.9	2.1

The analytical results were shown in Tables 3-7 with good recoveries of all elements and RSDs below 3%. This demonstrates the robustness and reproducibility of the digestion process with the ETHOS UP – MAXI-24 HP.

Figure 3 shows the temperature profile of the digestion as well as the multiple temperature visualization and recording for all the samples digest in the run.

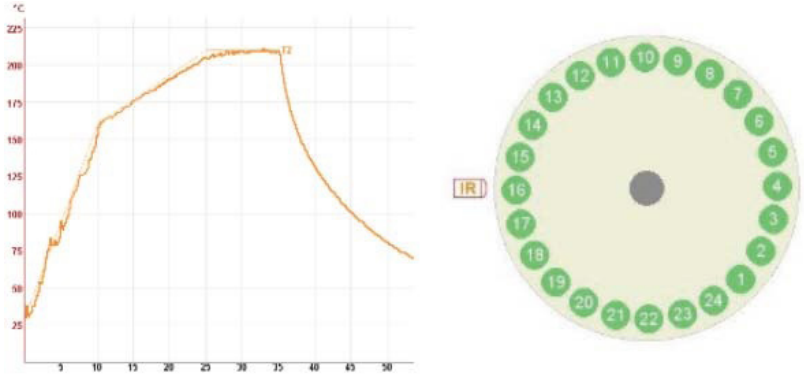

Figure 3. MAXI-24 HP Microwave Run Report and Multiple temperature traceability.

CONCLUSION

The data shown in this industry report demonstrates full recovery of the elements reported in the certificates of the reference material. Highly reactive samples such as fertilizer has been completely digested even in large sample amounts. The digestion process has been accurately controlled by the easyTEMP sensor,

ensuring same digestion quality and reliable results. 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.

Further reading here

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