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.

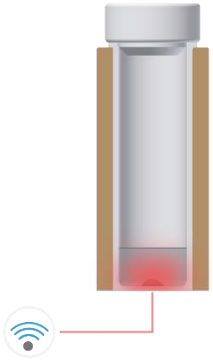


Figure 2 – easyTEMP contactless direct temperature sensor.

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.

Figure 4 – MAXI-44 easyTEMP High Throughput Rotor

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

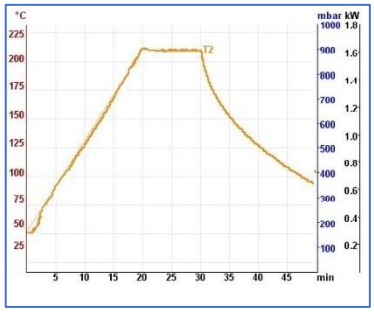
Table 1 - Sample amount and acid mixture used for the microwave digestion run

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



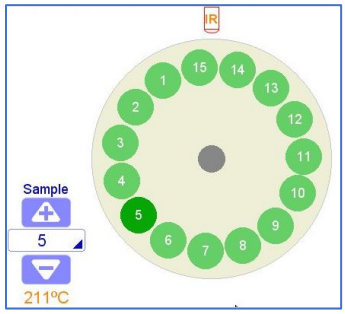


Figure 6 – SK-15 Microwave Run Report and Multiple temperature traceability

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

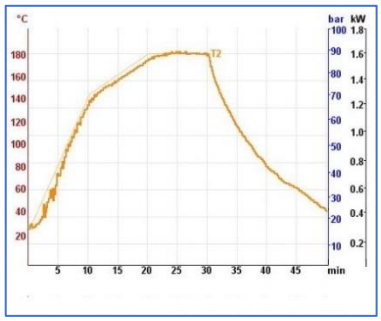


Figure 7 – MAXI-44 Microwave Run Report and Multiple temperature traceability

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.

		SK-15 eT		MAXI-44 eT	
	Certified value (mg/Kg)	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.

		SK-15 eT		MAXI-44 eT	
	Certified value (mg/Kg)	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.

MILESTONE Srl - Via Fatebenefratelli, 1/5 - 24010 Sorisole (BG) - Italy
Tel: +39 035 573857 - Fax: +39 035 575498
www.milestonesrl.com - email: analytical@milestonesrl.com