



Microwave Sample Prep for heavy metals determination in cannabis plants, concentrates and edibles for medical applications

Metals testing of various cannabis samples with Milestone's Ethos UP high pressure microwave digestion system.

Introduction

The medical cannabis industry is currently one of the fastest growing industries in the United States and is becoming more prevalent worldwide. Although systems for growing, production, and sale of medical cannabis and cannabis-related products are well established, regulation and enforcement of quality and safety testing have lagged behind. As the industry matures, many challenges have been encountered. One of those challenges is how to ensure safe products that are free from potential contaminants such as heavy metals.

Like all plants, cannabis absorbs metals from its environment, a result of normal plant metabolism. Some of these metals are naturally occurring and leach into groundwater. Others precipitate in rainwater or may be introduced into the plant's environment as constituents of fertilizers, pesticides, herbicides, and fungicides used to increase crop yield. Regardless of their prevalence, when metabolized, metals are absorbed and transported through the plant roots and into plant tissue. Cannabis is so effective at absorbing metals from its environment that it has been referred to as a hyperaccumulator of trace metals, including lead, cadmium, copper, chromium, arsenic, mercury and cobalt. This leads to concern that these elements may occur in high concentrations in cannabis plants.

State governments and private laboratories are focusing on product safety testing with special emphasis on As, Cd, Hg and Pb, as they are extremely hazardous to human health, even at low levels.

The combination of ICP-MS with Milestone's Ethos UP equipped with SK-15 High-Pressure Rotor enables digestion and testing of heavy metals in all medical cannabis related products from the plant material to edibles and concentrates.

Milestone's Ethos UP microwave digestion system incorporates all the benefits of closed vessel microwave digestion, making sample preparation fast, easy and efficient.

The ETHOS UP with SK-15 helps speed up the sample preparation process, improves recovery of all elements (including volatiles) and reduces possible sources of contamination.



Experimental

Instrument

The ETHOS UP meets many requirements of the new medical cannabis regulations. 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 element and routine determinations in many fields of analysis. It is constructed of 18/8 stainless steel, features a built-in camera and can accommodate both high-pressure and high-throughput rotors.

The Ethos UP includes 300 built-in digestion methods, which virtually eliminate method development. Additionally, the Ethos 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 perfectly matches the needs of a cannabis testing lab to determine trace elements, thanks to its capability for digesting large sample amounts at high temperature (300 °C) and pressure (100 bar).

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.



Analytical Procedure

Sample	Ethos UP – SK15 8 mL HNO ₃ 65%, 1 mL HCl 37%, 1 mL H ₂ O ²
Cannabis plant material	1 g
CBD oil	0.5 g
Cannabis vape cartridge	0.5 g
Cannabis salve	0.5 g
Cannabis flavored cookies	1 g
Cannabis flavored gummy bear	1 g

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



Microwave program used to digest samples:

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

Final dilution: 50 mL with deionized water





Quantification	
ICP-MS Instrumental Parameters:	
RF Power	1600 W
Sampling depth	10 min
Carrier gas	0.8 L/min
Sweeps/Reading	20
Readings/Replicate	1
Number of replicates	3
Integration time	1000 ms
Dwell time per AMU	50 ms
Mode	KED
Scan mode	Peak hopping
Cell Gas A	0.6
RP a	0
RP q	0.25

Results and Discussion

The performance of the Milestone's Ethos UP equipped with SK-15 rotor was evaluated through a recovery study on samples of interest for the medical cannabis industry, from plant material to edibles and concentrates. As cannabis-containing representatives of the above categories, plant material, CBD oil, a vape cartridge, a salve, cookies and gummy bears were digested with Milestone's Ethos UP equipped with SK-15 High-Pressure Rotor and subsequently analyzed via ICP-MS.

All samples were fortified with a spike solution containing 20 ppb of As, Cd, Pb, Ag, Ba, Co, Cr, Cu, Mn, Ni, Se, V, Zn and 10 ppb of Hg. The analytical results are shown in Table 2 with recoveries of all elements between 80-120% and RSDs below 3%. This demonstrates the robustness and reproducibility of microwave digestion using the Ethos UP equipped with SK-15 technology.

Table 2. Data of the recovery study.

		As	Cd	Hg	Pb	Ag	Ba	Co	Cr	Cu	Mn	Ni	Se	V	Zn
Cannabis plant material	Recovery (n=3) (%)	91.8	92.3	101.4	98.7	95.4	93.5	87.6	92.6	-*	-*	94.2	94.7	105.3	-*
	RSD (%)	2.3	0.7	1.3	1.5	2.6	1.4	0.4	0.6	-*	-*	0.3	2.9	0.6	-*
CBD oil	Recovery (n=3) (%)	91.3	87.3	105.8	97.3	82.6	89.7	102.5	103.8	92.3	92.6	84.5	92.6	107.1	96.8
	RSD (%)	2.7	2.8	1.4	2.3	2.7	2.9	1.6	2.2	2.6	2.5	2.7	2.9	1.5	2.0
Cannabis vape cartridge	Recovery (n=3) (%)	94.5	92.8	99.3	102.5	97.3	88.7	107.3	96.8	102.3	95.3	98.2	106.3	90.8	91.3
	RSD (%)	1.5	2.2	1.2	1.5	1.1	1.3	1.4	1.3	1.1	1.7	1.7	2.1	1.2	1.9
Cannabis salve	Recovery (n=3) (%)	90.7	95.8	102.3	89.2	89.3	90.2	89.4	99.8	94.8	98.6	99.1	91.3	92.9	91.0
	RSD (%)	2.1	1.0	1.5	2.5	2.0	1.8	1.6	2.2	1.2	1.8	1.4	1.5	1.5	1.7
Cannabis flavored cookies	Recovery (n=3) (%)	92.2	96.2	95.6	93.5	90.7	94.4	90.7	93.2	98.8	97.5	91.9	93.2	95.3	-*
	RSD (%)	2.1	1.3	1.4	1.0	1.3	1.1	2.3	2.1	0.8	1.8	1.7	2.3	1.8	-*
Cannabis flavored gummy bear	Recovery (n=3) (%)	97.8	96.7	98.2	96.7	96.8	94.6	91.8	93.1	91.3	90.2	92.7	88.3	96.2	93.1
	RSD (%)	2.0	0.3	0.4	1.8	2.3	0.8	0.7	0.6	0.3	1.0	1.5	2.3	0.3	1.4

* The ratio between spiked/unspiked concentration was too low.





Conclusion

The data shown in this technical note demonstrates full recovery of the most common elements occurring in cannabis plants and related products. Highly reactive samples such as gummy bears, cookies and CBD oil have been completely digested even in large sample amounts, ensuring reliable analysis. In addition to full analyte recovery, microwave digestion using Milestone's Ethos UP with SK-15 rotor provides the highest level of reproducibility, even for volatile elements such as As and Hg.

Milestone's ETHOS UP equipped with SK-15 rotor offers multiple benefits for sample preparation for trace metals analysis and is the most suitable microwave digestion system for cannabis testing labs requiring digestion of all cannabis-related products.

This new era of acceptance and legalization has opened new opportunities for labs. Standardization of these methods for the industry will give regulators the resources they need to include sensible requirements for regulation and legislation that are being crafted to monitor and control the use of medical cannabis within the United States.

About Milestone

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