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SIMULTANEOUS MIXED-BATCH SAMPLE PREPARATION FOR METALS TESTING OF CANNABIS AND CANNABIS PRODUCTS

Trace metals analysis via Single Reaction Chamber (SRC) Technology to ensure high productivity and ease of use

INTRODUCTION

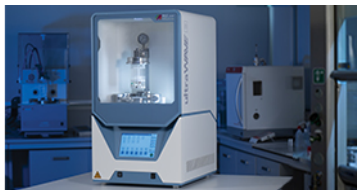
The medical cannabis industry is one of the fastest growing industries in the United States and becoming more prevalent worldwide. Although systems for growing, production, and sale of cannabis and related products are well established, regulation and enforcement of quality and safety testing have lagged. As the industry matures, many challenges are being faced, such as ensuring consumer safety and providing products free from potential contaminants such as heavy metals.

Like all plants, cannabis uptakes 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 and Milestone's ultraWAVE 3 with Single Reaction Chamber (SRC) technology allows cannabis analytical testing labs to analyze a broad variety of matrices on which metals determination could be very different, toxic elements in some cases and nutrients in others.

This industry report describes how a variety of samples from the cannabis industry were digested simultaneously using the Milestone's ultraWAVE 3,



and this can be replicated in previous ultraWAVE generation, without sample-to-sample cross contamination.
The reliability of the system was evaluated through a recovery study on samples of interest for the cannabis industry.

EXPERIMENTAL

INSTRUMENT

- Milestone's ultraWAVE 3
- 20-positions rack with 15 mL glass vials
- Analytical balance
- ICP-MS

Figure 1 – Milestone's ultraWAVE 3

Developed and patented by Milestone, the ultraWAVE 3 represents another significant step forward for SRC technology and embrace Milestone 20+ years of experience. The stainless-steel reactor with a high-purity PTFE-TFM liner and cover, enables to achieve, higher pressures and temperatures regardless the sample type and acid mixture. The digestion process is continuosly controlled by easyTEMP, an advance contactless sensor that measure the temperature directly of the reaction chamber. The simplified rack construction eliminates the time need to assemble and disassemble the vessels. Just as important, dissimilar samples can be processed simultaneously using disposable glass, quartz or PTFE-TFM vials, thus saving time and money. The ultraWAVE 3 addressed all the sample preparation challenges related to performance, time, workflow, and cost of ownership.

SAMPLES

As cannabis-containing representatives, plant material, CBD oil, vape cartridge, salve, cookies and gummy bears were fortified with spike solutions containing 20 ppb of As, Cd, Pb, Ag, Ba, Co, Cr, Cu, Mn, Ni, Se, V, Zn and 10 ppb of Hg and then simultaneously digested as a mixed batch. A soil reference material (SRM 2711a) was also included in this study as a quality control sample.

SAMPLE PREPARATION

An amount of 0.5 g of each cannabis-containing representatives were weighed inside a 15 mL glass vial and 4 mL of 65% HNO_3 + 1 mL of 37% HCl mixed with 500 µL of spike solution were added to the glass vial containing the sample.

The rack with the 20 glass vials was positioned inside the SRC TFM liner inside the reaction chamber, which was previously filled with 120 mL of H_2O and 5 mL of HNO_3. The SRC system was pressurized up to 40 bar with Nitrogen gas to close the glass vials. The following microwave heating program was applied:

Step	Time (min)	Power (W)	T1 (°C)	T2 (°C)	P (bar)
1	10	1000	80	60	90
2	10	1000	180	60	90
3	10	1500	240	60	130
4	25	1500	240	60	130

Table 1 - Microwave Program

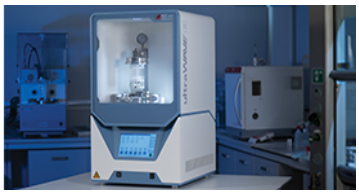


Figure 2 – Microwave run profile

After cooling the sample digests were diluted with deionized water up to 50 mL and then analyzed by ICP-MS.

ultraWAVE 3 – SRC Technology

SAMPLE	SAMPLE AMOUNT	ACID MIXTURE
Cannabis flower	0.5 g	4 mL of HNO_3 65%, 1 mL of HCl 37%
CBD oil	0.5 g	
Cannabis vape cartridge	0.5 g	
Cannabis salve	0.5 g	
Cannabis flavored cookies	0.5 g	
Cannabis flavored gummy bear	0.5 g	
Soil (SRM 2711a)	0.5 g	

Table 2 – Sample amount and acid mixture used for the microwave digestion run

QUANTIFICATION

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

RESULTS AND DISCUSSION

The analytical results are shown in Tables 3 and 4 with good recoveries of all analytes and RSDs below 3%. This demonstrates the robustness and reproducibility of microwave digestion using the ultraWAVE 3 with SRC technology.

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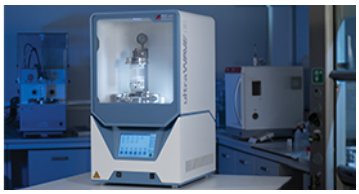

		As	Cd	Hg	Pb	Ag	Ba	Co	Cr	Cu	Mn	Ni	Se	V	Zn
Cannabis plant material	Recovery (n=3) (%)	91.7	93.0	98.7	88.3	88.6	93.0	96.5	93.7	-*	-*	91.3	95	94.5	-*
	RSD (%)	1.9	2.1	2.1	2.6	1.5	1.3	2.3	2.4	-*	-*	91.3	1.6	2.1	-*
CBD oil	Recovery (n=3) (%)	95.8	98.5	97.6	89.7	92.7	99.7	93.0	88.2	91.0	91.2	96.2	102.7	90.2	95.8
	RSD (%)	1.8	2.3	1.1	2.2	2.2	2.5	2.2	2.0	2.8	2.4	1.4	0.7	1.9	2.7
Cannabis vape cartridge	Recovery (n=3) (%)	90.8	87.3	91.8	92.0	89.2	91.2	93.2	87.7	94.4	94.8	92.2	96.8	89.7	95.9
	RSD (%)	1.1	2.0	1.2	1.5	2.0	1.9	2.0	1.8	2.1	1.6	1.5	2.6	2.5	1.4
Cannabis salve	Recovery (n=3) (%)	95.8	91.5	94.3	95.3	87.0	90.0	92.2	86.8	92.9	90.4	94.8	100.8	91.3	90.1
	RSD (%)	0.3	1.1	1.4	2.2	1.5	1.0	2.0	1.1	2.4	2.5	2.4	1.8	0.7	0.3
Cannabis flavored cookies	Recovery (n=3) (%)	92.8	93.8	96.1	93.3	89.0	94.0	94.2	98.2	-*	-*	92.8	92.8	89.5	-*
	RSD (%)	2.8	0.7	1.3	1.4	2.4	1.2	1.8	1.0	-*	-*	2.4	2.6	0.6	-*
Cannabis flavored gummy bear	Recovery (n=3) (%)	90.2	89.5	94.1	91.8	90.7	91.5	95.5	91.3	96.4	95.8	90.5	91.2	89.5	92.2
	RSD (%)	2.1	2.0	1.0	2.2	2.4	2.6	2.3	1.4	2.3	2.8	1.5	0.8	1.0	1.1

Table 3- Data of the recovery study.

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

		As	Cd	Hg	Pb	Ag	Ba	Co	Cr	Cu	Mn	Ni	Se	V	Zn
Soil (SRM 2711a)*	Recovery (n=3) (%)	90.4*	94.1*	98.7*	93.3*	92.1*	42.0*	89.2*	35.6*	97.8*	89.4*	90.8*	98.9*	47.9*	92.1*
	RSD (%)	2.1	1.9	1.6	1.1	1.0	0.6	1.2	2.4	0.9	1.5	1.8	1.9	0.6	0.9

Table 4- Data of the recovery study

*The recoveries were calculated according to the total element content and represent the leachable fraction (please, refer to NIST Certificate of Analysis for SRM 2711a for further details).

CONCLUSIONS

The data shown in this technical note demonstrates full recovery of the most common elements occurring in cannabis plants[1]. In addition to full analyte recovery, microwave digestion using Milestone's SRC technology provides the highest level of reproducibility, even for volatile elements such as As and Hg. Due to its higher sample capacity, use of disposable vials and faster cool down time, sample processing throughput is 2 to 3 times higher than conventional closed vessel digestion systems. The ability to digest different sample types together and larger sample weights with minimal acid volume makes it the optimal technique to perform sample prep for all cannabis related products, from the plant material to edibles, concentrates and even soil samples.

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