



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 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.

Milestone's SRC microwave digestion is a revolutionary new approach, incorporating all the benefits of closed vessel microwave digestion while making sample preparation fast, easy and efficient. SRC technology operates at very high temperature and pressure (up to 300 °C and 199 bar, respectively), allowing for complete digestion of even the largest sample size (up to 3-5 g) and difficult-to-digest matrices. Samples can be weighed directly into disposable glass vials, eliminating the need for acid cleaning and vessel assembly. Furthermore, SRC eliminates the need to batch samples, as mixed sample types, masses and acid chemistries can be successfully processed in the same run. This technical note describes how a variety of samples from the cannabis industry were digested simultaneously without sample-to-sample cross contamination.

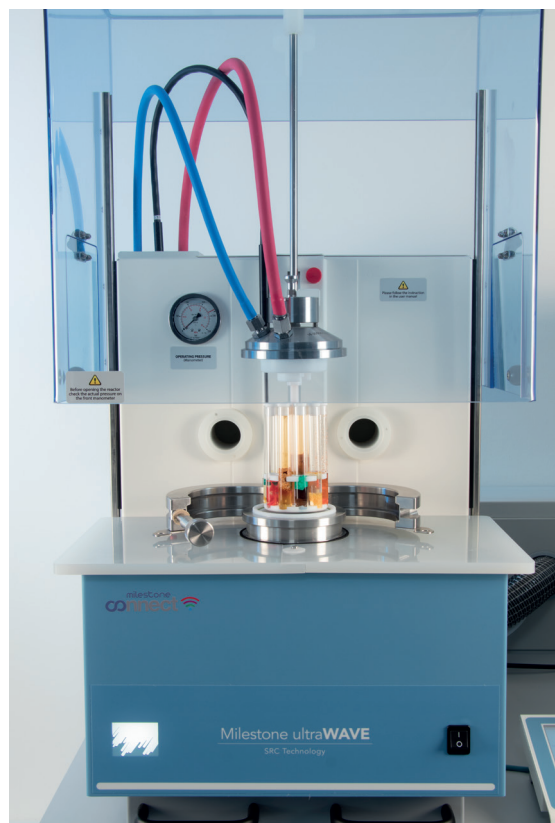
EXPERIMENTAL

INSTRUMENT

The ultraWAVE meets the requirements of the new cannabis regulations, thanks to its unique benefits, such as:

- Superior digestion quality

- High productivity
- Ease of use
- Superb safety and flexibility



Developed and patented by Milestone, the ultraWAVE with Single Reaction Chamber (SRC) technology utilizes high-performance stainless steel, allowing for higher pressures and temperatures to be reached and any type of acids to be used. Disposable vessels eliminate the need to assemble, disassemble or clean between processing. Just as important, dissimilar samples can be processed simultaneously using any mixture of disposable glass, quartz or TFM vials, thus saving time and money. The ultraWAVE is simply the fastest, easiest and most efficient digestion system ever made.

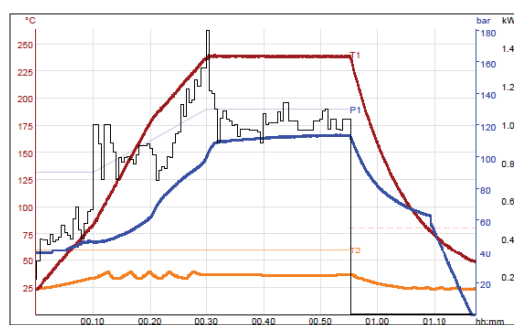


ANALYTICAL PROCEDURE

ultraWAVE — SRC Technology

SAMPLE	SAMPLE AMOUNT	ACID MIXTURE
Cannabis Flower	0.5 g	4 mL of HNO ₃ , 1 mL HCl
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 1 - Sample amount and acid mixture used for the microwave digestion run



*Samples were digested using this multi-step microwave program at 240 °C: dilution to final volume with 50 mL of DI Water

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 performance of the Milestone ultraWAVE was evaluated through a recovery study on samples of interest for the cannabis industry, from plant material to edibles, concentrates and soil. As cannabis-containing representatives of the above categories, plant material, CBD oil, a

vape cartridge, cannabis salve, cookies, gummy bears and even soil were digested as a mixed batch, simultaneously, with the ultraWAVE and subsequently analyzed via ICP-MS. Cannabis 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. A soil reference material (SRM 2711a) was also included in this study as a quality control sample. The analytical results are shown in Table 3 with good recoveries of all analytes and RSDs below 3%. This demonstrates the robustness and reproducibility of microwave digestion using the ultraWAVE with SRC technology.



		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).

CONCLUSION

The data shown in this technical note demonstrates full recovery of the most common elements occurring in cannabis plants. 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. 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.

