

Microwave Digestion and Trace Metals Analysis of Cannabis and Hemp Products



Abstract

In 1970, marijuana was designated a Schedule 1 drug, under the Controlled Substances Act, making it nearly impossible for laboratories to perform cannabis research. However, medicinal use of cannabis is now legal in Canada and 35 US states. With the passage of the Farm Bill in 2018, it is now federally legal to grow and process hemp in all 50 states. All of this interest in medical cannabis and Cannabidiol (CBD) has highlighted the need for good analysis methodology in this relatively young market. Cannabis analysis is still developing standardized protocols, requirements, and acceptable testing practices. Typical testing requirements for cannabis and its products include heavy metals analysis, pesticide residue, and the potency of active ingredients such as tetrahydrocannabinol (THC). The terpene content of cannabis is also important. Terpenes have been shown to have beneficial uses for treatment of conditions ranging from cancer and inflammation to anxiety and sleeplessness. It is believed that the combination of terpenes and cannabinoids in cannabis produces a synergistic effect with regards to medical benefits.

Introduction

Certain heavy metals can cause adverse effects on human health. Toxic heavy metals such as arsenic, cadmium, lead, and mercury are persistent once released into the environment and can accumulate in cannabis plants. Since hemp is a strain of cannabis that contains very little THC, it is susceptible to bioaccumulation of heavy metals like its THC-containing parents. Cannabis-based products such as foods, oils, tinctures, and salves should be tested for the presence of heavy metals to ensure patient safety and product quality.

Cannabis and CBD-infused products have grown in popularity in states that allow recreational use of cannabis and hemp. This application note details the digestion and analysis of various forms of hemp and cannabis products.

Materials and Methods

Instrumentation

A CEM MARS™ 6 microwave digestion system was used to digest the varied cannabis and hemp sample types. The MARSXpress™ Plus vessels use a vent and reseal design, which allows for acid vapors to be exhausted, while maintaining the sample integrity and elements within the vessel. The 110 mL vessel volume provides for a larger headspace, allowing a larger sample size for a more homogenous sample. The MARSXpress Plus vessel design uses only three pieces that are easily assembled and placed into a 24-position turntable, prior to placing into the MARS 6 system.

Sample Preparation

All samples required special preparation, based on the qualities of each sample, in order to obtain a homogeneous 0.5 g approximate sample weight. It is important to carefully deposit the oil, cream, and peanut butter, as to prevent the sample from sticking to the sides of the vessel. For peanut butter and cream, a long plastic spatula was used to deposit the sample on the bottom of the liner. For the oil, the sample was deposited to the bottom of the vessel with a plastic pipette,

then the inside of the liner was washed down with the acid to make sure all the sample reached the bottom. **Table 1** shows the 11 samples that were digested and the preparation method used, prior to placing them into the vessels.

Nine mL of HNO₃ and 1 mL HCl were added to each vessel and the samples were allowed to predigest for 15 minutes, prior to sealing and placing the vessel in the turntable. The turntable was then placed in the MARS 6 and the Cannabis One Touch™ Method was chosen on the touchscreen. The MARS 6 then counted the number of vessels and applied the precise power required to achieve the optimal digestion temperature of 210 °C. This temperature ensured the complete digestion of all samples, for the most accurate analysis.

Samples were prepared in two batches. One batch was spiked with a spike solution containing 5 ppb As, Hg, Cd, and Pb. The second batch was left unspiked. All samples were run in duplicate with blanks in each run. Upon successful digestion, all samples were diluted to 50 mL with deionized water for analysis.

Analysis

All samples were analyzed on a Shimadzu ICP-MS 2030 to determine spike recoveries and actual sample content of the “big four” heavy metals of As, Cd, Hg, and Pb. Instrument conditions are detailed in **Table 2**.

Results and Discussion

The MARS 6, with MARSXpress Plus vessels was able to successfully digest all samples in a mixed batch. All of the samples were completely digested, yielding clear and particulate-free solutions upon dilution with deionized water. The MARS 6 is an ideal digestion system for the cannabis industry because it is able to successfully digest batches of mixed materials, including foods, oils, tinctures, creams, and plant materials, in as little as 35 minutes.

The clear and particulate-free digestate solutions that were analyzed by ICP-MS showed excellent spike recovery as shown in **Table 3**.

After confirmation of method through spike recovery study, unspiked samples were analyzed for their total trace metals content, using the same technique. **Table 4** shows the trace metals content of unspiked samples. **Table 5** contains the California action levels for medical cannabis samples. It is worth noting that all of the unspiked samples contained far less than the maximum allowable limit for each element.

Conclusion

The MARS 6 with MARSXpress Plus vessels was able to digest a wide variety of cannabis and hemp samples in mixed batches producing digestate that was suitable for analysis. Analysis of all samples showed excellent spike recovery and gave great confidence in the actual levels of trace metals in the unspiked samples.

Table 1. Preparation of Hemp and Cannabis Samples

Sample	Preparation
Hemp Flower	Torn into small pieces
Hemp Oil	Deposited on bottom of vessel with plastic pipette
MCT Oil	Deposited on bottom of vessel with plastic pipette
Topical Cream	Placed on plastic spatula to deposit on bottom of vessel
Crude CBD Extract	Placed on plastic spatula to deposit on bottom of vessel
Beef Jerky	Torn into small pieces
Peanut Butter	Placed on plastic spatula to deposit on bottom of vessel
Ghee	Placed on plastic spatula to deposit on bottom of vessel
Granola Bar	Crushed
Gummy Snacks	Cut in half with ceramic scissors
Hard Candy	Crushed

Table 2. ICP-MS Conditions

Parameter	Setting
Radio Freq. Power	1.20 kW
Sampling Depth	5.0 mm
Plasma Gas	8.0 L/min
Auxiliary Gas	1.10 L/min
Carrier Gas	0.70 L/min
Mix Gas	0.00 L/min
Cell Gas	6.0 mL/min
Cell Voltage	-21 V
Energy Filter	7.0 V
Chamber Temp.	5 °C

Table 3. Spike Recoveries of Cannabis Samples

Sample	⁷⁵ As (%)	¹¹¹ Cd (%)	²⁰⁰ Hg (%)	²⁰⁸ Pb (%)
Fortified Blank	105	105	102	102
Fortified Hard Candy	102	98	101	102
Fortified Granola Bar	91	99	102	101
Fortified MCT Oil	108	101	103	99
Fortified Ghee	109	101	103	100
Fortified Hemp Oil	105	104	104	100
Fortified Lotion	99	102	103	101
Fortified Froggy	95	98	97	101
Fortified Hemp Flower	96	101	102	100
Fortified Beef Jerky	96	100	101	103
Fortified Peanut Butter	104	103	103	101
Fortified Conc. CBD Oil	108	103	99	100

Table 4. Elemental Content of Samples

		⁷⁵ As (µg/kg)	¹¹¹ Cd (µg/kg)	²⁰⁰ Hg (µg/kg)	²⁰⁸ Pb (µg/kg)
Blank	Mean value	n.d.	n.d.	n.d.	n.d.
	RSD (n = 3)	---	---	---	---
Hard Candy	Mean value	n.d.	n.d.	n.d.	0.0116
	RSD (n = 3)	---	---	---	1.79
Granola Bar	Mean value	n.d.	0.125	n.d.	0.0186
	RSD (n = 3)	---	1.63	---	3.02
MCT Oil	Mean value	n.d.	n.d.	n.d.	n.d.
	RSD (n = 3)	---	---	---	---
Ghee	Mean value	n.d.	n.d.	n.d.	n.d.
	RSD (n = 3)	---	---	---	---
Hemp Oil	Mean value	n.d.	n.d.	n.d.	0.0723
	RSD (n = 3)	---	---	---	1.6
Lotion	Mean value	n.d.	n.d.	n.d.	n.d.
	RSD (n = 3)	---	---	---	---
Froggy	Mean value	n.d.	n.d.	n.d.	0.0407
	RSD (n = 3)	---	---	---	0.66
Hemp Flower	Mean value	0.0231	n.d.	n.d.	0.163
	RSD (n = 3)	6.07	---	---	1.09
Beef Jerky	Mean value	n.d.	0.0482	n.d.	0.057
	RSD (n = 3)	---	0.77	---	1.65
Peanut Butter	Mean value	n.d.	0.111	n.d.	0.0511
	RSD (n = 3)	---	3.92	---	1.32
Conc. CBD Oil	Mean value	n.d.	n.d.	n.d.	0.0662
	RSD (n = 3)	---	---	---	0.78

Table 5. Medical Action Levels for the State of California

Medical Action Levels*	As (µg/g)	Cd (µg/g)	Hg (µg/g)	Pb (µg/g)
Edible Cannabis Products	1.5	0.5	3	0.5
Inhaled Cannabis Products	0.2	0.2	0.1	0.5
Topical	3	5	1	10

* Based on 0.5 g sample size

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