

Abstract

As of January 2020, 11 US States, and all of Canada, have legalized recreational cannabis with Illinois posed for legality in January 2020. In addition, 33 states are approved for medical uses. As more products make it to consumers it is imperative that proper testing be performed to ensure the safety of the consumer. Canada and all US states require testing of what is known as the big four heavy metals (As, Cd, Pb, Hg). Some states require additional metals as, in the US, cannabis is regulated by each state. Health Canada is the sole regulator in Canada.

Better analytical results begin with better sample preparation. Due to the complex nature of the cannabis plant, a complete and reliable analysis can be difficult to achieve. If the matrix is not completely broken down it is common to encounter interferences resulting in false positive analyses. A MARS 6 microwave digestion system with MARSXpress Plus vessels was used to simultaneously digest a variety of cannabis and hemp materials. These included hemp crude oil, tinctures, and hemp flower along with food materials that are popular items for edibles. The preparation and analysis will be discussed and presented.

Introduction

As more and more cannabis and hemp based products make their way to market, cannabis testing labs are faced with the challenge of testing a wide variety of samples for heavy metals, and other contaminants, in a short amount of time. To improve throughput and productivity, digesting all samples in a single batch is preferred.

In this study a variety of cannabis product samples were digested in a single batch for heavy metals analysis by ICP-OES. These samples varied in composition and appearance but were digested in a single batch. Pre-digestion sample handling techniques for optimal digestion were also utilized.

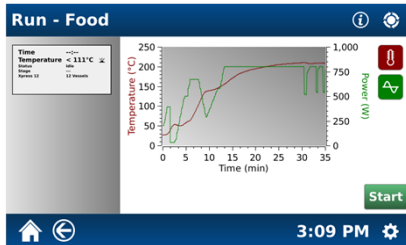


Figure 1. Graph of run showing all samples are heated to 200 °C

Procedure and Method

Twenty four samples, including spikes, blanks, and duplicates, were prepared according to **Table 1** in order to achieve the best digestion possible. After initial preparation, 0.5 g of each sample was weighed into a MARSXpress Plus vessel. All spiked samples were spiked with 0.4 ppm As, 0.4 ppm Cd, 1 ppm Hg, and 1 ppm Pb. Ten mL of a 9:1 HNO₃:HCl acid mixture was added to each vessel and they were sealed and heated in a CEM MARS 6 using the One Touch™ Cannabis Method. The One Touch method automatically detected the type of vessel being used, counted the number of samples, and applied enough power to heat the samples to 200 °C and hold them at this temperature for 15 min. **Figure 1** shows the temperature graph for this digestion run. The total digestion time for 24 samples was 45 min including cool down. Upon completion of the digestion run all samples were diluted with DI H₂O for analysis. All samples were analyzed on an Agilent 5110 ICP-OES with conditions shown in **Table 2**.

Table 1. Samples and pre-digestion sample handling

Sample	Preparation
Topical Cream	Placed on plastic spatula to deposit on bottom of vessel
Fruit Snack*	Cut in half with ceramic scissors
Cannabis Flower	Torn into small pieces
CBD Oil 4.5k	Deposited on bottom of vessel with plastic pipette
CBD Oil 6k	Deposited on bottom of vessel with plastic pipette

*Cryogenic grinding is also a suitable option for particle size reduction.

Table 2. ICP-OES Conditions

Parameter	Setting
Plasma viewing mode	Axial
Read time	30 s
Measurement replicates	3
RF incident power	1400 W
Plasma argon flow rate	12 L/min
Nebulizer argon flow rate	0.7 L/min
Auxiliary argon flow rate	1.0 L/min
Inner diameter of the torch injector	1.8 mm
Nebulizer type	Seaspray
Spray chamber type	Glass cyclonic double-pass
Sample tubing	White/white 1.04 mm ID
Internal standard tubing	Orange/green 0.38 mm ID

Results

All digestate solutions were clear and colorless upon dilution with DI H₂O. Presence of particulate or cloudiness is an indicator of an incomplete digestion. Incomplete digestions will not yield accurate analytical results and may give false positives or negatives.

All samples were analyzed in triplicate using two wavelengths for each element for the purpose of confirmation. Readings were taken axially with a standard torch.

Table 3. Spike recoveries of cannabis samples

Sample	As (%)	Cd (%)	Hg (%)	Pb (%)
Topical Cream	104.7	105.6	101.8	102.2
Fruit Snack	105.0	106.1	99.7	91.0
Cannabis Flower	106.8	102.0	95.6	102.5
CBD Oil 4.5k	101.5	101.3	92.3	102.6
CBD Oil 6k	100.9	100.4	91.9	101.8

Conclusion

CEM has developed a simple digestion protocol for digestion of a wide variety of cannabis and hemp samples in a single run. 24 samples were completely digested and cooled in about 45 minutes. Labs can achieve the lowest cost per test with the CEM solution because the MARS 6 with MARSXpress Plus vessels can digest all cannabis samples in a single batch.

Although there has not been federal standardization on heavy metals testing of cannabis and cannabis products, CEM is intimately involved with ACL and AOAC organizations to create standard methodologies across the country.

