The Importance of Temperature on Complete Digestion of Cannabis-Based Products for Metals Analysis

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Introduction

Certain elements can cause adverse effects on human health. Heavy metals such as arsenic, cadmium, lead, and mercury are particularly toxic, plus, once released into the environment they accumulate in cannabis plants. As more is learned about cannabis and its products, elements beyond just the four main toxic heavy metals are of interest. Cannabis-based products such as foods, oils, tinctures, as well as others, should be tested for the presence of multiple elements to ensure consumer safety and product quality.

Cannabis-infused products have grown in popularity and must adhere to increased regulation, necessitating the need for rapid and accurate trace metals analysis. Good analysis relies heavily on a robust, reliable, and reproducible sample preparation technique. For trace metals analysis, this technique starts with a good digestion. Given the diversity of sample matrices and the desire for one digestion method that is suitable for all, the digestion parameters are crucial. In this study the importance of temperature to achieve the complete microwave-assisted acid digestion of a variety of different cannabis-based products is explored. This approach provides a rapid, efficient and simple process for trace metals analysis of cannabis-based products.

Materials and Methods

Samples were digested in triplicate using the MARS 6™ microwave digestion system with MARSXpress Plus vessels. The MARSXpress Plus vessels are simple to assemble and offer the ability to perform up to 24 digestions in a single batch.

Samples:
- NIST SRM 1575a Pine Needles
- NIST SRM Plant 4 (CannaQAP)
- Cannabis Flower
- CBD Isolate

Digestion Method:

1. Weigh 0.5 g of sample into MARSXpress Plus TFM liner.
2. Add 9 mL HNO_3 and 1 mL HCl (both trace level grade acids).
3. Cap vessel and place into the turntable.
4. Use digestion parameters as shown below.

Method	Control Type	Sample Type	Power (W)	Ramp (min)	Temp (ºC)	Hold Time (min)
Classic	Ramp to Temp	Organic	1600	20	180, 210, 240	15

5. Dilute all digests to 50 g prior to analysis on an Agilent 7850 ICP-MS.

Results

After analysis, the residual carbon content (one indicator of digestion completeness and important for accurate As analysis) and SRM recovery values for each digest temperature was compared. The results are presented in Table 1 and 2. Figure 1 provides a visual guide to determining a complete vs incomplete digest.

Table 1. Average carbon concentrations (ppm) in cannabis samples at various digestion temperatures. (n=3)

Sample	180 ºC	210 ºC	240 ºC
NIST 1575a	204.6	108.4	42.5
NIST Plant 4	298.0	161.7	92.9
Cannabis Flower	172.1	100.8	46.6
CBD Isolate	831.5	271.1	173.8

Table 2. Percent recovery for NIST SRM 1575a and Plant 4 at various digestion temperatures. (n=3)

Sample	Temperature (ºC)	As	Cd	Hg	Pb
1575a	180	104.1	90.7	80.4	81.9
	210	98.9	91.9	85.1	84.7
	240	100.2	89.2	79.6	81.0
Plant 4	180	103.4	101.8	77.6	96.8
	210	102.0	99.0	102.3	103.5
	240	94.8	89.8	89.3	92.1

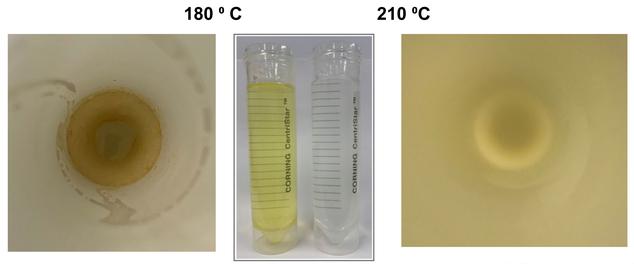

Figure 1: Pictures of the inside of the vessel after digestion and the digest of CBD Isolate at 180 ºC and 210 ºC

Conclusion

Incomplete digestions can result in high recoveries for As due to higher concentrations of residual carbon.

A complete digestion at 210 ºC results in the best recoveries across the board.

A digestion at 240 ºC doesn't significantly improve recoveries compared to 210 ºC. It can also cause loss of volatile elements and increased vessel wear.