

# Application Note XF2

## Microwave Digestion of Cannabis Oil

### Summary

A reliable sample preparation method to determine trace elements in cannabis oil is introduced below. The samples are digested using speedwave XPERT in high pressure DAK-100 vessels. During the digestion, the reaction temperature is controlled via contactless in-situ temperature sensor to ensure efficient digestion. Afterwards, the digested cannabis solutions are analysed by ICP-MS technique.

### Instrumentation

	Rotor and Vessel Type	Liner Type		
Microwave Digestion	<input type="checkbox"/> DAP-40X			<input type="checkbox"/> MiniVessels
	<input type="checkbox"/> DAP-60X	<input type="checkbox"/> DAQ-20H		<input type="checkbox"/> MiniVessels
	<input type="checkbox"/> DAP-100X	<input type="checkbox"/> DAQ-22H	<input type="checkbox"/> DAC-17	<input type="checkbox"/> MiniVessels
	<input checked="" type="checkbox"/> DAK-100X		<input type="checkbox"/> MultiTube	<input type="checkbox"/> MiniVessels
Analysis Technique	<input type="checkbox"/> AAS/AES	<input checked="" type="checkbox"/> ICP-MS	<input type="checkbox"/> ICP-OES	

### Procedure

Sample Amount	500 mg
Sample Preparation	n/a
Reagents <sup>[2]</sup>	8 ml HNO <sub>3</sub> (65%), 1 ml H <sub>2</sub> O <sub>2</sub> (35%), 1 ml HCl (37%) and 5 ml H <sub>2</sub> O
Experiment	<p>Weigh sample into the vessel. Add the reagent/s.</p> <p>Swirl the mixture carefully or stir with a clean PTFE or glass bar.</p> <p>Keep the vessel in the fume hood at least 1 hour for pre-reaction.</p> <p>Seal and close the vessels as described in the operation manual.</p> <p>Start the digestion according to the following temperature program.</p> <p>Allow the vessels to cool down to room temperature and open them carefully as described in the operation manual.<sup>[1]</sup></p> <p>Transfer the sample into centrifugal tubes and dilute them to 25 ml before the analysis.</p>

Temperature Program <sup>[2]</sup>	Step	T [°C]	p [bar] <sup>[3]</sup>	Ramp [min]	Hold [min]	Power [%] <sup>[4]</sup>
	1	150	60	10	5	25
	2	210	60	5	20	40
	3	50	60	1	15	0

## Results

Inductively coupled plasma mass spectrometry (ICP-MS) is used to determine the concentrations of arsenic (As), selenium (Se), cadmium (Cd), mercury (Hg) and lead (Pb) inside the cannabis oil samples.

The results for three samples are presented in the table below. Samples „a“ and „b“ are the digested solutions of the cannabis oils. Sample „c“ is the digested solution that is spiked with 5 ppm stock solution.

Samples	As [µg/l]	Se [µg/l]	Cd [µg/l]	Hg [µg/l]	Pb [µg/l]
a	0.007	0.083	0.002	0.021	0.019
b	0.012	0.101	0.004	0.012	0.221
c + 5 ppb	4.493	4.792	4.703	4.745	4.949

## Discussion

The rapid increasing interest of cannabis products requires proper sample preparation and analysis techniques for cannabis products. Due to the toxicity of heavy metals that can contaminate these products, it is crucial to test variety of cannabis samples to maintain consumer safety. At this point, speedwave XPERT provides very fast (30-40 min) and reproducible microwave digestion to break the complex organic matrix of the cannabis products. When the matrix is completely destroyed with the help of concentrated acids at high temperatures, the elements are extracted in the digested solution for qualitative and quantitative analysis. This reproducible digestions at elevated temperatures are achieved by contactless in-situ temperature and pressure sensors.

Formation of gaseous products (e.g. CO<sub>2</sub>) during the reaction between organic matrix with the oxidizing acids and vapor pressure of the acids at high temperatures increases the pressure inside the digestion vessel. If high sample weights are required for the digestion, usage of high pressure vessels, namely DAK-100, is crucial to ensure safe digestions. This is due to the fact that DAK-100 vessels can be safely operated up to the pressure of 100 bar.

In this application, 500 mg of cannabis oil is digested by following EPA 3052 Norm for organic based matrices. The cannabis based oil used in this digestion is a type of a food ingredient in salad dressings or used for cooking purposes. Since the oil sample can trigger temperature spikes as a result of exothermic reactions, deionized water is added to the oxidizing acid mixture (HNO<sub>3</sub> and H<sub>2</sub>O<sub>2</sub>). This is also consistent with the cautions written in EPA 3052 Norm. Additionally, sample to sample variety can result in different experimental results, especially for oil containing organic samples. Therefore, we suggest that microwave digestion can be optimized by starting with a smaller initial weight of 250 mg and increasing the weight stepwise to 500 mg.

After the microwave digestion of cannabis oil finished, the digested solution was clear and colorless. The digested solutions are analyzed by ICP-MS to determine the concentration of trace metals inside the sample. The results show acceptable concentrations of heavy metals inside the cannabis oil. The spiked sample „c“ shows good recoveries. Further method validation of ICP-MS analysis is not in the scope of this application note.

To conclude, this work demonstrated the ability of the speedwave XPERT to prepare microwave digested cannabis oil in high pressure DAK-100 closed vessels for trace metal analysis.

## Notes

- [1] To avoid foaming and splashing, wait until the vessels have cooled to room temperature (about 20 min). Carefully open the digestion vessel in a fume hood wearing hand, eye and body protection, since a large amount of fumes will be produced during the digestion process.
- [2] This application serves only as a guideline and may need to be optimized for your sample.
- [3] Pressure is the maximum value given to the program that is limited by the vessel and/or rupture disc specifications.
- [4] This application is outlined for 4 samples. Increase or decrease the power by 10% per sample, when using more or less sample. Minimum is 40% independent of the sample number.

## Reference

EPA 3052