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speedwave XPERT
Food & Feed

Application Note XF1
Microwave Digestion of Cannabis Tea

Summary

A reliable sample preparation method to determine trace elements in cannabis tea leaves 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	☐ DAP-40X			☐ MiniVessels
	☐ DAP-60X	☐ DAQ-20H		☐ MiniVessels
	☐ DAP-100X	☐ DAQ-22H	☐ DAC-17	☐ MiniVessels
	☑ DAK-100X		☐ MultiTube	☐ MiniVessels
Analysis Technique	☐ AAS/AES	☑ ICP-MS	☐ ICP-OES	

Procedure

Sample Amount	1000 mg
Sample Preparation	n/a
Reagents [2]	8 ml HNO_3 (65%), 1 ml H_2O_2 (35%) and 1 ml HCl (37%)
Experiment	Weigh sample into the vessel. Add the reagent/s.
	Swirl the mixture carefully or stir with a clean PTFE or glass bar.
	Keep the vessel in the fume hood at least 1 hour for pre-reaction.
	Seal and close the vessels as described in the operation manual.
	Start the digestion according to the following temperature program.
	Allow the vessels to cool down to room temperature and open them carefully as described in the operation manual. [1]
	Transfer the sample into centrifugal tubes and dilute them to a volume of 25 ml before analysis.

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

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Results

The 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 tea leaves.

The results for three samples are presented in the table below. Sample „a" and „b" are the digested solutions of the cannabis tea leaves. 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	4.404	1.691	1.667	0.293	17.001
b	4.299	1.801	1.753	0.286	16.800
c + 5ppb	10.028	7.738	5.946	4.966	21.178

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. These reproducible digestions at elevated temperatures are controlled by contactless in-situ temperature and pressure sensors.

Formation of gaseous products (e.g. CO_2) 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 (i.e. 1000 mg) 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, microwave digestion of cannabis leaves in 100 ml vessels results in clear and colorless digested solutions with some white precipitates at the bottom. These precipitates resulted from the silica content of the leaves. This can be confirmed by observing dissolution of the precipitates after adding HF acid to the precipitates. If digestion of silica in cannabis leaves is not required, the microwave digestion procedure above is sufficient to digest the organic matrix for elemental analysis. If the digestion of silica is also the purpose of interest, the digestion reagents can be modified by addition of HF (1-2 ml) into the reagents. If the silica content is the purpose of interest, it can be filtered out for further analysis.

As shown in the results part, concentrations of the As, Se, Cd and Hg elements are determined below 5 µg/l; while the concentration of Pb is determined as ~16 µg/l in cannabis samples „a" and „b" by ICP-MS. The reliable recoveries is observed within the spiked sample „c". ICP-MS analysis provides reliable and reproducible results and good recoveries. It is important to note that 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 tea leaves 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.