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Application News

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Spectrophotometric Analysis

Analysis of "The Big Four" Heavy Metals in Cannabis by USN-ICP-OES

Dan Davis, Keith Long, Justin Masone, Patricia Firmin

■ Introduction

Plants concentrate metals by absorbing them from the soil in which they are grown. Some metals are beneficial and essential for life whereas other metals are highly toxic and have negative effects with even the lowest of levels. Because of their toxicity, quantification of these elements is required and their limits are based on daily consumption.

This application news will investigate the preparation and analysis for heavy metals in cannabis using ICP-OES with an ultrasonic nebulizer (USN-ICP-OES) and a low-flow mini-torch as a lower cost alternative to ICP-MS.

Spike and Recovery tests were performed at the Regulation target levels to ensure accuracy and sensitivity of the technique.

Element	Regulation Target ppm	As Prepped (50x dil.) ppb
Lead	1.20	24
Cadmium	0.82	16
Methyl Mercury	0.40	8.0
Inorganic Arsenic	2.00	40

Table 1: Metals Analysis Target Based on 5 gram daily intake

■ Sample Preparation

0.5 grams of cannabis flower was added to a closed vessel digester, and 2 mL of Nitric Acid, 2.0 mL of 30% H_2O_2, and 4.0 mL of DI H_2O were added. The sample was ramped to 900 watts over 15 minutes and held for another 20 minutes at that power. The sample was allowed to cool. 0.5 mL of HCl acid was added along with Yttrium as an Internal Standard and a mercury stabilizer. The solution was brought to 25.0 mL total volume with DI H_2O.
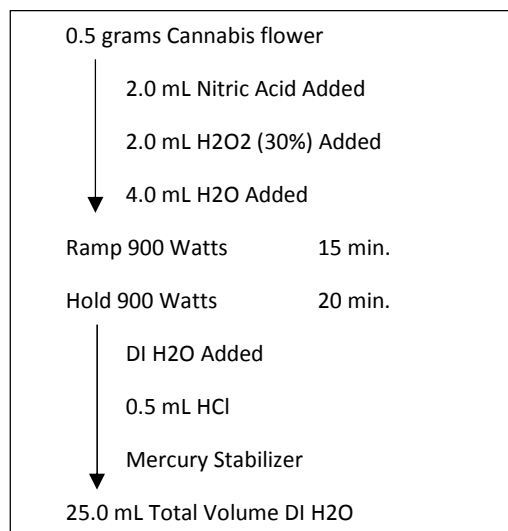
0.5 grams Cannabis flower

  2.0 mL Nitric Acid Added

  2.0 mL H2O2 (30%) Added

  4.0 mL H2O Added

Ramp 900 Watts   15 min.

Hold 900 Watts   20 min.

  DI H2O Added

  0.5 mL HCl

  Mercury Stabilizer

25.0 mL Total Volume DI H2O

Fig. 1: Digestion/Preparation Procedure

■ Analytical Method and Conditions

Measurement was conducted using the calibration curve method with an Internal Standard. The main measurement parameters of the instrument are shown in Table 2.

	As	Cd	Hg	Pb	Y
Wavelength nm	193	214	194	220	360
View Mode	Axial				
Attached Instruments:	Mini Torch, Ultrasonic Nebulizer, Inside Pump				
Radio Frequency Power:	1.00 kW				
Plasma Gas:	8.00 L/min				
Auxiliary Gas:	0.60 L/min				
Carrier Gas:	0.60 L/min				
Exposure Time:	30 sec				
Condition:	Wide Range				

Table 2: Measurement Conditions

The standard solutions were prepared by diluting commercially available standard solutions for atomic absorption measurement. 100 mL of stock calibration solution was prepared at 320 ppb As, 192 ppb Pb, 64 ppb Hg and 128 ppb Cd stabilized with 1% nitric acid.

Add 50 mL of the stock solution to a 100 mL volumetric and add 0.5 mL HCl, 100 uL of 100 ppm Y standard and mercury stabilizer. Bring to 100 mL; this is Calibration solution 4. Add 25 mL of the stock solution to 100 mL volumetric. Add HCL, Y and mercury stabilizer, and bring to 100 mL; this is calibration solution 3. Add 12.5 mL of the stock solution to a 100 mL volumetric, add HCl, Y and mercury stabilizer, and bring to 100 mL; this is calibration solution 2. Calibration solution 1 is blank with HCl, Y and mercury stabilizer.

Calibration Solution	1	2	3	4	Units
As	0	40	80	160	ppb
Cd	0	16	32	64	ppb
Hg	0	8	16	32	ppb
Pb	0	24	48	96	ppb
Y	100	100	100	100	ppb

Table 3: Calibration Solutions

■ Results and Conclusion

Figures 2, 3, 4 and 5 show the generated calibration curves for each of the analytes. All calibration curves achieved a good linear coefficient of 0.9999 or better, showing the instrument is capable of measuring the specified target range shown in Table 1 and Table 3.
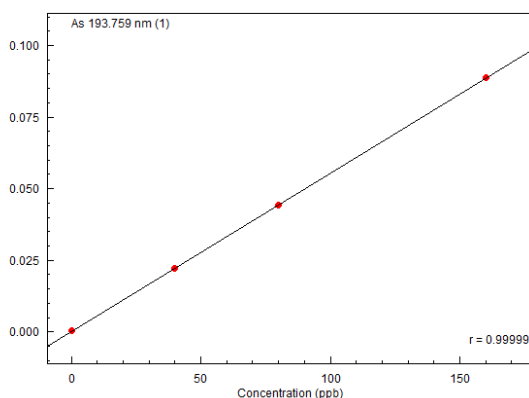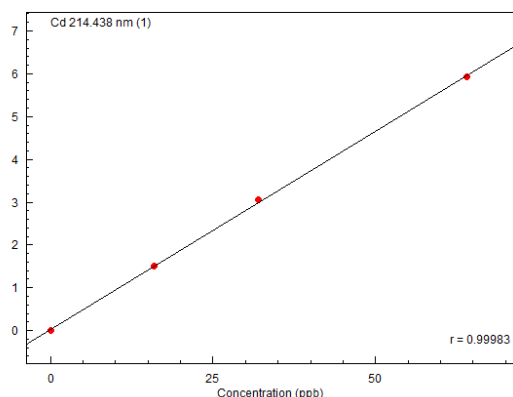
Fig. 2: As Calibration Curve
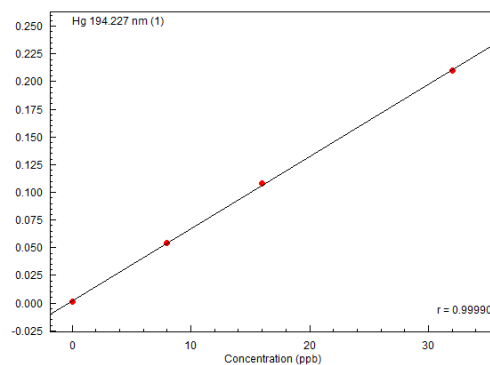
Fig. 3: Cd Calibration Curve
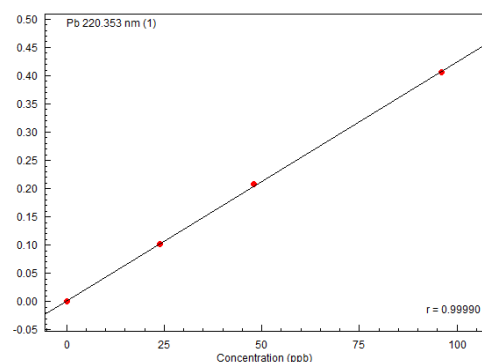
Fig. 4: Hg Calibration Curve

Fig. 5: Pb Calibration Curve

Figures 6 and 7 shows the signals of each analyte of the unknown samples and spikes for As, Cd, Hg and Pb. There is good signal distinction at each of the target analysis levels indicated in Table 1.
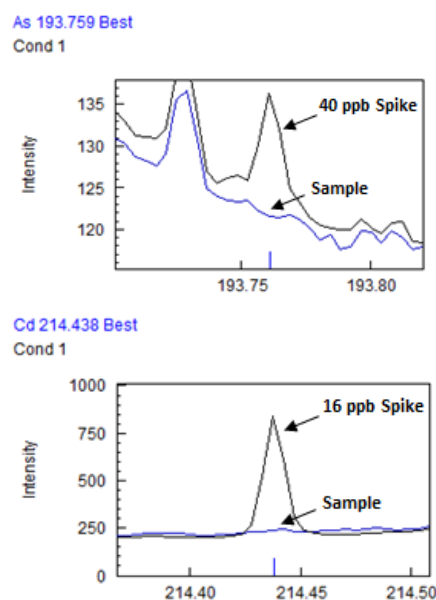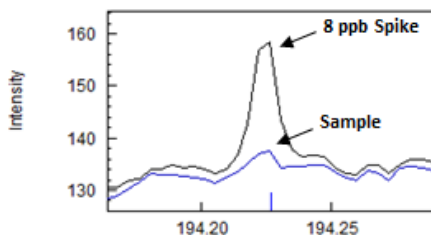
Fig. 6: As & Cd Sample & Spike
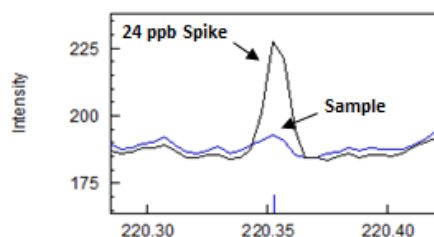
Fig. 7: Hg & Pb Sample & Spike

Element	Sample (ppb)	Spike (ppb)	Recovery %
As	0.05	43.3	108.1
Cd	0.37	15.9	97.1
Hg	3.16	11.5	104.3
Pb	3.96	27.7	98.9

Table 4: Results of Sample and Spikes

The results in Table 4 show that all of the elements were within the target analysis and the sample spikes achieved recoveries of 100% (+/-10%) of their theoretical values. The recoveries show that the system is capable achieving good sensitivity and accuracy at the desired elemental concentration levels. The results also show that USN-ICP-OES is a suitable alternative to costlier techniques like ICP-MS.

Additionally, the ICP incorporates a vertically mounted mini-torch that achieves the same sensitivity as conventional torches but uses half the amount of argon, leading to substantial cost savings over the lifetime of the instrument. The vertical orientation of the torch allows it to handle high dissolved solids without fouling, extending the time between maintenance operations and making for a highly sensitive, robust ICP-OES system for analyzing heavy metals in cannabis samples.

ICPE-9800
Emission Spectrometer

AA-7000
Atomic Absorption

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