APPLICATION BRIEF

ICP – Mass Spectrometry

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Analysis of Plant Materials for Toxic and Nutritional Elements with the NexION ICP-MS

Introduction

Plants primarily serve as a food substance, being an important source of nutrients. However, toxic elements can also be found in plants, primarily through uptake from the soil, water, and fertilizer. Therefore, it is important to measure both the nutritional and toxic elemental content of plants and plant materials.

Several challenges arise in the elemental analysis of plants. First, because both toxic and nutritional elements must be measured, a wide dynamic range is required. Plants are complex biological entities which require sample preparation, usually consisting of homogenization followed by digestion in order to break down the complex matrix and extract the elements. Despite these steps, matrix-induced spectral interferences still persist which could cause false readings, especially for the toxic elements. Therefore, Collision or Reaction Cell technology has to be used to remove the interferences.

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One plant species which is gaining considerable interest in the U.S. is cannabis (i.e. marijuana) since its use has been legalized in several states, both for recreational and medicinal purposes through inhalation and consumption in food products. With its increased use, interest in the toxic and mineral element content has also risen.

An additional challenge of cannabis analysis in the U.S. is legally attaining samples, since it is illegal in some states. However, hops are a generally accepted surrogate for cannabis due to its similar chemical and physical properties.

This work discusses the analysis of hops (as a surrogate for cannabis) for both toxic and nutritional elements with ICP-MS.

Experimental

Sample Preparation

Hops were purchased from a commercial store and chopped into small pieces, both to homogenize the sample and expose more surface area for increased digestion efficiency. The Titan MPS™ microwave sample preparation system with the standard 75 mL PTFE vessels was used for digestion, following the program in Table 1. Each vessel contained 0.25 g of plant material, 5.0 mL of concentrated nitric acid, 5.0 mL water, and 3.0 mL of 30% hydrogen peroxide. After digestion, the samples were diluted to 50 mL with deionized water, along with the addition of gold (Au) to stabilize mercury (200 µg/L Au in the final solution).

Table 1. Titan MPS Microwave Digestion Program

Step	Target Temp (°C)	Pressure, Max (bar)	Ramp Time (min)	Hold Time (min)	Power
1	150	30	5	5	60
2	200	30	5	20	90
3	50	30	1	10	0

Instrumental Conditions

All analyses were performed on a PerkinElmer NexION® ICP-MS with the standard sample introduction components and conditions. The elements and analysis mode used are shown in Table 2. The internal standards were added on-line via a mixing tee. The final concentration introduced to the instrument were 10 mg/L Sc, 5 mg/L Ge, and 0.1 mg/L Rh, In, Tb in 10% methanol and 1% nitric acid. Using both Standard and Collision modes, the analysis time was 100 seconds per sample.

Results and Discussion

Table 3 shows the average results for two digestions of hops. To test the accuracy, pre-digestion spikes were added for those elements present at less than 50 mg/kg. The spike levels were 20 mg/L for all elements, except Hg, which was spiked at 2 mg/L. All spike recoveries were within 15% of the added amounts, further validating the methodology.

Table 2. Elements and Analysis Mode

Element	Mass	Mode
Beryllium (Be)	9	Standard
Boron (B)	11	Standard
Sodium (Na)	23	Collision
Magnesium (Mg)	24	Collision
Aluminum (Al)	27	Collision
Phosphorus (P)	31	Collision
Sulfur (S)	34	Collision
Potassium (K)	39	Collision
Calcium (Ca)	44	Collision
Vanadium (V)	51	Collision
Chromium (Cr)	52	Collision
Manganese (Mn)	55	Collision
Iron (Fe)	56	Collision
Cobalt (Co)	59	Collision
Nickel (Ni)	60	Collision
Copper (Cu)	63	Collision
Zinc (Zn)	66	Collision
Arsenic (As)	75	Collision
Selenium (Se)	78	Collision
Strontium (Sr)	88	Collision
Molybdenum (Mo)	95	Collision
Cadmium (Cd)	111	Collision
Tin (Sn)	118	Standard
Antimony (Sb)	121	Standard
Barium (Ba)	137	Standard
Mercury (Hg)	202	Standard
Thallium (Tl)	205	Standard
Lead (Pb)	208	Standard
Thorium (Th)	232	Standard
Uranium (U)	238	Standard

Table 3. Results for Analysis of Hops

Element	Experimental (mg/kg)	% Recovery
Be	0.00	86
B	27.7	106
Na	13.2	113
Mg	3617	---
Al	10.8	108
P	6580	---
S	2001	---
K	34358	---
Ca	10936	---
V	0.04	101
Cr	0.23	96
Mn	17.3	113
Fe	58.6	---
Co	1.33	105
Ni	2.27	107
Cu	6.27	90
Zn	31.8	122
As	0.03	100
Se	0.20	102
Sr	17.9	99
Mo	0.93	102
Cd	0.02	97
Sn	0.28	96
Sb	0.01	96
Ba	18.1	94
Hg	0.04	97
Tl	0.00	88
Pb	0.65	87
Th	0.00	95
U	0.00	95

Conclusions

This work has demonstrated the ability of the NexION ICP-MS, combined with a Titan MPS microwave, to effectively analyze hops (as a surrogate for cannabis) for both nutritional and toxic elements. Analyses are accomplished in both Collision and Standard modes and require only 100 seconds per sample. The accuracy of the applied method was previously validated by analyzing a variety of NIST™ plant materials[1].

References

1. Bosnak, C., Pruszkowski, E., *"The Determination of Toxic, Essential, and Nutritional Elements in Food Matrices Using the NexION 300/350 ICP-MS"*, PerkinElmer Application Note.

Consumables Used

Component	Description	Part Number
Sample Uptake Tubing	0.38 mm id (green/orange), PVC, flared, 2-stop	N0777042
Drain Tubing	1.30 mm id (gray, gray), Santoprene, 2-stop	N0777444
Internal Standard Addition Tee	Tee for on-line addition of internal standard	N0777295
Internal Standard Uptake Tubing	0.25 mm id (red/orange), PVC, flared, 2-stop	N0773111
Multielement Standard Solution	100 mg/L Ag, Al, As, Ba, Ve, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Sr, Tl, V, Zn	N9301721 (125 mL)
Multielement Salt Solution	1000 mg/L Ca, Mg, Na, K	N9307805 (125 mL)
Mercury Solution	10 mg/L Hg	N9300253 (125 mL)
Internal Standard Solution	Sc (100 mg/L), Ge (50 mg/L), and In, Rh, Tb (1 mg/L)	N9308592 (125 mL)
Pure-Grade Au Standard	1000 mg/L	N9303728 (125 mL)
Autosampler Tubes	Conical, metal-free, sterile	N0776118 (15 mL) N0776116 (50 mL)

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