Multi-element Analysis of Cannabis using the Agilent 7800 ICP-MS

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Introduction

In the U.S., marijuana remains a Schedule I controlled substance. Worldwide, marijuana use is legal for medicinal purposes in countries including Australia, Canada, Croatia, Czech Republic, Macedonia, Poland, and Puerto Rico. More than 30 U.S. states have legalized medical and/or recreational marijuana (cannabis) for adult use [1]. This is also the case in some countries including the Netherlands, Spain, South Africa, and Uruguay.

Countries and U.S. states that permit use of medicinal and recreational marijuana require rigorous testing of cannabis and associated products to ensure safety from inorganic contaminants, including toxic elements such as As, Cd, Pb, and Hg. The analysis of mineral and additional trace elements provides labeling information that is required when these products are used as nutritional supplements. As contamination can occur during the manufacturing process, analysis is necessary at all stages of production.

Trace element analysis of plant and nutritional supplement materials is well established [2]. Following acidic digestion to break down the primary components of the plant-based samples, ICP-MS is often used for multi-element analysis because of its high sensitivity, robustness, and wide dynamic range.

Some plant materials may contain rare earth elements (REEs) or lantanides (LA) which may bias the final results due to interference from doubly charged ions (REE^{++}) on elements such as As and Se. The Agilent 7800 ICP-MS corrects for these interferences using "half mass correction" tuning, which is automatically set up in the ICP-MS MassHunter software. The software also collects semiquantitative data across the entire mass region, referred to as Quick Scan. Quick Scan provides data for elements that may not be present in the calibration standards.

In this study, the 7800 ICP-MS was used to analyze 25 elements in a range of cannabis and cannabis-related products.

Experimental

An Agilent 7800 ICP-MS with the standard High Matrix Introduction (HMI) system was used for the analysis. Sampling was performed by the Agilent SPS4 autosampler. The ICP-MS was configured with the standard sample introduction system consisting of a Micromist concentric nebulizer, quartz spray chamber, and quartz torch with 2.5 mm id injector. The cones were nickel plated with a copper core sampler. Instrument operating conditions are listed in Table 1.

Parameter	Value
RF power (W)	1600
Sampling depth (mm)	10
Carrier gas (L/min)	0.80
Dilution (HMI) gas (L/min)	0.15
Helium cell gas (mL/min)	4.3
Energy discrimination (V)	3.0

Table 1. ICP-MS operating conditions.

Step	Ramp time (Min)	Temp (°C)	Hold time (Min)	Nitrogen gas starting pressure (bar)
1	20	240	15	35

Table 2. Parameters for microwave digestion.

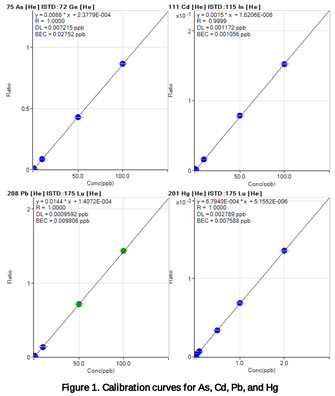


Figure 1. Calibration curves for As, Cd, Pb, and Hg

Various SRMs purchased from National Institute of Standards and Technology (NIST) were analyzed in this study to verify the sample preparation digestion process. NIST 1547 Peach Leaves, NIST 1573a Tomato Leaves, and NIST 1575 Pine Needles.

Experimental

Samples

A range of cannabis and products containing cannabis were analyzed in this study. The samples included cannabis, cantabs, CAN CBD Tincture, Cannabella, and a hemp-based body cream.

Standard and sample preparation

Calibration standards were prepared using a mix of 1% HNO_3 and 0.5% HCl. Na, Mg, K, Ca, and Fe were calibrated from 0.5 to 10 ppm. Hg was calibrated from 0.05 to 2 ppb. All remaining elements calibrated from 0.5 to 100 ppb.

4 mL HNO_3 and 1 mL HCl were added and the samples were microwave digested using the program given in Table 2. HCl was included to ensure the stability of Ag and Hg in solution

Mass	Element	ISTD	R	DL, ppb	BEC, ppb
9	Be	^{6}Li	1.0000	0.0130	0.0055
23	Na	^{45}Sc	1.0000	2.2898	63.9621
24	Mg		1.0000	0.2617	0.4355
27	Al	^{6}Li	1.0000	0.2685	0.5958
39	K	^{45}Sc	0.9999	2.8386	60.8172
44	Ca	^{6}Li	1.0000	1.0935	14.7211
51	V		1.0000	0.0046	0.0978
52	Cr	^{45}Sc	1.0000	0.0070	0.0339
55	Mn		0.9999	0.0123	0.0772
56	Fe		0.9999	0.0048	0.7538
59	Co		1.0000	0.0007	0.0042
60	Ni		0.9999	0.0127	0.0282
63	Cu	^{72}Ge	0.9999	0.0060	0.1106
66	Zn	^{45}Sc	0.9999	0.0335	0.2284
75	As		1.0000	0.0162	0.0509
75	As		1.0000	0.0107	0.0420
78	Se	^{72}Ge	1.0000	0.2533	0.4015
78	Se		1.0000	0.1102	0.4232
95	Mo		0.9998	0.0024	0.0091
107	Ag	^{115}In	0.9998	0.0048	0.0090
111	Cd		0.9999	0.0026	0.0064
137	Ba	^{175}Lu	1.0000	0.0075	0.0361
201	Hg		1.0000	0.0057	0.0182
205	Tl	^{209}Bi	1.0000	0.0068	0.0499
208	Pb	^{175}Lu	0.9999	0.0042	0.0300
232	Th		1.0000	0.0006	0.0037
238	U		1.0000	0.0010	0.0027

Table 3. Calibration summary data acquired in He mode.

Type	ICV (ppb)	True Value	%Recovery
Sample Name	NIST 1640a		
9 Be [He]	3.0484	3.0260	100.74
23 Na [He]	3082.9875	3112.0000	99.07
24 Mg [He]	1031.3340	1050.2000	98.20
27 Al [He]	52.7528	53.0000	99.53
39 K [He]	597.2875	575.3000	103.82
44 Ca [He]	5552.9514	5570.0000	99.69
51 V [He]	14.5807	15.0500	96.88
52 Cr [He]	38.3580	40.5400	94.62
55 Mn [He]	40.6565	40.3900	100.66
56 Fe [He]	36.4852	36.8000	99.14
59 Co [He]	19.6547	20.2400	97.11
60 Ni [He]	24.4958	25.3200	96.74
63 Cu [He]	82.7862	85.7500	96.54
66 Zn [He]	54.2268	55.6400	97.46
75 As [He]	7.8543	8.0750	97.27
75 As [NP He]	7.7797	8.0750	96.34
78 Se [He]	19.6682	20.1300	97.71
78 Se [NP He]	19.7380	20.1300	98.05
95 Mo [He]	44.1769	45.6000	96.88
107 Ag [He]	7.8097	8.0810	96.64
111 Cd [He]	3.8842	3.9920	97.30
137 Ba [He]	152.5349	151.8000	100.48
201 Hg [He]	0.0304		
205 Tl [He]	1.5925	1.6190	98.36
208 Pb [He]	11.8922	12.1010	98.27
232 Th [He]	0.0025		
238 U [He]	23.6379	25.3500	93.25

Table 4. ICV recovery

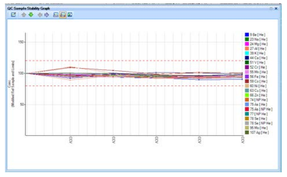
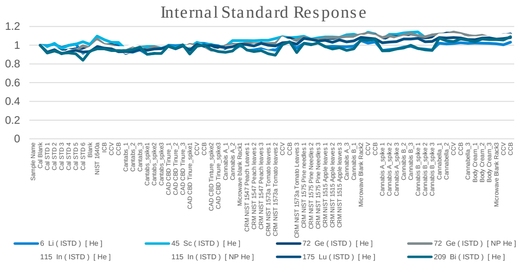


Figure 1. CCV Stability



Results and Discussion

As part of the instrument quality control (QC), NIST 1645a Natural Water was used as an Initial Calibration Verification (ICV) standard. The results given in Table 4 show that the recoveries for all the certified elements present in 1645a were excellent, ranging from 93-104%. A mid-level calibration standard comprising mineral elements at 5 ppm, Hg at 1 ppb and all trace elements at 50 ppb was used as the Continuing Calibration Verification (CCV) solution. The CCV was analyzed six times throughout the run. The mean recoveries and range are shown in Figure 1. All CCV recoveries were within ±10% of the expected value. Three SRMs were analyzed to verify the digestion process (results not shown).

Results and Discussion

For most elements, the mean 7800 results were in good agreement with the certified concentrations where certified values are provided. The measured results for As in NIST 1547 and Se in both NIST 1547 and 1573a did not show such good agreement. Some plant materials may contain high levels of rare earth elements (REEs), also known as lanthanides (LA). These elements have low second ionization potentials, so readily form doubly-charged ions (REE^{++}). As the quadrupole mass spectrometer separates ions based on their mass to charge ratio (m/z), these doubly-charged ions appear at half their true mass. Doubly-charged ions of the REEs ^{150}Nd, ^{150}Sm, ^{156}Gd, ^{156}Dy, ^{160}Gd, and ^{160}Dy therefore appear at m/z 75, 78 and 80, potentially causing overlaps that can bias the results for As and Se in samples that contain high levels of the REEs. The Agilent 7800 ICP-MS corrects for these interferences using "half mass correction", which is automatically set up in the ICP-MS MassHunter software. The improvement provided by half-mass correction was observed.

Quantitative results are given in Table 5 for two cannabis-related products, Cantabs and CAD CBD Tincture, and two lots of cannabis samples (A and B). The concentrations of As (160.0 ppb), Cd (11.33 ppb), Pb (24.00 ppb), and V (162.1 ppb) were relatively high in cannabis sample A. Pb and V were also high in cannabis sample B, at 55.40 and 143.4 ppb respectively. To check the accuracy of the method for actual sample analysis, **a spike recovery test** was carried out. The four samples were spiked with an Environmental Mix Spike containing multiple elements at 200 ppb, Na, Mg, K, Ca, Fe at 2000 ppb, and Hg at 4 ppb.

Element	Cantabs	CAD CBD Tincture	Cannabis A	Cannabis B	Canabella	Body Cream
	Average n=3, ppb	Average n=3, ppb	Average n=3, ppb	Average n=3, ppb	Average n=3, ppb	Average n=3, ppb
Dilution:	119	173	310	338	132	94
9 Be [He]	3.78	2.87	5.32	3.66	3.78	4.03
23 Na [He]	20193.75	<200.00	7372.86	50017.97	656227.91	52120.68
24 Mg [He]	33193.13	384.98	5425747.92	5807461.17	12127.89	15979.89
27 Al [He]	2310.13	240.44	5024.14	4586.44	888.27	1270.47
39 K [He]	46302.13	<500.00	41156140.45	34100713.88	30866.85	158272.30
44 Ca [He]	18382.32	1186.94	11394561.72	9681049.08	37459.26	33088.74
51 V [He]	16.46	1.10	37.04	25.09	13.00	13.12
52 Cr [He]	104.85	28.52	81.39	272.52	94.47	84.23
55 Mn [He]	183.66	16.34	114103.02	229650.39	174.74	264.32
56 Fe [He]	1914.93	569.44	252188.38	219811.08	2255.66	2369.72
59 Co [He]	5.62	3.18	162.08	143.45	4.23	9.72
60 Ni [He]	38.98	16.06	108.25	185.75	41.95	50.33
63 Cu [He]	121.63	24.87	10865.80	13467.10	204.91	269.06
66 Zn [He]	172.18	644.74	72504.02	126816.44	481.65	479.49
75 As [He]	7.04	8.86	159.71	24.43	6.06	6.58
75 As [NP He]	7.84	10.10	160.04	25.94	7.56	6.88
78 Se [He]	29.08	51.20	50.68	83.80	24.46	15.87
78 Se [NP He]	27.41	38.50	45.78	72.68	24.55	14.25
95 Mo [He]	24.12	34.20	3280.64	1589.42	36.14	28.79
107 Ag [He]	0.88	2.85	8.61	10.78	4.55	5.23
111 Cd [He]	4.54	5.58	11.33	7.47	4.35	5.30
137 Ba [He]	99.95	7.98	345.65	888.67	35.83	46.62
201 Hg [He]	<0.700	12.72	29.19	27.06	9.72	2.20
205 Tl [He]	<0.800	1017.08	1739.49	2038.31	1043.18	241.84
208 Pb [He]	17.49	6.89	24.00	55.40	10.08	11.37
232 Th [He]	4.56	2.46	5.55	4.05	2.59	4.39
238 U [He]	5.35	2.14	4.79	3.24	2.77	4.45

Table 5. Quantitative data for two cannabis-related products and two cannabis samples plus mean spike recovery results.

- The analysis of cannabis and associated products is easily performed using the Agilent 7800 ICP-MS.
- The Agilent 7800's HMI capability enables the routine analysis of samples that contain high and variable matrix levels, while minimizing the need for conventional liquid dilution.
- Agilent's ICP-MS MassHunter Quick Scan function provides a complete picture of the elements present in the sample, as data can be reported for elements not included in the calibration standards.
- The automated tuning of the ICP-MS for half mass correction allows As and Se to be determined with good accuracy, reducing the impact of doubly-charged interference caused by high levels of REEs.

References

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