

Introduction

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 [1]. 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 lanthanides (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.

Various SRMs bought 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.

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% HNO₃ 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 HNO₃ 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

Four samples (see Table 5) were fortified in triplicate using an Environmental Mix Spike solution and analyzed. The samples, spikes, and SRMs were diluted 5x before analysis to reduce the acid concentration.

Results and Discussion

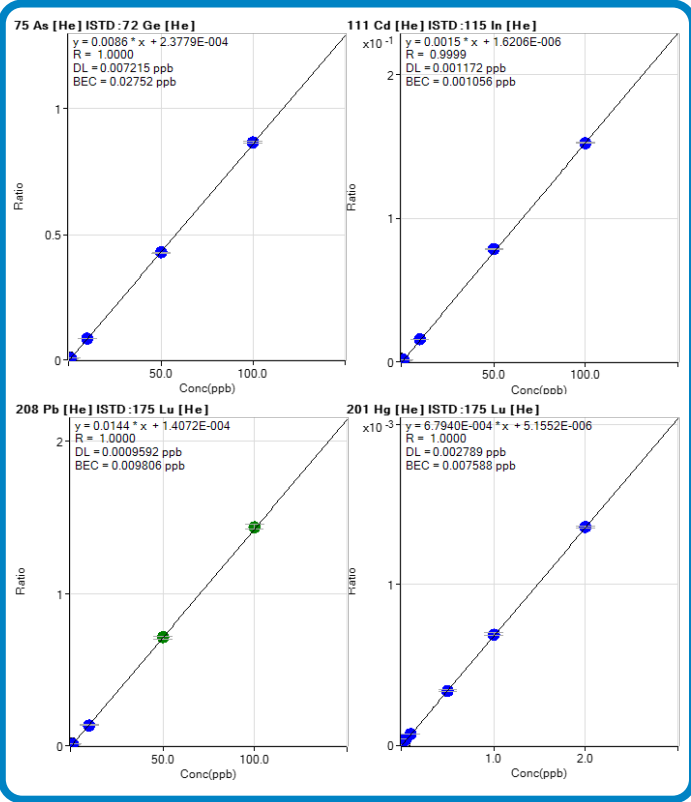


Figure 1. Calibration curves for As, Cd, Pb, and Hg

Sample Name	NIST 1640a		
Element	ICV (ppb)	True Value	%Recovery
9 Be [He]	3.05	3.03	100.74
23 Na [He]	3082.99	3112.00	99.07
24 Mg [He]	1031.33	1050.20	98.20
27 Al [He]	52.75	53.00	99.53
39 K [He]	597.29	575.30	103.82
44 Ca [He]	5552.95	5570.00	99.69
51 V [He]	14.58	15.05	96.88
52 Cr [He]	38.36	40.54	94.62
55 Mn [He]	40.66	40.39	100.66
56 Fe [He]	36.49	36.80	99.14
59 Co [He]	19.65	20.24	97.11
60 Ni [He]	24.50	25.32	96.74
63 Cu [He]	82.79	85.75	96.54
66 Zn [He]	54.23	55.64	97.46
75 As [He]	7.85	8.08	97.27
75 As [NP He]	7.78	8.08	96.34
78 Se [He]	19.67	20.13	97.71
78 Se [NP He]	19.74	20.13	98.05
95 Mo [He]	44.18	45.60	96.88
107 Ag [He]	7.81	8.08	96.64
111 Cd [He]	3.88	3.99	97.30
137 Ba [He]	152.53	151.80	100.48
201 Hg [He]	0.03		
205 Tl [He]	1.59	1.62	98.36
208 Pb [He]	11.89	12.10	98.27
232 Th [He]	0.00		
238 U [He]	23.64	25.35	93.25

Table 4. ICV recovery

Three SRMs were analyzed to verify the digestion process (results not shown). As in NIST 1547 and Se in both NIST 1547 and 1573a did not show such good agreement. Plant materials may contain high levels of REEs. 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 ¹⁵⁰Nd, ¹⁵⁰Sm, ¹⁵⁶Gd, ¹⁵⁶Dy, ¹⁶⁰Gd, and ¹⁶⁰Dy 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.



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		0.9998	0.0048	0.0090
111	Cd	¹¹⁵ In	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		0.9999	0.0042	0.0300
232	Th	¹⁷⁵ Lu	1.0000	0.0006	0.0037
238	U		1.0000	0.0010	0.0027

Table 3. Calibration summary data acquired in He mode.

As part of the instrument quality control (QC), NIST 1645a Natural Water was used as an Initial Calibration Verification (ICV) standard. Table 4 shows 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 (n=6). Recoveries and range shown in Figure 2. All CCV recoveries were within ±10% of the expected value.

Results and Discussion

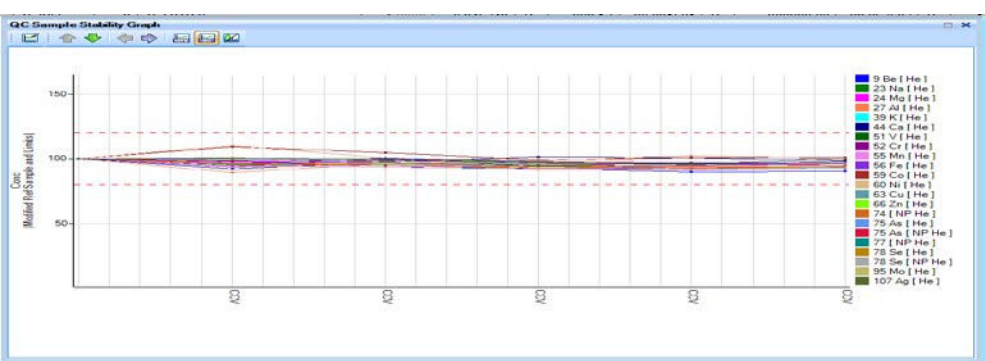


Figure 2. CCV Stability

	Cantabs	Cannabidiol tincture	Cannabis A	Cannabis B	
Element	Measured conc, n=3, ppb	Measured conc, n=3, ppb	Measured conc, n=3, ppb	Measured conc, n=3, ppb	Mean spike recovery ± 1σ, %
9 Be	3.785	2.869	5.323	3.66	109±6
23 Na (ppm)	20.03	<0.250**	7.372	50.017	110±4
24 Mg (ppm)	33.19	0.385	5425.75	5807.461	113±10
27 Al (ppm)	2.31	0.24	5.024	4.586	112±12
39 K (ppm)	46.3	<250**	41156	34100.714	na
44 Ca (ppm)	18.382	0.1186	11394	9681.049	na
51 V	16.46	1.102	37.04	25.09	116±8
52 Cr	104.8	28.52	81.39	272.5	114±5
55 Mn	183.7	16.34	114103*	229650*	na
56 Fe	1914	569.4	252188	219811	114±6
59 Co	5.624	3.177	162.1	143.4	114±5
60 Ni	38.98	16.06	108.2	185.7	113±3
63 Cu	121.6	24.87	10865	13467	108±1
66 Zn	172.2	644.7	72504	126816	96±9
75 As	7.045	8.855	159.7	24.43	105±6
75 As	7.84	10.1	160	25.94	103±6
78 Se	29.08	51.2	50.68	83.8	95±14
78 Se	27.4	38.5	45.78	72.68	94±13
95 Mo	24.12	34.2	3280	1589	118±4
107 Ag	0.877	2.847	8.612	10.78	100±10
111 Cd	4.537	5.579	11.33	7.471	110±5
137 Ba	99.94	7.978	345.6	888.7	111±12
201 Hg	<DL	12.72	29.19	27.06	107±4
205 Tl	<DL	1017	1739	2038	97±5
208 Pb	17.49	6.892	24	55.4	112±4
232 Th	4.564	2.462	5.548	4.047	113±6
238 U	5.355	2.142	4.794	3.238	115±5

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

Conclusions

- 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

1. Filipiak-Szok et al. Journal of Trace Elements in Medicine and Biology 30, 2015, 54–58

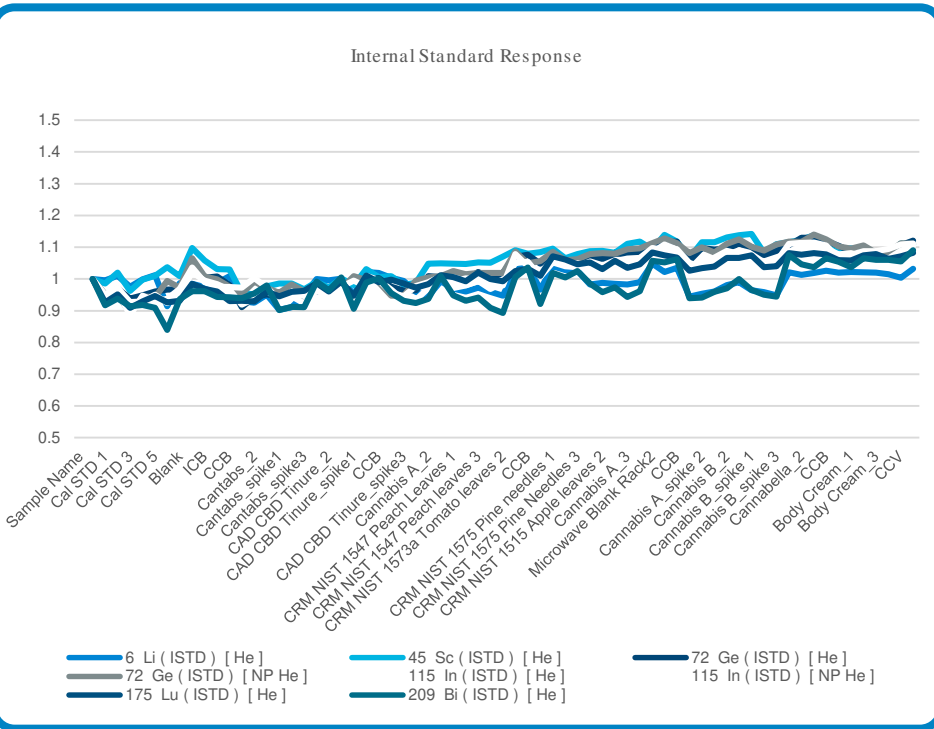


Figure 3. ISTD Stability

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.