CANNABIS APPLICATION

Heavy Metal Analysis Workflow Tools

- ICP-MS Analysis of Heavy Metals in Cannabis sativa
- Metal Certified Reference Material Mixes

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Application Note

ICP-MS Analysis of Heavy Metals in *Cannabis sativa*

Utilization of US State Specific TraceCERT® Heavy Metal CRM Mixes for the Analysis of As, Cd, Hg and Pb

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Introduction

Hemp and cannabis are strains of the *Cannabis sativa* plant. They are known to accumulate heavy metals such as lead, cadmium, arsenic, mercury, chromium, nickel, manganese and cobalt in their roots, shoots, buds and seeds. Due to this ability, hemp has been used for the remediation of contaminated soil (phytoremediation and phytoextraction). On the other hand, this inclination can hinder the use of hemp in the food or medical industry. As a consequence, all hemp material used in either food or pharma products needs to be tested regarding its heavy metal content.

As of February 2020, Canada and 24 US States have issued regulations for the testing of heavy metal content in cannabis, and all have provided limits for arsenic, cadmium, mercury and lead. In addition, four states set limits for one or more of these metals: chromium, barium, silver, selenium, antimony, copper, nickel and zinc.

This application describes the analysis of arsenic, cadmium, mercury and lead (often referred to as the "big four") by inductively coupled plasma coupled to mass spectrometry (ICP-MS) in three different *Cannabis sativa* varieties, all classified as industrial hemp:

- Hemp buds "Felina", CBD/CBG content 3.8/0.2%*
- Hemp buds "Santhica", CBD/CBG content 3.9/0.2%*
- Hemp buds "Finola", CBD/CBG content 3.8/0.2%*

*CBD/CBG information provided by manufacturer

Sample Preparation

Sample Milling: One hemp bud (approximately 2.5 g) was placed in a 50 mL stainless steel milling beaker equipped with a 25 mm stainless steel milling ball. The beaker was then mounted to a cryo ball mill cooled with liquid nitrogen; two grinding cycles of 90 s and 30 s were performed, resulting in a sample with particles ≤100 µm. 2 mL zirconia milling beakers with 15 mm zirconia milling balls could also be used as an alternative, though throughput will be decreased.

Preparation of Standard Solutions

Indium Standard Solution (internal standard): 3 mL nitric acid 60% and 1000 µL of indium ICP standard (1000 mg/L) were pipetted into a 100 mL volumetric quartz flask. Subsequently the flask was filled to mark with ultrapure water to obtain a final concentration of 10 µg/mL.

Blank Solution: 3 mL nitric acid 60% and 1 mL hydrogen peroxide 31% were pipetted into a 15 mL quartz microwave digestion vial and digested using a microwave digestion system (for conditions see section below). After completion of digestion the solution was quantitatively transferred into a 50 mL polypropylene tube, combined with 50 µL of indium standard solution and filled up to 50 mL with ultrapure water.

Sample Solution: 50 (±1) mg of ground sample was weighed into a 15 mL microwave quartz vial and after addition of 3 mL nitric acid 60% and 1 mL hydrogen peroxide 31% the sample was digested using a microwave digestion system. After digestion was completed, the obtained solution was quantitatively transferred into a 50 mL polypropylene tube, combined with 50 µL of indium standard solution and filled up to 50 mL with ultrapure water.

Standard Addition Solutions (calibration): See section "sample solution". In addition, a volume of 10 to 200 µL of Supelco® Heavy Metal Mixes III, IV, V, VI, VII or VIII was added prior to microwave digestion. Three addition solutions were prepared for every hemp sample.

Sample Digestion

Digestion Conditions (MLS turboWAVE)	
Digestion Program	Nitric acid digestion at 280 °C
Microwave Vial	Quartz glass
Basic Load	110 mL ultrapure water and 5 mL nitric acid or 115 mL ultrapure water
Charging Pressure	40 bar
Deflation Rate	5 bar/min (from T < 80 °C)
Vessel Cooling	Yes (> 40 °C)

ICP-MS Analysis

Operating Conditions (Thermo Scientific Element 2)	
Plasma Output	approx. 1300 W
Plasma Gas Flow	approx. 16 L/min
Assist/nebulizer Gas Flow	approx. 1 L/min
Sample Delivery	Peristaltic pump (or equivalent), approx. 1 mL/min
Nebulizer	quartz spray chamber / Meinhardt nebulizer
Mass Resolution	4000 + 10000
Calibration	Standard addition
The analysis is performed in the sequence: Blank, sample 1 – x, additions.	

Experimental Results

	Hemp Felina		Hemp Santica		Hemp Finola	
Element	"Heavy Metal Mix III (ppm)"	"Heavy Metal Mix IV (ppm)"	"Heavy Metal Mix V (ppm)"	"Heavy Metal Mix VI (ppm)"	"Heavy Metal Mix VII (ppm)"	"Heavy Metal Mix VIII (ppm)"
As	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Cd	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Hg	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Pb	0.5	0.5	0.4	0.4	0.7	0.7

Conclusion

Using the conditions described above, final results were consistent and revealed a Cd and Hg concentration of < 0.1 ppm for all hemp samples. Among the three varieties of hemp tested, we found that arsenic, cadmium and mercury were within specified state ranges, whereas lead content in the Felina and Santica hemp varieties was above state levels for NY, and Finola was above state levels for AR, CA, MA, RI, MI, MO and NY, respectively. This indicates the crucial need to monitor levels of heavy metals in hemp and cannabis samples to ensure overall product safety and quality.

Disclaimer:

Notwithstanding recent changes in certain state laws, federal law remains unchanged and the cultivation, possession, and/or sale of marijuana and related cannabis products continues to be illegal under federal law. In addition, the distribution of marijuana to minors under the age of 21 years remains illegal under state law.

Featured Products

Description	Cat. No.
Ultrapure water from Milli-Q® IQ 7003/05/10/15 system, or bottled water	**101262**
Nitric acid 60% Ultrapur	**101518**
Hydrogen peroxide 31% Ultrapur	**106097**
Indium single element standard for ICP Certipur® 1000 mg/L	**170324**
Heavy metal mix III TraceCERT® acc. to Colorado state regulation (As, Cd, Hg, Pb)	**94772**
Heavy metal mix IV TraceCERT® acc. to Nevada and Washington state regulation (As, Cd, Hg, Pb)	**95094**
Heavy metal mix V TraceCERT® acc. to Connecticut and New Mexico state regulation (As, Cd, Hg, Pb)	**95117**
Heavy metal mix VI TraceCERT® acc. to Vermont state regulation (As, Cd, Hg, Pb)	**04295**
Heavy metal mix VII TraceCERT® acc. to Pennsylvania state regulation (As, Cd, Hg, Pb)	**03056**
Heavy metal mix VIII TraceCERT® acc. to New Hampshire state regulation (As, Cd, Hg, Pb)	**95562**
Centrifuge tubes polypropylene, 50 mL	**T2193**
Laboratory glass bottles, 100 mL	**Z232173**

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Metal Certified Reference Material Mixes

Cannabis Testing

It is critical to accurately monitor levels of elemental impurities in cannabis products, both to ensure regulatory compliance—and more importantly—to ensure consumer and patient safety. Trace levels of heavy metals such as arsenic, cadmium, mercury and lead may accumulate in plant material through uptake from soil and, more commonly, may be introduced through the use of certain fertilizers.

With a new line of Supelco® heavy metal mixes, spend less time preparing standards, leaving more time to help solve your customers' challenges. These mixes are each tailored to US state specific regulations and offer concentrations that match your specific regulatory bodies' action and reporting limits. And with certification under ISO/IEC 17025 and ISO 17034 for all *Trace***CERT**® mixes, you can be sure sure that you have the most accurate, consistent and stable certified reference materials available.

Unique Features

- Solutions are specifically designed to meet the state specific heavy metal limits
- Concentration ratios correspond to state requirements: easily bracket action levels
- Minimize handling steps and reduce error sources
- All metal solutions are Certified Reference Materials (CRMs), produced under the double accreditation of ISO/IEC 17025 and ISO 17034
- Innovative packaging format ensures certified concentrations during storage
- Comprehensive certificate of analysis provided for each batch

Our high standards match yours.

Please check our website for more information:
SigmaAldrich.com/cannabis

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Product List

Cat. No.	Description	Certified Metal Concentrations (mg/L)			
All mixes are in 12% HNO_3. Unit Volume: 100 mL		As	Cd	Hg	Pb
94846	Heavy metal mix I, *Trace*CERT®, acc. to California state regulation (inhalable cannabis and cannabis products) and Arkansas state regulation	20	20	10	50
94794	Heavy metal mix II, *Trace*CERT®, acc. to California state regulation for other cannabis & cannabis products	15	5	30	5
94772	Heavy metal mix III, *Trace*CERT®, acc. to Colorado state regulation	40	40	20	100
95094	Heavy metal mix IV, *Trace*CERT®, acc. to Nevada and Washington state regulation	200	82	40	120
95117	Heavy metal mix V, *Trace*CERT®, acc. to Connecticut, and New Mexico state regulation	14	9	29	29
04295	Heavy metal mix VI, *Trace*CERT®, acc. to Vermont state regulation	100	41	20	100
03056	Heavy metal mix VII, *Trace*CERT®, acc. to Pennsylvania state regulation	15	3	5	10
95562	Heavy metal mix VIII, *Trace*CERT®, acc. to New Hampshire state regulation	5	3	9	9

Additional Information

So far, 10 U.S. states have legalized cannabis for recreational use, and 33 for medical use. Australia (medical), Uruguay and Canada (medical & recreational) also legalized the use of cannabis. Alongside tests for cannabinoinds, terpenes, residual solvents, pesticides and microbiological contamination, it is important to test for harmful heavy metals such as Cd, As, Hg and Pb.

When processing cannabis into concentrations and extracts, monitoring for elemental impurities becomes even more important. This is because the common industrial techniques, such as supercritical fluid and hydrocarbon extraction, have the potential to concentrate not only the compounds of interest—cannabinoids and/or terpenes—but also impurities such as heavy metals.

When preparing standards for calibration and QC of the ICP instruments used to monitor elemental impurities, labs typically need to accurately prepare stock solutions consisting of several different standards. This involves multiple handling steps with hazardous acids and introduces the potential for increased error/uncertainty with each transfer and dilution.

Not anymore: with our comprehensive portfolio of Supelco® certified reference material mixes for cannabis samples, your heavy metal analysis will be accurate, and also meet your specific regulatory compliance needs.

To get an overview on our cannabis testing offer, visit us at **SigmaAldrich.com/cannabis**

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