# The Metals in Cannabis Method with first action status by AOAC, the road to get there...

Presented by: Jenny Nelson and Craig Jones-Agilent Sam Heckle and Leanne Anderson-CEM Hosted-PJLA, Tracy Szerszen, President



Thursday, March 24, 2022 1:00-2:00 PM EDT





#### **Presentation Overview**

PJLA



Tracy Szerszen President Perry Johnson Laboratory Accreditation (PJLA) Introduction of the new AOAC Method 2021.03-Heavy Metals (Cadmium, Arsenic, Lead, and Mercury) and additional elements in a variety of Cannabis and Cannabis Derived Products

Challenges, Research, Studies to Develop Method

**Questions & Answers** 

### ISO/IEC 17025:2017 - 7.2 Selection Verification and Validation of Methods

- Methods shall be appropriate for the test
- Methods shall be up to date and available to personnel
- Labs shall use the latest version of the method unless its not appropriate to do so
- It is recommended to select methods that are published either in international, regional or national standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer of the equipment
- In-house developed methods are also accepted
- Additional criteria required for validation of in-house methods

#### Possible Future State Regulator Expectations

- Laboratories may be required to use nationally recognized test methods available in the industry over time
  - Equal playing field between all labs
  - Consistent and reliable results
  - Possibly less review of data packages and monitoring by states
- As an accreditation body, PJLA is starting to see more labs in the industry participate in method studies and prepare to establish protocols in accordance with available methods
- We recommend all cannabis labs monitor industry changes



# Webinar Housekeeping

- This webinar will be recorded
- All PJLA webinars are made available on our website & YouTube channel
  - https://www.pjlabs.com/training/

pjla-webinars/past-webinars

- All attendees are muted
- Please utilize the question tool bar to submit questions
  - To be answered at the end of presentation

# Presenters

Jenny Nelson & Craig Jones | Agilent Technologies, Inc.

- ▶ 5301 Stevens Creek Blvd, Santa Clara CA 95051, U.S.
- Sam Heckle & Leanne Anderson | CEM, Inc.
  - ▶ 3100 Smith Farm Rd, Matthews, NC, 28104, U.S.



#### Official AOAC method: Heavy Metals in Cannabis

Jenny Nelson, Craig Jones, Sam Heckle, and Leanne Anderson







DE74778354

#### AOAC First Action Method

https://www.aoac.org/news/aoac-approval-granted-to-agilent-for-method-of-metals-analysis-in-cannabis/



# of Metals Analysis in Cannabis

#### November 10, 2021

Rockville, Maryland, November 10, 2021 - AOAC INTERNATIONAL announced that Agilent has received approval of an analytical method for the determination of heavy metals in cannabis. The method, OMA 2021.03, jointly developed by Agilent and CEM, which uses inductively coupled plasma mass spectrometry (ICP-MS) for the guantitation of arsenic (As), cadmium (Cd), mercury (Hg), lead (Pb), and other elements, was recognized by AOAC, an international organization that helps advance the safety of food and related products through its involvement in the development and standardization of analytical methods.

Legalization of medicinal and recreational marijuana in many regions in the United States and Canada has dramatically increased the demand for cannabis, hemp, and related products. Like food, which is regulated under U.S. Food and Drug Administration (FDA) methods and guidelines, and pharmaceuticals, which adhere to United States Pharmacopeia (USP) regulation, cannabis is a consumer product that requires testing to ensure its safety. Among the necessary tests is analysis of heavy metals, which may be toxic if ingested or inhaled. However, states and provinces have their own guidance and set their own regulations due to a lack of federal oversight, making it difficult to guarantee consistent product quality.

Related Links
Cannabis SMPRs
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#### **AOAC First Action Method**

#### http://www.eoma.aoac.org/methods/info.asp?ID=52491





# **Current Regulations**

	State	California	California	Missouri	Missouri	Massachusetts	Massachusetts	Rhode Island	Rhode Island	Colorado	Colorado	Colorado	Arkansas	lowa	Minnesota	Maryland	Maryland	Oklahoma	Pennsylvania	Alaska	Connecticut	Florida	Illinois	Louisiana	Maine		Maine	Michigan	Michigan	Michigan	Michigan	Montana	Nevada	New Mexico	New York	North Dakota	Ohio	Washington State	West Virginia
		CA	CAN	10	NOI	MAI	MA	RI	RI	CO	CO	CO	AR I	Α	MN	MD	MD	ОК	PA	AK	СТ	FL	IL	LA	ME	Μ	IE M	IE MI	MI	MI	MI	MT	NV	NM	NY	ND	ОН	WA	WV
Analyte	Units	ppm	ppm p	pm p	opm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm p	pm	ppm	µg/kg Bw/day	ppm	ppm	ppm		µg/kg Bw/day			ppm	ppm	рр	om pp	m ppm	ppm	ppm	ppm	µg/day	ppm	µg/kg Bw/day	ppm	ppm	ppm	µg/day	
Arsenic		0.2	1.5 C	).2	1.5	0.2	1.5	0.2	1.5	0.2	3	1.5	0.2 1	5	1.5	0.14	0.4	0.4	0.4		0.14			10	0.2	1.	.5 1	l <mark>1.5</mark>	0.2	0.14	0.98	10	2	0.14	0.2	0.4	0.14	10	
Cadmium		0.2	0.5	).2 (	0.5	0.2	0.5	0.2	0.5	0.2	3	0.5	0.2 0	).3	0.3	0.09	0.4	0.44	0.3		0.09			<mark>4.1</mark>	0.2	0.	.5 5	5 <mark>0.5</mark>	0.2	0.09	0.63	4.1	0.82	0.09	0.2	0.3	0.09	4.1	
Lead		0.5	0.5	).5 (	0.5	0.5	1/1 0	0.5	10	0.5	10	1	0.5	1	1	0.29	1	1	1		0.29			10	0.5	0.5 1	5/ 0 1	0 0.5	0.5	0.29	2.03	6	1.2	0.29	0.2	1	0.29	6	
Mercury		0.1	3 (	).1	3	0.1	1.5	0.1	1.5	0.1	1	1.5	0.1 0	).5	0.5	0.29	0.2	0.2	0.2		0.29			2	0.1	3/ 5	'1. 5 1	. 3	0.1	0.29	2.03	2	0.4	0.29	0.2	0.2	0.29	2	
Chromium			C	).6	2												0.6											2	0.6						2				
Barium																	60																		-				
Silver																	1.4																		-				
Selenium																	26																		-				
Antimony																	-																		2				
Copper																	-																		2				
Nickel																	-																		2				
Zinc																	-																		20				

#### inhalation

generic/final product

<mark>Oral</mark>

Topical



## Spotlight

#### Michigan- iAs, Cr, Ni, Cu

#### Table 6. Heavy Metal Action Limits.

		Action Limit (ppm)	
	Inhaled Marijuana Flower and Inhalable Compound Concentrate	Inhaled Marijuana Concentrates	Other Marijuana Products
Heavy metal	Products		
Lead	1.00	0.500	0.500
Inorganic Arsenic	0.400	0.200	1.50
Mercury	0.200	0.100	3.00
Cadmium	0.400	0.200	0.500
Total Chromium	1.20	0.600	2.00
Nickel*	1.00	0.500	N/A
Copper*	N/A	3.00+	N/A

+ Copper is required for vaping products only.



# Samples





# Samples- Oral

Sample Type	Sample
Pill/Capsule	"Full Spectrum" softgels
	(capsules)
Tincture	Tincture- full spectrum
	made with distillate +
	coconut oil carrier
	Tincture- made with isolate
	+ coconut oil carrier
Edibles	
	CBD Coffee Grounds
High Fat	Hemp Butter
High Fat	Hemp Seed Oil
High Protein	CBD Beef Jerky
High Carbohydrate	"chews" hard candy
High Carbohydrate	CBD Pineapple Juice/Drink





# **Samples-Topical**

Sample Type	Sample
Lotion	full spectrum balm made with distillate
	Pain relief cream made
	with isolate
	CBD Balm
Oil	CBD Oil- Topical
Soap or	Hemp Soap
Patch	





# Samples-Inhaled

Sample	Sample
Туре	
Plant material	flower / biomass
Vape Pen/ cartridges	CBD Vape Oil
Shatter	Hemp Isolate Extract





# Samples-Manufacturing

Sample Type	Sample
	Raw biomass with high metals results
Biomass	Spent Biomass
Crude	CBD raw crude (different suppliers)
Extract	CBD Crude Extract
	CBD Distillate
Refined Extract	CBD isolate





#### **Microwave Digestion Equipment**

#### MARS 6 with iWave temperature sensor technology

- Contactless temperature measurement
- Sensors to detect temperature, vessel type, and number of vessels
- Preprogrammed One Touch methods
- Custom methods

#### MARSXpress Plus vessels

- 110 mL TFM Teflon vessel
- Simple three-piece design







#### **Microwave Digestion Method**

- Weigh 0.5 g of homogenized sample.
- Add 9 mL HNO $_3$  and 1 mL HCl.
- Select the Cannabis One Touch method on the MARS 6 Digestion System.
- Samples will then be digested at 210 °C for 15 minutes.
- Dilute your samples to weight/volume, and they're ready to analyze!



### Instrumentation

- Agilent 7850 ICP-MS, standard Ultra High Matrix Introduction (UHMI) system
  - Pre-set Settings 4x uHMI
- Agilent SPS4 autosampler
- Micromist concentric nebulizer, quartz spray chamber, and quartz torch (2.5 mm id)
- Ni plated/Pt tipped sampler cones
- KED mode using He





# **Example ISTD Recovery**



#### Example CCV Recovery





### Example Calibration curves As, Cd, Pb, Hg

All curves show excellent linearity across the calibration range





### Example Calibration curves Optional Elements- V, Cr, Mn, Co, Ni

All curves show excellent linearity across the calibration range





# All the data from the SLV can be found in the AOAC method document

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#### Heavy Metals in a Variety of Cannabis and Cannabis-Derived Products

Analyte	Metals, Metals/Arsenic, Metals/Calcium, Metals/Lead, Elemental Analysis/Mercury
Analytical Technique	Inductively Coupled Plasma–Mass Spectrometry
Equipment	Mass Spectrometer/Inductively Coupled Plasma Mass Spectrometer
Approved By	AOAC
Method Number	2021.03

You must login to access this method. After login in the popup window, click the following button.

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#### **New Productivity Tools**

#### Automated half mass correction

- Doubly charged ions appear at half their mass and are not polyatomic interferences
- Unique automated correction significantly reduces the risk of erroneous results



ICP-MS MassHunter IntelliQuant heat map showing complete major and trace element content of a surface water sample.



## The Need for Half-Mass Measurement

Some samples contain high and variable levels of Rare Earth Elements (REE's)

REE's have relatively low second ionization potentials meaning they are able to form ions with a 2<sup>+</sup> charge

Even at only 1% formation if concentrations are high the interferences can be significant enough

Quadrupole mass filters separate ions based upon their mass-to-charge ratio (m/z) any ion possessing a 2<sup>+</sup> charge will appear at half its actual mass

Example: Sm has an isotope at  $m/_z 150$  but if a proportion of that isotope exists as Sm<sup>++</sup> then 150/2 = 75 - this would interfere with <sup>75</sup>As measurement



#### **REE++ Correction**

board		
	MassHunter Method Wizard <b>REE++ Correction</b> Select whether you process this batch by either "REE++ Correction" or "Acq. Defined".	
Mainframe	<ul> <li>REE++ Correction</li> <li>Use the interference correction automatically calculated by REE++ isotopes from half-mass acquisition data, for As, Se or Zn.</li> </ul>	Quadrupole
Maintenance Feedback		
r Maintenance Counters	○ Acq. Defined	Instrum
inge Foreline Pump Oil	Use the conventional interference correction method from the acquisition data.	D X Pov Plas
:uum ON Days: 0/180		Foreline Reset Turbo
an Sampling Cone		Solution
utions Measured: 0/200		
	To continue, click Next.	
	Help < Back Next > Finish Cancel	
	ii.	L



#### Periodic Table Of Elements Template





#### Intelliquant Cannabis Flower





#### Intelliquant









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Mass

Agilent

### NEXT-ASTM



In 2017, globally recognized industry leaders came to ASTM International with a shared goal — to develop standards addressing quality and safety in the burgeoning cannabis and hemp industry.



#### ASTM Ballot D37.03 (21-02)

MyASTM / Membership / MyCommittees / MyBallots

#### ASTM Ballot D37.03 (21-02)

**RETURN TO MYBALLOTS** 

1

Thank you for your vote. If you would like to change your vote, please email standard@astm.org before the ballot close date.



#### Ballot Number: D37.03 (21-02)

Item No. Sub No. Item

Test Method for Analysis of Multiple Elements in Cannabis Matrices by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) WK74576 PDF (833K) (REFERENCE Z0748Z) TECHNICAL CONTACT: Jenny Nelson jenny.nelson@agilent.com



#### Issues that we hear from Cannabis Labs when being audited

- Do I have to use a matrix sample for you Method Blank?
  - You will never find a Cannabis matrix blank for ICP-MS that is free of metals.
  - You have to use acid as your method blank.
- Can I prepare my cannabis samples on a hot block or is microwave digestion required?
  - Closed vessel microwave digestion is required to reach the necessary temperature of 210 °C and to prevent loss of volatile analytes.



#### Issues that we hear from Cannabis Labs when being audited

- Can I digest my cannabis samples at temperatures lower than 210 °C, for example 190 °C?
  - No, 210 °C is required to achieve a complete digestion of some of the tougher cannabis derived products like concentrates and some foods.
- What's the least amount of acid I can use to digest cannabis samples?
  - 5 mL of mineral acid is the bare minimum volume necessary to achieve a complete digestion.



#### Thank you! Any questions?

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Join us for our Next Webinar Thursday, March 31, 2022 -1:00pm EST

A Look at ISO/IEC 17025:2017 - Section 7.8 "Reporting of Results"



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# Thank You!