

Inorganic & Organic Certified Reference Materials



Challenges and Economics of Analytical Testing and Reference Material Production in New Markets and a Sub-PPB World

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A PPB and sub-PPB World

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- Analysis is changing to PPB and sub-PPB levels
- Battle between Public Health, Good Science and Economics
 - Some levels set not because they are safe but that is what the industry can afford to analyze

Markets & Industries

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Challenges in the Traditional Analytical Community

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Challenges in the Cannabis Community

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Environmental











The Case of Changing Regulations vs Economics: Environmental

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Heavy Metal Limits - Adults

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	FDA Bottled Water	EPA Drinking water	WHO Drinking water	EPA	WHO/ JECFA	ATSDR
	Action Level (ug/L)	Action Level MDL (ug/L)	Action Level (ug/L)	Oral Rfd (ug/day)	PTDI (ug/day)	Oral MRL (ug/day)
As (Total)	10	-	10	21 (chronic)	150	21 (chronic)
As (Inorg)	-	10	-	-	-	-
Cd	5	5	3	70	70	14
Hg (Total)	2	2		-	-	-
Hg (MeHg)	-	-	-	7	16	21
Hg (Inorg)	-	-	6	-	-	-
Pb	5	15	10	-	250	-

Levels at ug or at ppb levels



Trace Analysis



In Pursuit of True Values



- Accuracy & Precision
 - Accuracy = How close to you get to True Value
 - Instrument Bias, Contamination, Error
 - Precision = How close are your results to one another (RSD%)
 - Instrument Variation, Operator proficiency, Error





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Quality System

Clear to Follow - Understood by All Training & Testing Statistics & Data Addresses Problems, Error or Bias Standards

Lab Components

Samples & Prep Chemicals & Solvents Lab apparatus Glass/Plastic Ware & Storage In-house Standard Use & Preparation Instrumentation & Analysis



Lab Environment & Human Component



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Pharmaceuticals & Nutraceuticals



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The Case of Regulations vs Technology, Laboratory & Economics: USP <232>



USP <231> written over a century ago, depended on colorimetric methods and

precipitation reactions





USP <232>; 2018 depends on quantitative instrument methods



Comparison <231> & <232>

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	USP <231>	USP <232>			
History	>100 years old	started in 2008, implementation 2018			
Analysis Type	Colorimetric	Instrumental			
Flomonte	Only metals precipitated by sulfide ion	All instrumentally detectable heavy metals			
Elements	Pb, Hg, Bi, As, Sb, Sn, Cd, Ag, Cu, Mo	Multiple elements and speciation of Hg, As if over limit			
Results	Qualitative	Quantitative			
Preparation	Ashing for solids & Oils	Modern sample prep			
Limits	Total Heavy metals based on % in monograph	Set Limits depending on: Element, Form & Dosage			
	%	PPM & PPB levels			

Why was the industry slow to adapt to the new method changes?







Laboratory Challenges

- Sample & Collection: Glass can absorb Hg
- Stability: Change State
- Contamination: Plastics contain Target Analytes

	Economic Challenge			Material		Total # of Elements	Total ppm	Major Impurities
				PS		8	4	Na, Ti, Al
	 Specialized technologies Unavailable or High Cos 			Teflon-TFE		24	19	Ca, Pb, Fe, Cu
				LDPE		18	23	Ca, Ti, Zn
					PC		85	Cl, Br, Al
					PMP		178	Ca, Mg, Zn
				Teflor	ı-FEP	25	241	K, Ca, Mg
Arsenic Species		LD ₅₀ (mg/kg)	Description		O tr	Drice	C C	Total 6
Arsenite	As (III)	14	Description		Q.ly	PICE	€	10(a) €
Arsenate	As (V)	20	Arsenobetaine		5 gr			6,000.00
Arsine	AsH3	3	Arsenobetaine	1 gr				1.200,00
Monomethylarsonic Acid	MMA	700 - 1800	Arsenocholine		1 gr			1.200,00
Dimethylarsinic Acid	DMA	700 - 2600						
Arsenocholine		> 10000		αC	nan	enges	ior ac	curacy α
Arsenobetaine		> 10000						
	r P							

Cannabis

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Case Study: New market





10 years ago:

- Recreational cannabis emerged in CO
- Industry was suddenly flooded by pop-up labs
- Limited budget
 - Simple instruments
 - LOD & LOQ problems
 - Using FID for ppb
 - Instrumentation limitations
 - LCMS pesticides being tested GCMS or GCFID
 - Inexperienced analysts
 - Possibly complex analytical sample preparation to accommodate equipment
 - Derivatization
- No guidance to laboratories from an authority

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Key Problem: Lack of Consensus





- Garbage in Garbage out
- Lack key components hurt labs with good analytical procedures

Standards Agencies now writing regulation; not government



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Official Methods and Recommended Practices of the AOCS

All Cannabis Issues are Economic Issues



Retail Spice, Cannabis & Commodities (\$/lb.)



Lots of Commonality in the Challenges





Analysis Challenges & Issues: Methodology

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Environmental (Established)

- Well established QMS
- Hundreds of Methods
- Slow to adapt changes
- Based on legal limits & Official Methods
- Increasingly challenging targets and limits



Cannabis

(New Industry)

- Limited QMS adoption
- No established methods to date
- Constantly changing
- Limited legality and regulatory adoption
- No consensus on limits or targets

Since 1974 – 3x increase in compounds, and decrease in limits

No consensus on limits or numbers of target pesticides

Analysis Challenges & Issues: Standards

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Environmental (Established)

- Well established
- Hundreds of Standards, many labs require Certified Standards
- Slow to adapt changes: Speciation
- Increasingly challenging targets and limits – ppb contamination, species

Cannabis

(New Industry)

- Limited standards available
- High cost
- Constantly changing
- Limited legality & transportation issues
- No consensus on limits or targets

Interested in Improving Accuracy & Precision & Reducing Error



- Visit SPEX CertiPrep's Website for our Clean Laboratory and Statistics Webinars
- Request copies of our application notes on Error and Uncertainty
- Visit LCGC to download our webinar!



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Thank You! Questions?