

Cannabis Testing and Cannabis Introduction

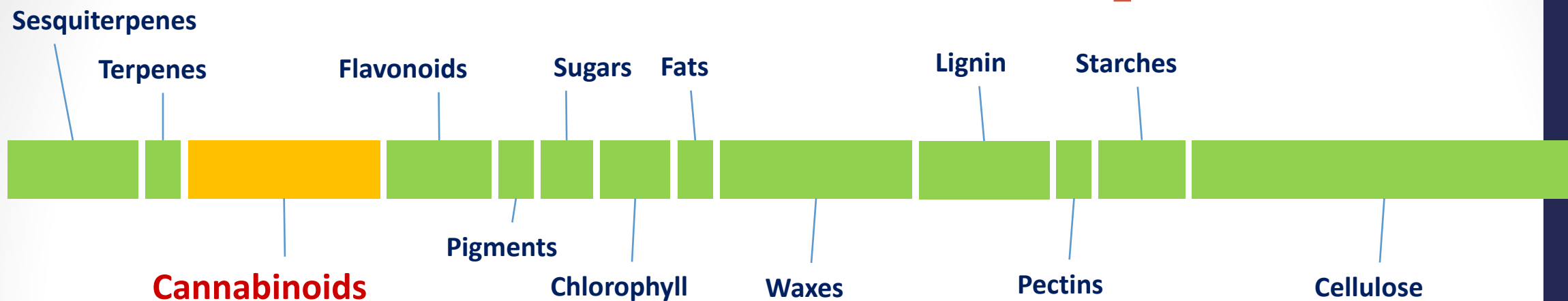
Stephen Goldman
Chief Scientific Officer

kaycha
LAB

Talking Points

- Cannabis is incredibly complex
- The regulatory environment doesn't make it any easier
- Testing Problems have difficult solutions
- What's Coming Next in inhalation testing
- Does testing, with all these problems, actually do anything?

Cannabis Inflorescence is a Complex Matrix



Cannabis Inflorescence is a Complex Matrix



***Cannabis* Plant Material**

Terpenes, Terpenoids, Flavonoids, Pigments, Sugars, Chlorophyll, Fats, Waxes, Lignin, Pectins, Starches, Cellulose



Essential Oils ('Hash oil' & resin concentrates)

Terpenes, Terpenoids, Flavonoids, Pigments, Fats, Waxes



Semi- Purified Cannabinoids

THC-A, THC, CBD-A, CBD, CBN and trace cannabinoids

Cannabis Inflorescence is a Complex Matrix



***Cannabis* Plant Material**

Terpenes, Terpenoids, Flavonoids, Pigments, Sugars, Chlorophyll, Fats, Waxes, Lignin, Pectins, Starches, Cellulose

- Hundreds of mixed individual compounds— *complex, by nature*
- Myriad potential for contaminants— *Agricultural & Industrial processes*
- Welcome to natural products research— *the matrix is unique, but the need for standardization is not!*

Cannabis Life Cycle



Cannabis Contaminants



Flower/Trim
Microorganisms
Potency
Heavy Metals
Pesticides

Concentrates
Residual Solvents
Potency
Pesticides
Heavy Metals



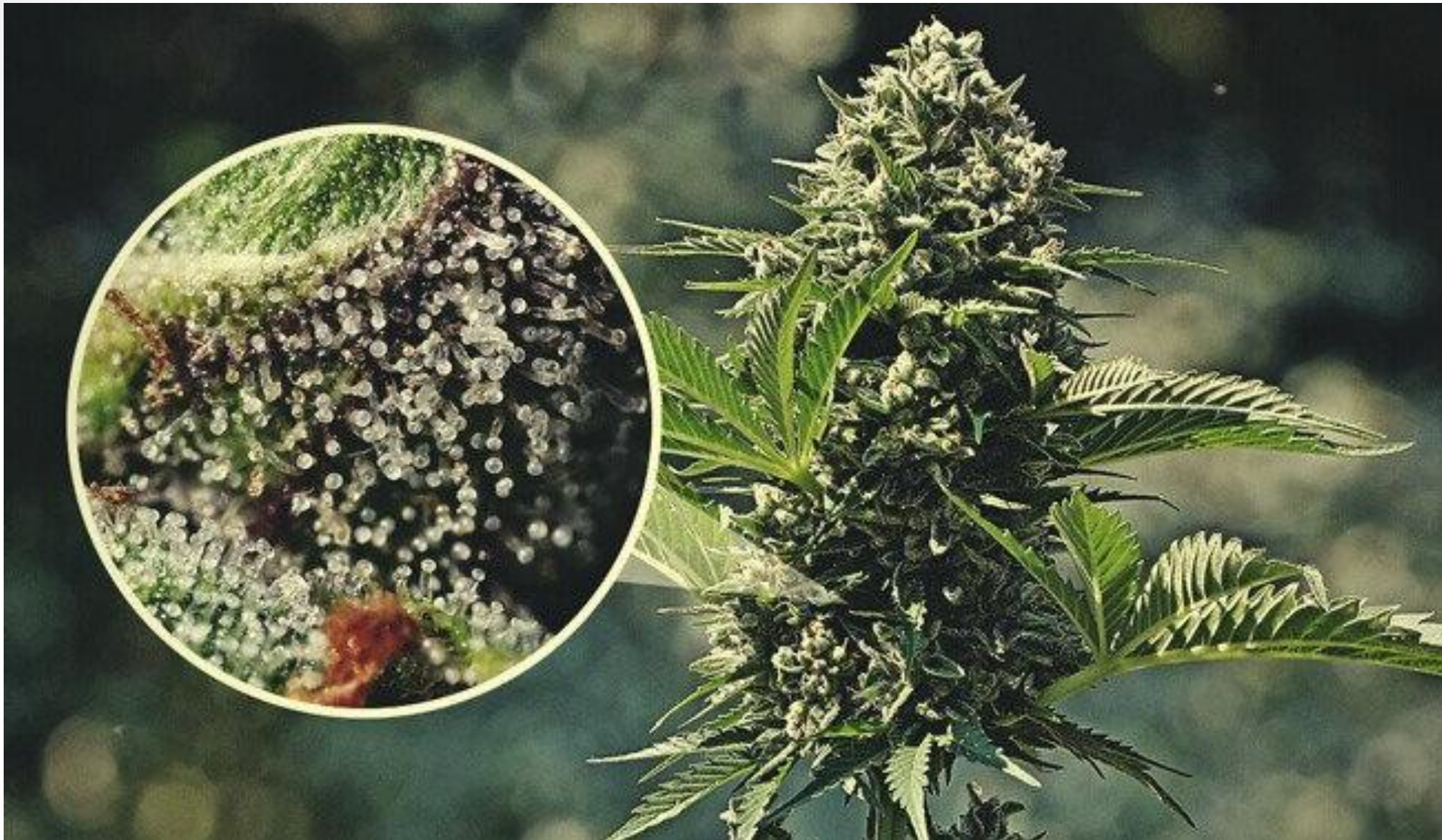
Processed Concentrates
Residual Solvents
Potency
Pesticides
Heavy Metals
API Degradation

Edibles
Potency
Homogeneity
Microorganisms





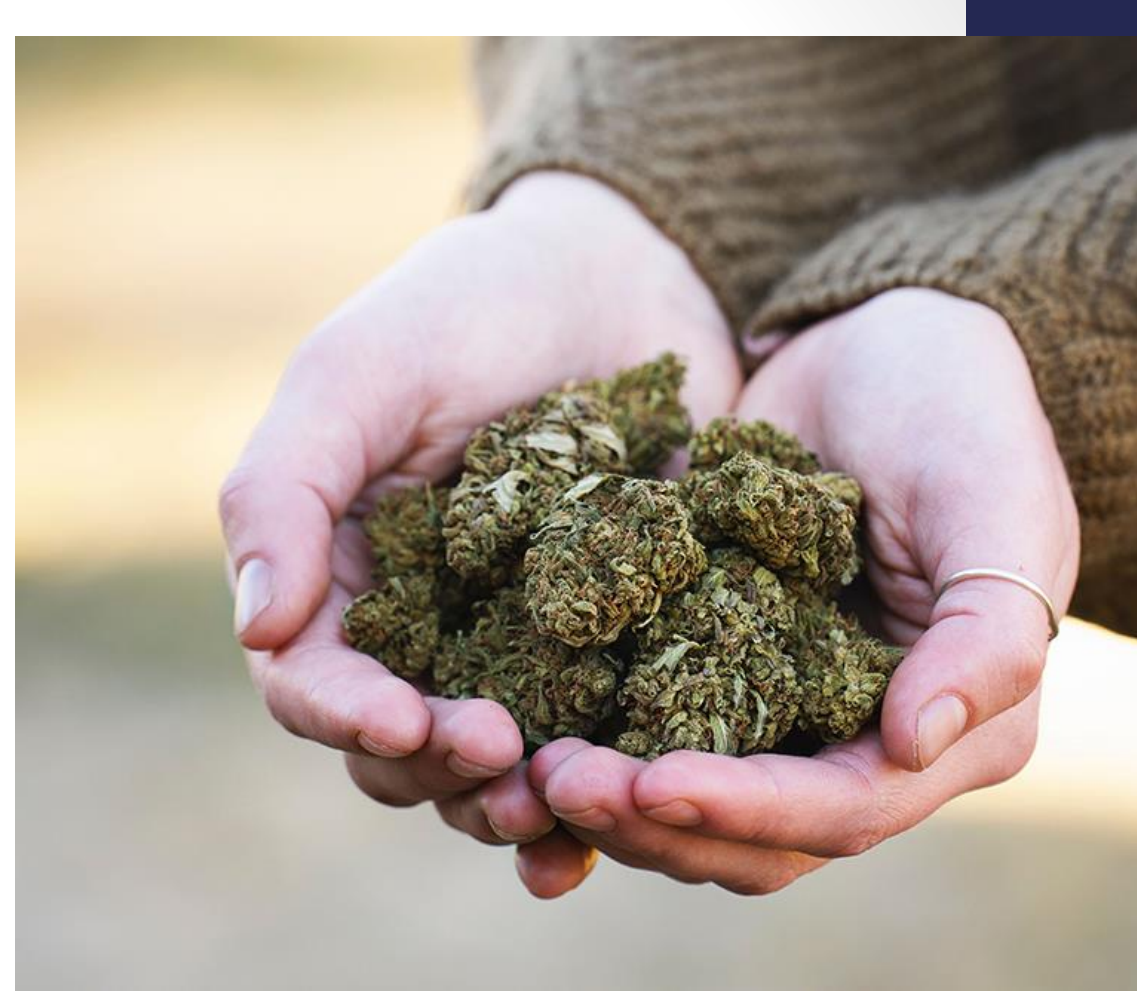
Cannabis inflorescence “Colas”



Trichomes – the good stuff

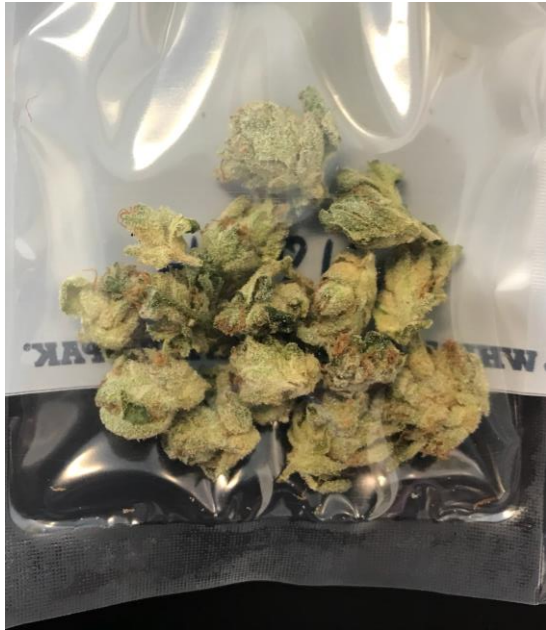


Dry/Cure



Cannabis/Hemp flower

Sold as flower, “bud” and suitable for inhalable applications



Samples submitted to the lab



Cannabis derived matrixes

- Derivates, extracts, etc
- Used for infusion manufacture, inhaled on their own
- Solvent extracted, solventless



SHATTER



BUDDER



WAX



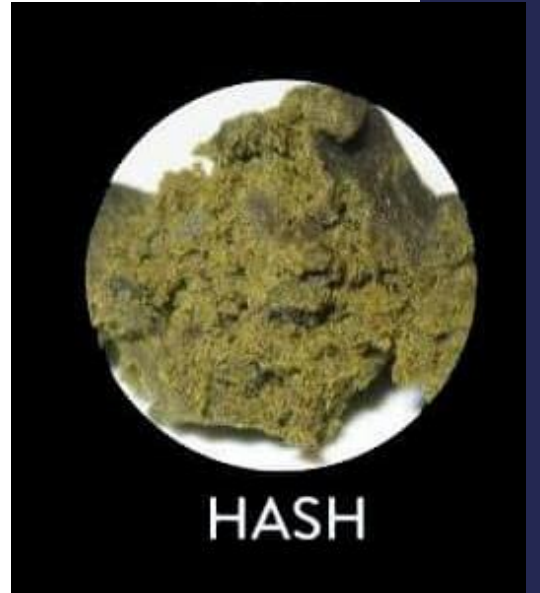
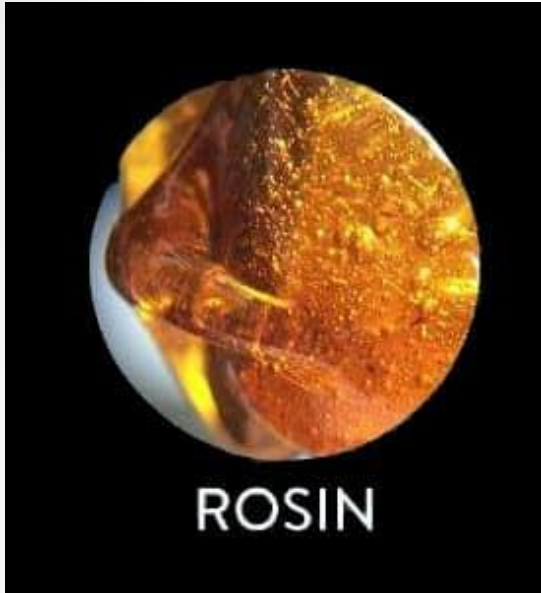
CRUMBLE



CO₂ OIL



LIVE RESIN





Sugar





Issues

- “Marijuana” remains a DEA Schedule 1 drug and is not recognized as a useable commodity, food, or drug by Federal regulators
- States must devise their own regulations for cannabis production, quality assurance, and standardization
- Sample preparation variability
- Commercial adulterants (pesticides, PGRs, synthetic cannabinoids)

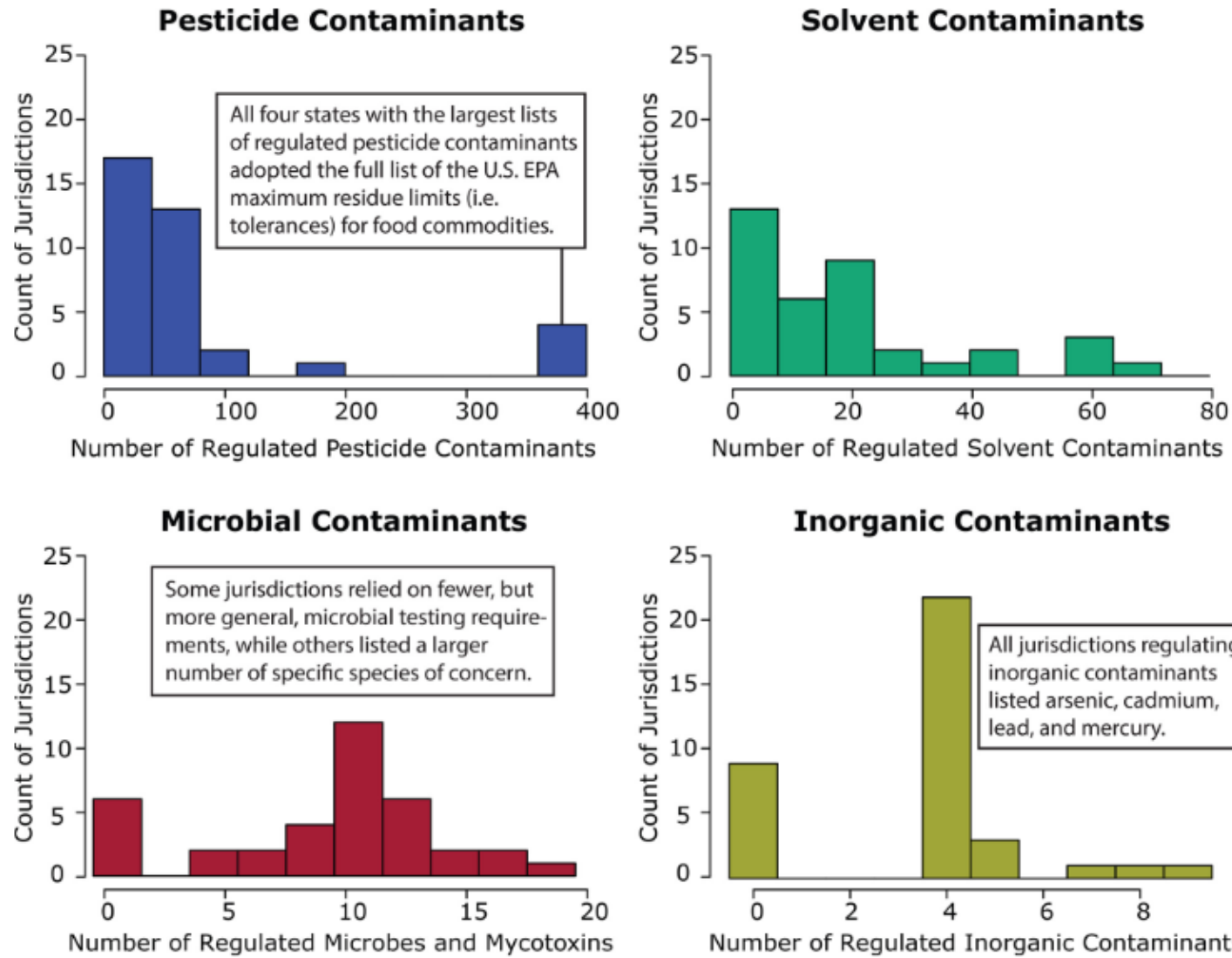
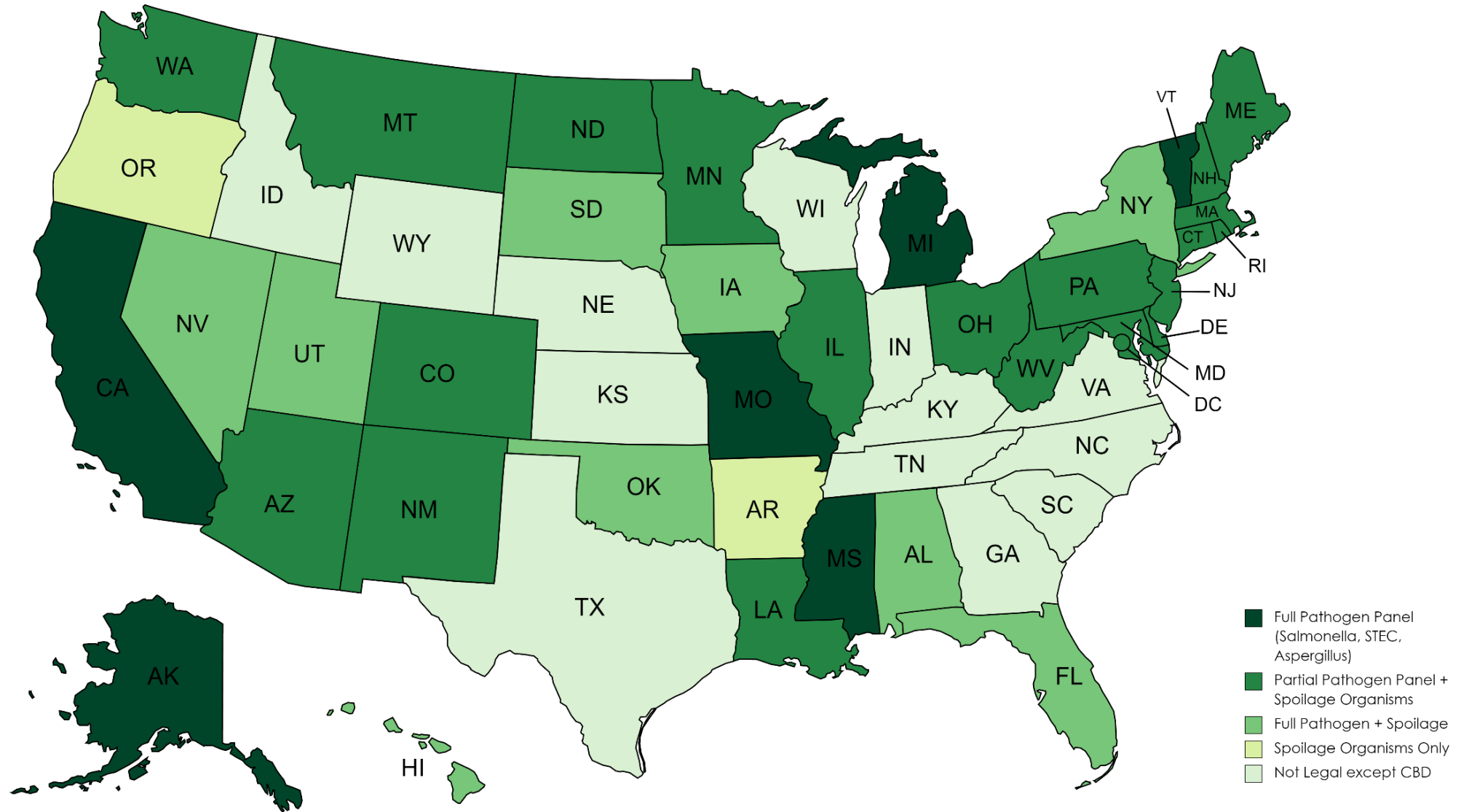


Figure 1. Histograms showing the number of listed cannabis contaminants regulated in each of the 36 legalized states and Washington, DC's regulatory documents for cannabis as of 18 May 2022. The four main categories of contamination listed are presented as separate panels. Five of the states named no individual contaminants for any category. All regulatory documents are listed in Supplemental Material, "Identified Regulatory Documents, Public Health Reports, Cannabis Testing Reports, and News Reports." Note: EPA, Environmental Protection Agency.

Regulatory Climate



Cannabis State Regulatory Outline

	<i>Salmonella</i>	STEC	<i>Aspergillus</i>	Total Aerobic Count	Total Yeast and Mold	Total Coliforms	BTGN or EB	<i>E. coli</i>	Other
Hawaii, Nevada	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
New York ¹ , Oklahoma ² , South Dakota	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
Iowa, Utah ²	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
Michigan	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Alaska ³ , California, Mississippi, Missouri, Vermont	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Illinois, Maine, Massachusetts, New Hampshire, New Mexico, North Dakota, Ohio, Rhode Island	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Colorado ² , Connecticut ² , Florida ² , Pennsylvania	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
Maryland ⁴ , New Jersey ⁴	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
Minnesota	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Louisiana, Montana	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Washington	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Delaware ²	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
District of Columbia	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Arizona	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
West Virginia ²	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
Arkansas, Oregon	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>

¹Requires testing for *Clostridium botulinum*, *Enterococcus*, *Mucor*, *Penicillium*, *Pseudomonas* and Thermophilic Actinomycetes species

²*S. aureus*, *P. aeruginosa* or *C. albicans* required for certain products

³Does not require *A. terreus* testing

⁴Requires *L. monocytogenes* testing for edible products

Standardization

- Batch size specification
- Sampling and testing frequency
- Sample preparation
- Certified reference standards
- Standard operating procedures (SOPs)
- Instrumentation
- Proficiency testing & accreditation
- Action levels for contaminants --- DATA!

Reference Methods?

- Currently no mandated methods
 - Mandated analytes
- SMPRs
 - Chemistry
 - Micro
- What other industries can we look to?

Things to consider when selecting methods

- Verification data, if available
- Sample size
- Extraction efficiency
- High background/poor pseudo matrices to choose

Sample Prep/Analysis - Chemistry

- Analytical Chemistry
 - Different prep for different analytes
 - Physical sample prep
 - Grinding the sample
 - Limited options to heat to change viscosity – leads to decarboxylation
 - Extraction solvent selection
 - Reference material availability
 - Limited sample size
 - Reference methods/Validated methods

Sample Prep/Analysis - Micro

- Microbiology & Chemistry
 - Sample size
 - Homogenization
 - Training of analysts/clients
 - Instrument manufacturer white label method quality
 - Manipulation and integrity of samples

A Look Inside the Massachusetts Cannabis Testing Controversy

Article 🕒 Sep 14, 2018 | By Alexander Beadle



A closer look at the testing facilities

ProVerde Laboratories, in addition to CDX Analytics and MCR Labs, are the only **independent testing laboratories** that are licensed for medicinal cannabis testing in the state, but the three labs do not use identical testing methodologies. The approval of both MCR Labs and CDX Analytics to test recreational cannabis samples despite using different testing methods has ignited debate over whether the current system is open to abuse by producers who can “shop around” for favorable results.

One key aspect of the debate is the use of differing methods for microbiological contamination testing. Two of the labs, **ProVerde Laboratories**, and **MCR Labs**, use a traditional testing technique known as “plating”. This involves taking samples of cannabis and loading them onto Petri dishes containing a gel-like growth medium that encourages the growth of microbes that may be present in the sample. By counting the number of microbial colonies that develop, highly-trained technicians can statistically assess the type and prevalence of different microbes present in the sample, and then compare the observed levels against the state regulations to determine if the batch should pass or fail contaminant testing.

In contrast to the other two testing facilities, **CDX Analytics** carries out its testing using an arguably more targeted approach based on polymerase chain reaction (PCR) technology. The **PCR method** works by amplifying identifying segments of DNA in the sample that are characteristic of cannabis and known microbiological contaminants. This information can then be used to calculate the extent of any contamination present.

R 1504 – Retail Marijuana Testing Program – Sampling Procedures

A. Collection of Samples

1. Sample Collection. All Samples submitted for testing pursuant to this rule must be collected by Division representatives or in accordance with the Division's sampling policy reflected in the marijuana laboratory testing reference library available at the Colorado Department of Public Health and Environment's website. This reference library may be continuously updated as new materials become available in accordance with section 25-1.5-106(3.5)(d), C.R.S..
2. Sample Selection. The Division may elect, at its sole direction, to assign Division representatives to collect Samples, or may otherwise direct Sample selection, including, but not limited to, through Division designation of a Harvest Batch or Production Batch in the Inventory Tracking System from which a Retail Marijuana Establishment shall select Samples for testing. A Retail Marijuana Establishment, its Owners and employees shall not attempt to influence the Samples selected by Division representatives. If the Division does not select the Harvest Batch or Production Batch to be tested, a Retail Marijuana Establishment must collect and submit Sample(s) that are representative of the Harvest Batch or Production Batch being tested.
3. Adulteration or Alteration Prohibited. A Licensee or its agent shall not adulterate or alter, or attempt to adulterate or alter, any Samples of Retail Marijuana, Retail Marijuana Concentrate, or Retail Marijuana Product for the purpose of circumventing contaminant testing detection limits or potency testing requirements. The Sample(s) collected and submitted for testing must be representative of the Harvest Batch or Production Batch being tested. A violation of this sub-paragraph (A)(3) shall be considered a license violation affecting public safety.
4. Timing of Samples. A Licensee shall not collect or submit Samples for testing until the Retail Marijuana, Retail Marijuana Concentrate, or Retail Marijuana Product has completed all steps required prior to Transfer to another Retail Marijuana Establishment as outlined in the standard operating procedures of the Licensee submitting the Test Batch.

Colorado Marijuana Enforcement Division: 2020 Annual Update

Table 15: Marijuana Testing Results as of December 31, 2020 (Percentage of Packages that Passed Testing)

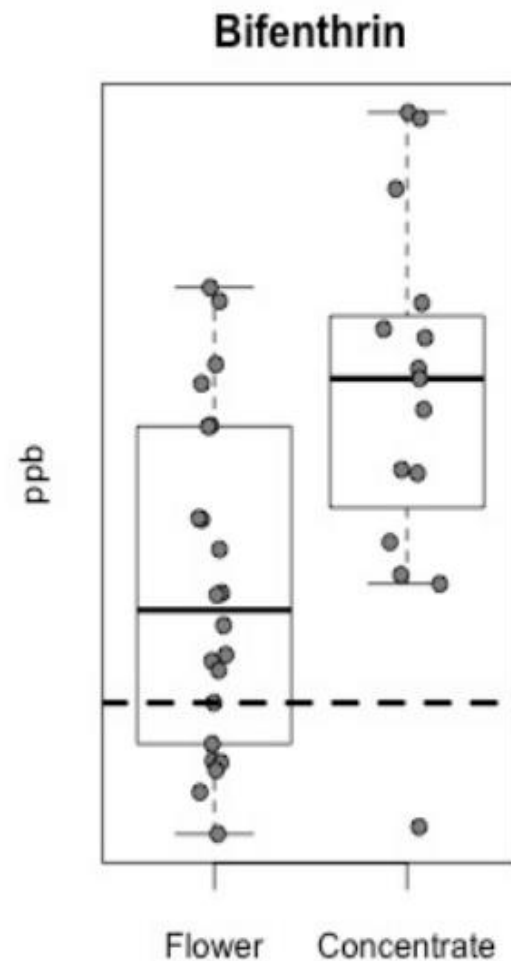
	Market	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Total
Concentrate Microbial Contaminants Results	Adult Use	99.19%	99.17%	97.34%	95.73%	99.02%	99.55%	95.93%	97.21%	98.50%	99.08%	99.61%	99.29%	98.28%
	Medical	99.65%	99.67%	99.23%	98.77%	98.38%	99.53%	100%	98.81%	100%	98.34%	99.60%	99.07%	99.21%
Concentrate Potency Results	Adult Use	99.62%	99.72%	99.83%	99.53%	99.58%	99.76%	99.82%	99.77%	99.72%	99.66%	99.88%	99.76%	99.72%
	Medical	99.94%	99.97%	99.70%	99.95%	99.86%	99.83%	99.91%	99.81%	99.96%	99.90%	100%	99.97%	99.90%
Concentrate Residual Solvents Results	Adult Use	95.90%	95.25%	94.90%	93.92%	95.43%	95.66%	94.98%	95.03%	94.44%	94.86%	96.67%	96.04%	95.22%
	Medical	95.77%	96.31%	95.30%	95.27%	94.86%	95.51%	95.69%	95.27%	97.11%	95.56%	96.27%	96.07%	95.71%
Concentrate Metals Results	Adult Use	95.28%	96.62%	95.09%	98.66%	98.08%	98.88%	98.14%	98.74%	97.72%	98.87%	98.71%	98.02%	98.15%
	Medical	97.37%	96.47%	98.55%	99.13%	97.82%	99.36%	99.41%	98.61%	98.82%	97.86%	98.12%	98.55%	98.68%
Flower and Shake/Trim Microbial Contaminants Results	Adult Use	84.89%	82.25%	83.45%	86.99%	85.71%	84.44%	85.48%	82.93%	82.94%	82.59%	86.37%	85.25%	84.43%
	Medical	86.33%	86.04%	86.05%	85.42%	86.30%	87.76%	88.94%	84.65%	85.43%	85.87%	84.70%	81.93%	85.78%
Flower and Shake/Trim Pesticides Results	Adult Use	99.33%	99.25%	98.47%	98.20%	97.83%	98.87%	98.48%	98.25%	98.48%	96.75%	97.49%	97.95%	98.24%
	Medical	99.65%	99.48%	99.19%	98.20%	97.38%	97.97%	97.84%	98.20%	98.88%	98.79%	97.67%	99.08%	98.58%
Flower and Shake/Trim Potency Results	Adult Use	99.87%	99.56%	98.48%	99.28%	99.25%	99.73%	99.58%	99.09%	99.71%	99.17%	99.39%	98.96%	99.35%
	Medical	99.96%	99.83%	99.14%	99.19%	98.94%	99.33%	99.29%	99.42%	99.59%	99.47%	99.34%	99.44%	99.42%
Flower and Shake/Trim Metals Results	Adult Use	96.70%	95.88%	93.71%	91.15%	93.19%	95.54%	94.45%	91.57%	93.19%	91.31%	88.97%	90.53%	93.32%
	Medical	97.17%	95.89%	93.85%	91.56%	94.27%	94.72%	96.11%	97.58%	97.23%	92.90%	96.51%	97.37%	95.36%
Infused Edible Potency Results	Adult Use	86.10%	84.93%	84.27%	87.74%	74.42%	84.49%	86.44%	86.55%	90.64%	86.09%	84.97%	89.39%	85.48%
	Medical	91.75%	84.92%	89.59%	91.61%	87.70%	88.72%	88.01%	80.33%	93.56%	91.96%	88.07%	94.85%	89.38%
Infused Edible Homogeneity Results	Adult Use	87.79%	84.94%	83.18%	88.01%	72.82%	84.23%	86.19%	85.57%	89.08%	83.56%	83.91%	88.89%	84.77%
	Medical	93.26%	85.96%	89.30%	94.14%	88.41%	89.66%	88.21%	79.89%	93.21%	92.92%	90.39%	95.59%	90.25%
Infused Edible Microbial Contaminants Results	Adult Use	99.84%	100.00%	99.05%	98.67%	99.31%	99.84%	99.84%	99.53%	99.36%	99.18%	99.81%	99.58%	99.50%
	Medical	100.00%	99.44%	97.81%	97.78%	99.46%	100.00%	99.39%	97.50%	100.00%	100.00%	100.00%	99.32%	99.23%
Infused Non-edible Microbial Contaminants Results	Adult Use	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%
	Medical	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%
Infused Non-Edible Potency Results	Adult Use	90.85%	94.64%	83.33%	73.20%	85.94%	85.05%	92.91%	90.38%	87.23%	88.00%	80.65%	89.33%	87.44%
	Medical	88.68%	92.31%	92.00%	73.08%	88.89%	89.13%	74.29%	100.00%	100.00%	100.00%	80.00%	100.00%	89.01%

Concentrate quality & safety:

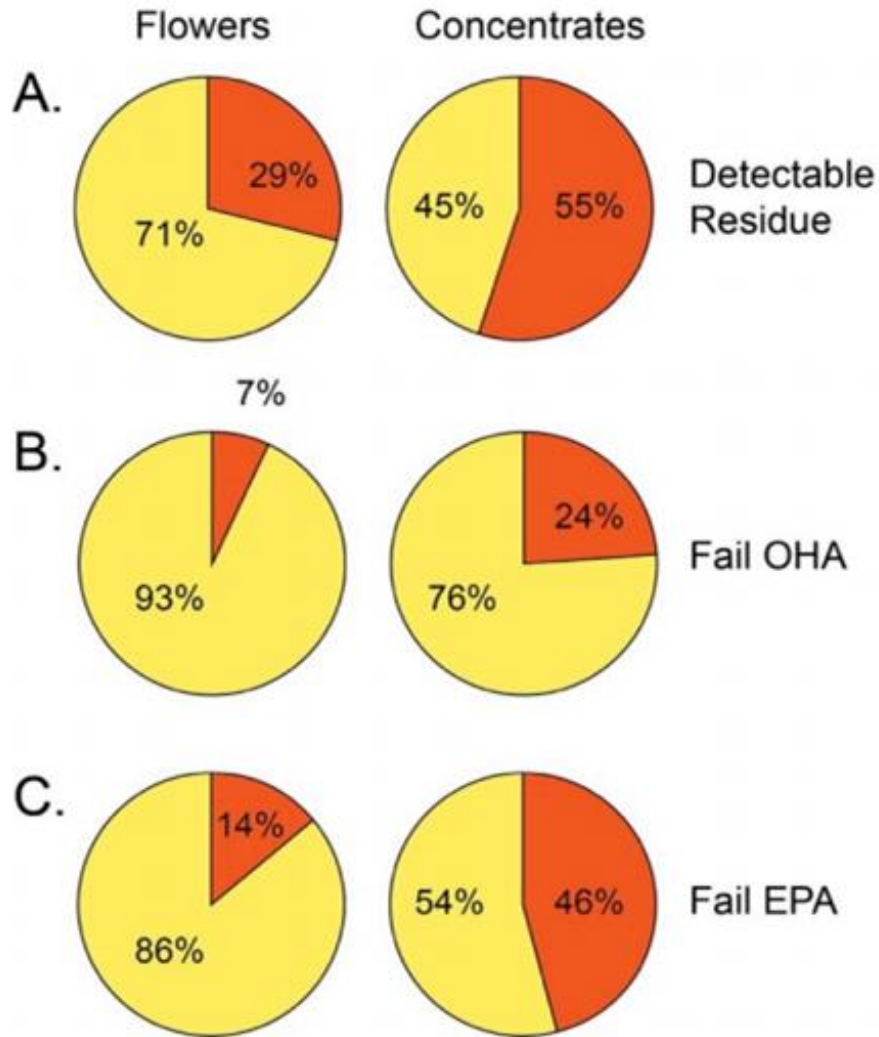
10X concentration of pesticides!

1. Increased affinity for the solvents used
2. Chronic contamination of extraction equipment
3. Use of trim

<http://cannabissafetyinstitute.org>



Concentrate quality & safety



<http://cannabissafetyinstitute.org>

New Regulations - Colorado

5. Elemental Impurities Testing.

- a. Each Harvest Batch and Production Batch of Regulated Marijuana must be tested for elemental impurities by a Regulated Marijuana Testing Facility at the frequency established in paragraphs (A) and (B) of this Rule. The elemental impurities test must include, but need not be limited to, testing to determine the presence of, and amounts present of, arsenic, cadmium, lead, and mercury.
- b. Emissions Testing. This subsection (C)(5)(b) is effective January 1, 2022. Each Harvest Batch and Production Batch of Regulated Marijuana Concentrate in a Vaporized Delivery Device must be tested for elemental impurities via emissions testing by a Regulated Marijuana Testing Facility at the frequency established in subparagraphs (A) and (B) of this Rule. The elemental impurities test must include, but need not be limited to, testing to determine the presence and amounts of arsenic, cadmium, lead, and mercury.

Methods from ENDS

- Impinger Methods – Needed Optimization
- Smoke machines (EPT) – Never used on *cannabis* products
- Filter based methods – Mostly new

Impinger Based Techniques

3 Experimental Protocol

A setup like the configuration in figure 1 will be used. A standardized puff protocol will be used that mimics human smoking behaviour. After every 20 puffs, trap 1 and trap 2 are changed out. It is expected that an electronic e-cigarette cartridge can produce around 140 puffs, which would result in $(140/20=)$ 7 samples. If detection limits of the analytical equipment are higher than desired, the experiment can be repeated with multiple electronic cigarette cartridges and the same puff blocks can be collected in the same traps to get a more concentrated sample. The process described above will be performed in triplicate as to yield data for statistical analysis.

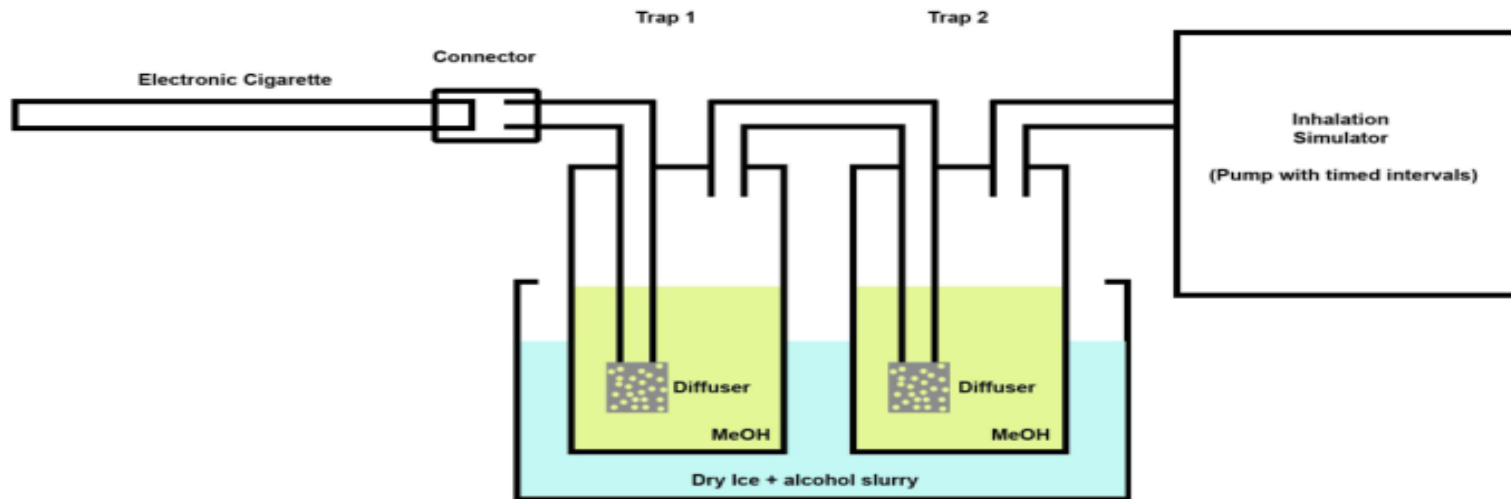


Figure 1. The experimental setup

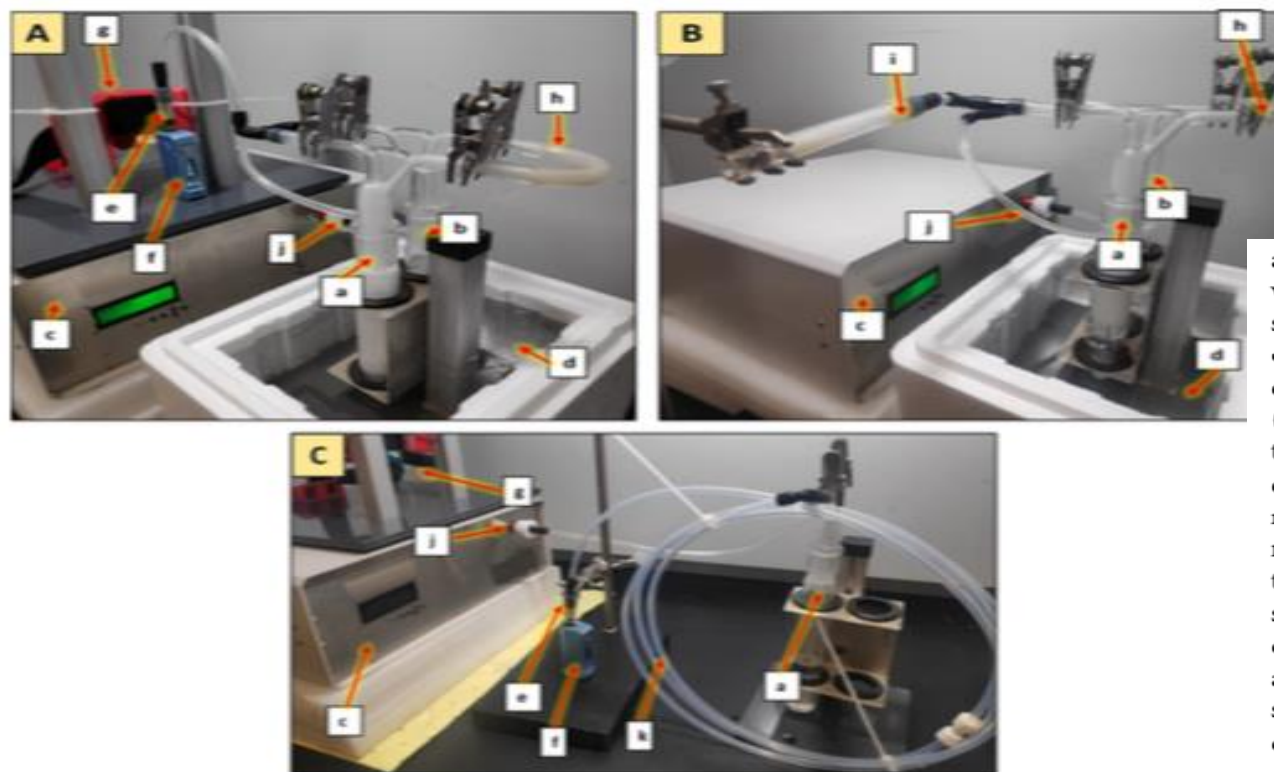


Strategies for Nonpolar Aerosol Collection and Heavy Metals Analysis of Inhaled Cannabis Products

Srinivasa Reddy Mallampati, Charles McDaniel, and Amber R. Wise*

Cite This: *ACS Omega* 2021, 6, 17126–17135

Read Online



aqueous media was problematic. Overall, average recoveries were quite low for many of the metals while also having large standard deviations, which has also been demonstrated in e-cigarette studies^{8,10} suggesting metals do not aerosolize efficiently. The highest recovery metals are the most volatile (As and Hg). The large standard deviations are indicative of the wide range of metal aerosolization that is seen in any single experiment. This points to the importance of requiring multiple experimental collections when considering regulatory requirements for aerosol testing of cannabis products. Further, the data in Figure 2 shows that without using an organic solvent, the aqueous-only impinger combinations would not capture as many of the aerosolized metals. However, the aqueous impinger was important to capture mercury, so both should be incorporated if a full suite of metals is to be reliably collected.

Figure 1. Experimental setup for vaporized aerosol collection from the cannabis concentrate cartridge with impingers only (A), combusted aerosol collection from the cannabis flower (B), and tubing condensation and impinger (C). Components of the setup include (a) first impinger, (b) second impinger, (c) smoke machine, (d) ice bath, (e) cartridge with concentrate, (f) battery, (g) button pusher, (h) tubing between impingers, (i) combustion sample holder, (j) vacuum inlet, and (k) condensation tubing.



STATUS /DEV/TTYACI **CONNECTED**

SESSION

PUFF VOLUME 30 10 90	INHALE 2.5 1.0 10.0	EXHALE 2.0 1.0 10.0	OUTPUT POWER 150 0 255	PREHEAT 1.0 -4.0 4.0
PURGE VOLUME 30 10 100	PURGE INHALE 4.5 1.0 10.0	PURGE EXHALE 4.5 1.0 10.0	<input type="text" value="0"/> Execute a total of Z purge puffs	<input type="text" value="10"/> Execute purge puff every N seconds

Execute a total of Z puffs Microsteps per ml

Execute inhale every N seconds

SERIES

Execute a total of Z sessions

Execute session every N seconds

LOG

```
Load settings pressed
Load settings pressed
Load settings pressed
Loading settings
from/home/pi/Desktop/Z-
CHANNEL/Methods/Puffer
Program T2.29.2021
Puff:10
Inhale:2.5
Puff volume:30.0
Exhale:2.0
Inhale:60.0
Num puffs:1
Output power:150.0
Session interval:60
Num sessions:60
Low vapor alert: false
Purge volume:30.0
Inhale duration:4.5
Exhale duration:4.5
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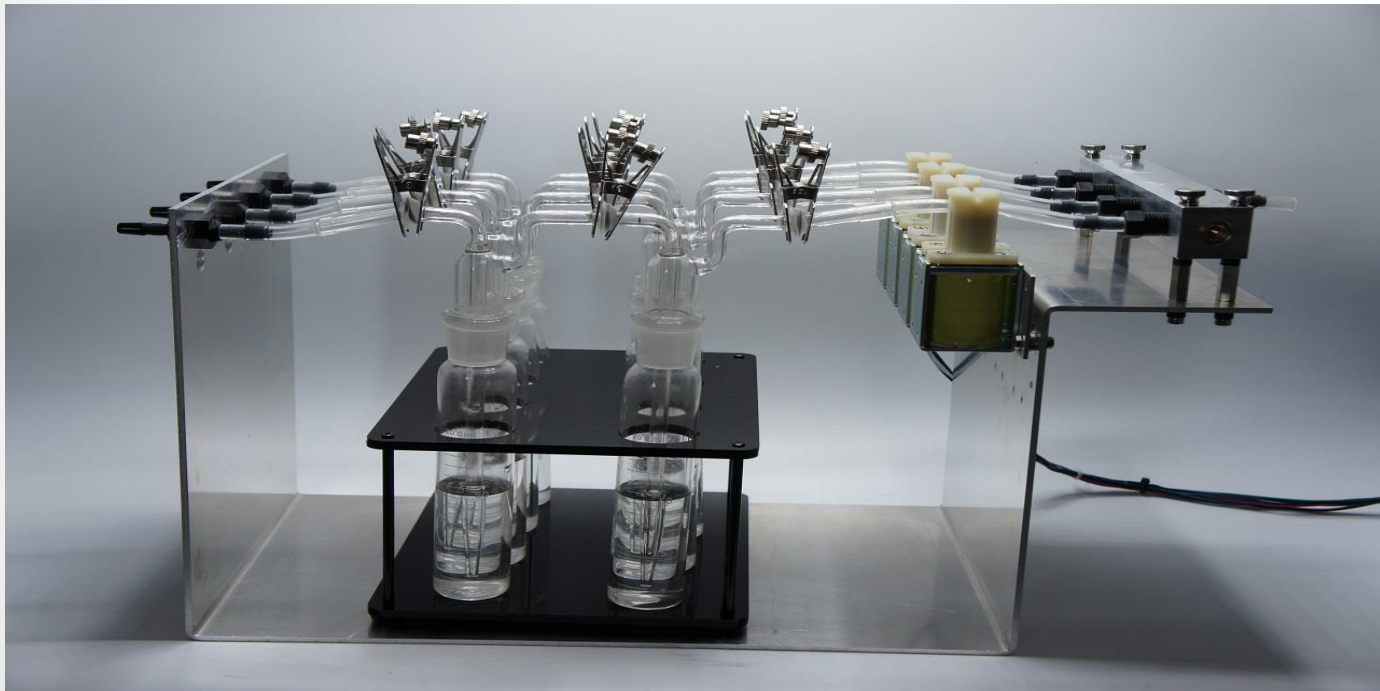

LOW VAPOR ALERT DISABLED

00:01:00.0

01:00:00.0

00:00:00.0

Channel 1 Channel 2 Channel 3 Channel 4



Control Matrices

Hemp seed oil: This was the initial trial, as it is used in the lab for many different testing assays as a concentrate matrix for testing. The hemp seed oil only worked in certain cartridges (Silver V9 1 gram) that had no air intake vents on the bottom of the atomizer. In other cartridges that have this feature, which is common in the most popular types (such as the V12 ceramic tip), it would leak out and short the energizing cables. This is due to the low viscosity of the hemp seed oil. Besides leaking out the air intakes, it would also clog these intakes creating clogs in the carts and not allowing the vapor smoke to pass through the system into the impingers.

Maple flavored Pancake Syrup:

This matrix was chosen for its viscosity that is more similar to cannabis and hemp distillates, which is much higher than the hemp oil previously used. Unfortunately, the smoke point of this corn syrup based product is too high to be effectively used in the Gram mechanical puffer.

Coconut Oil:

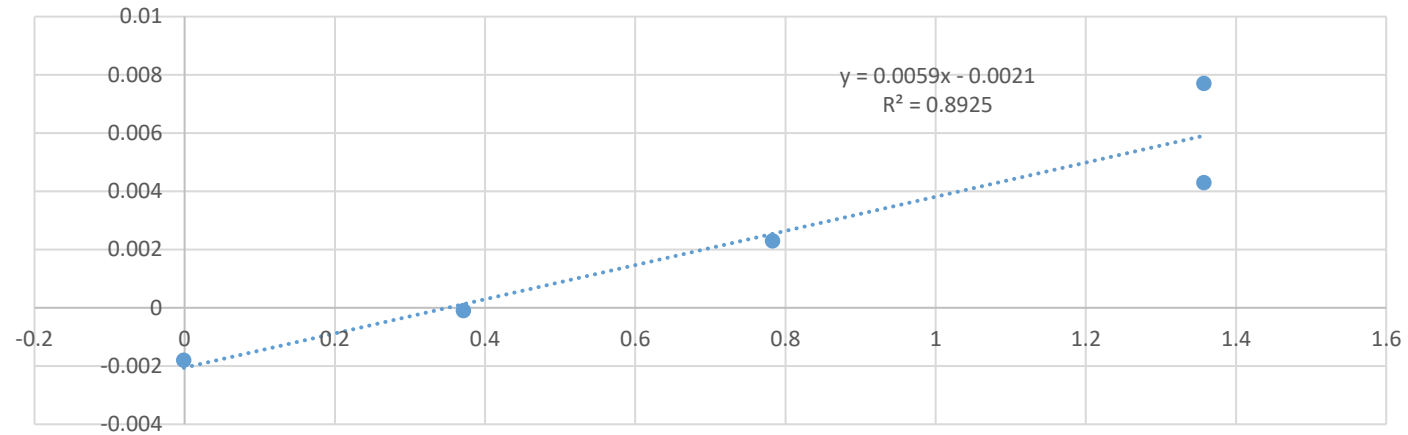
This matrix was chosen for its viscosity that is more similar to cannabis and hemp distillates, which is much higher than the hemp oil previously used. Unfortunately, this product also did not work for our testing purposes due to insufficient vape smoke created and insufficient weight loss during the cycle.

Cannabis and Hemp Distillate:

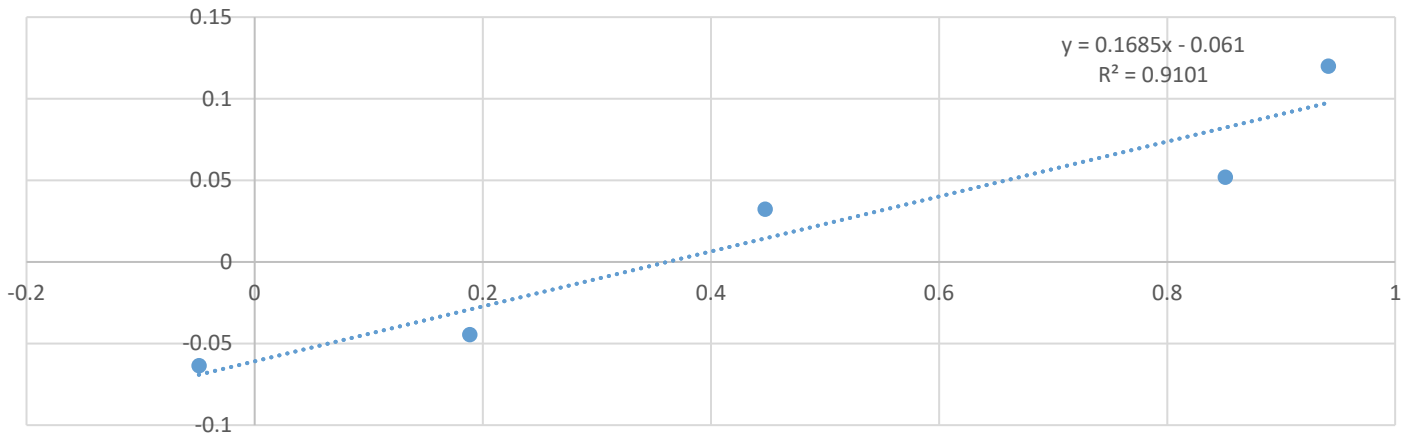
This matrix was chosen for the obvious reason that it will be equivalent to the hemp and cannabis distillate that would be expected in atomizers submitted for testing. The drawback is that it is very difficult to work with due to the viscous nature of the material. These difficulties aside, the functionality of the distillate in the Gram mechanical puffer is excellent.

Sample	Concentration (measured)					% Recoveries				Average
	Concentration (ppm)	As (ppm)	Cd (ppm)	Hg (ppm)	Pb (ppm)	As (ppm)	Cd (ppm)	Hg (ppm)	Pb (ppm)	
A	0	-0.0012	-0.0017	-0.0488	-0.0027	0%	0%	0%	0%	0%
B	0.3	0.3711	0.3469	0.1886	0.3699	81%	86%	159%	81%	102%
C	0.6	0.7825	0.7379	0.4476	0.8259	77%	81%	134%	73%	91%
D	0.9	1.3571	1.1905	0.851	1.3056	66%	76%	106%	69%	79%
E	1.2	1.3571	1.1839	0.9413	1.2956	88%	101%	127%	93%	102%

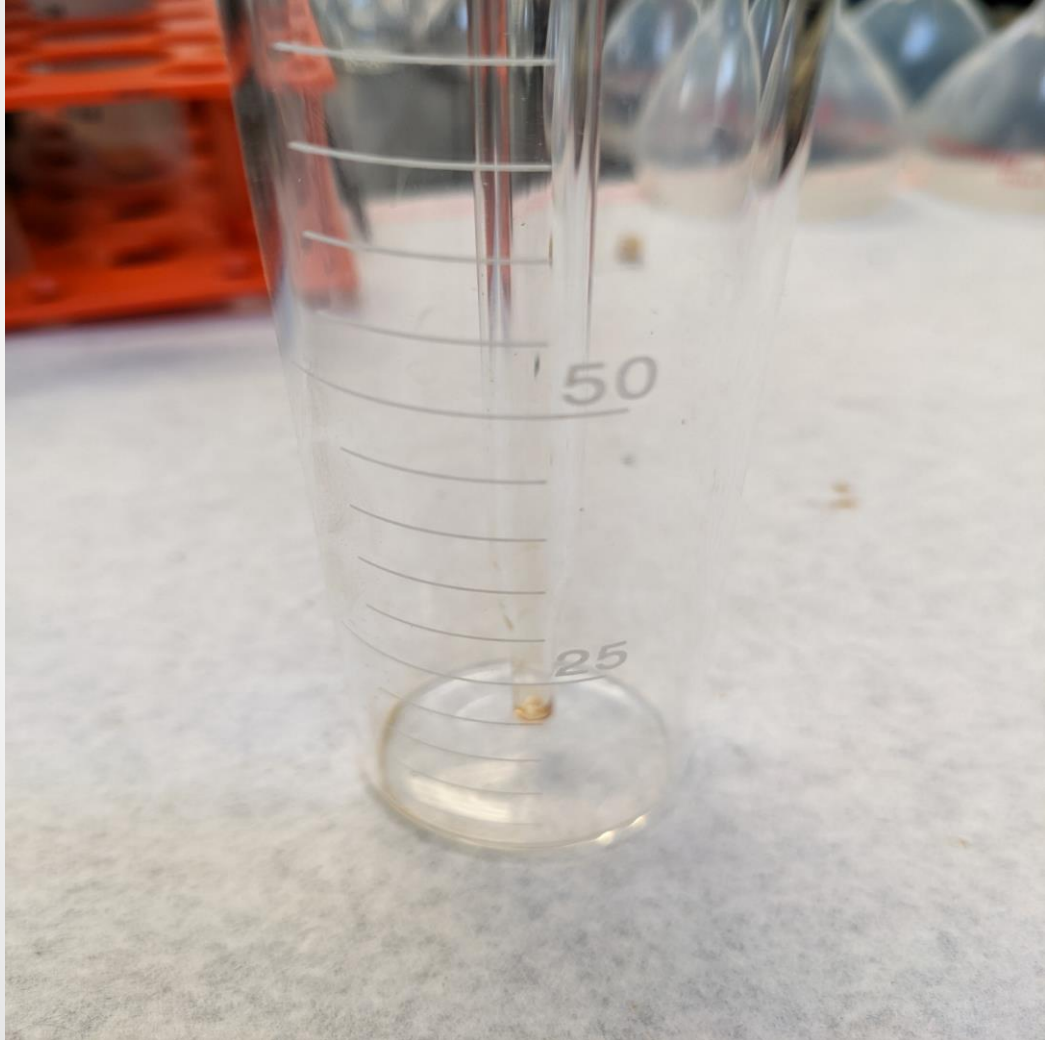
As Impinger Collection



Hg Impinger Collection



Low Recoveries



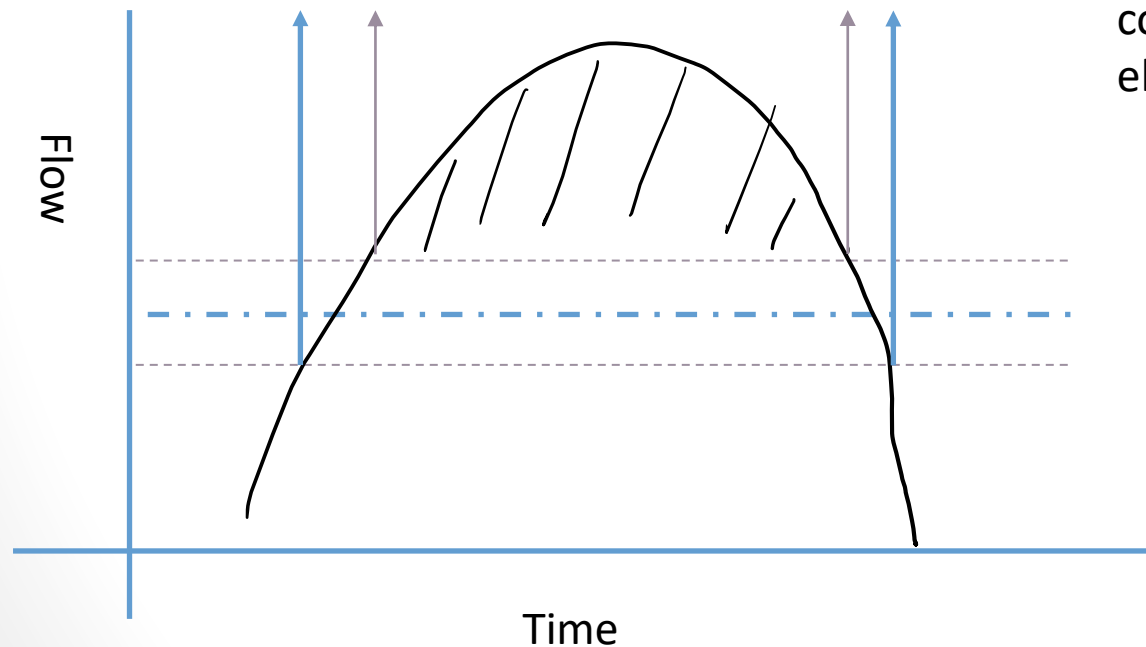


- Determine the amount of aerosol condensate that can be trapped by any single trapping system
 - Starting with a typical vape puff profile of 55ml puff volume/3 second puff duration, 30 second interval between puffs, with a square wave puff profile (55/3/30/sq) (Reference see CRM81).
 - Starting with a block of 10 puffs per product.
 - Maximum loading for CFPs of different size (e.g. 44mm, 55mm, 92mm).
 - Breakthrough considerations for pads.
 - Mass transfer to pads from devices.
 - Efficiency of capture (volatiles and non-condensate considerations).
 - Pressure drop (PD) changes during use and potential impact on capture ref flow activated devices vs button activated devices.
- Determine the efficiency of capture.
 - Losses and mass balance.
- Alternate metals capture systems.
 - Electrostatic Precipitation Trap (EPT) capture.
 - Efficiency of capture.
 - Contraindications (overload, coating).

1. Puff

The vapour dose in electronically heated products is dependent on the length of time the heater is on (to a first approximation) not on the total volume of puff

In flow activated devices the heater is activated once a flow rate threshold is exceeded



A cigarette ISO “bell shape” will produce considerable variation in aerosol generation due to electronic device tolerance

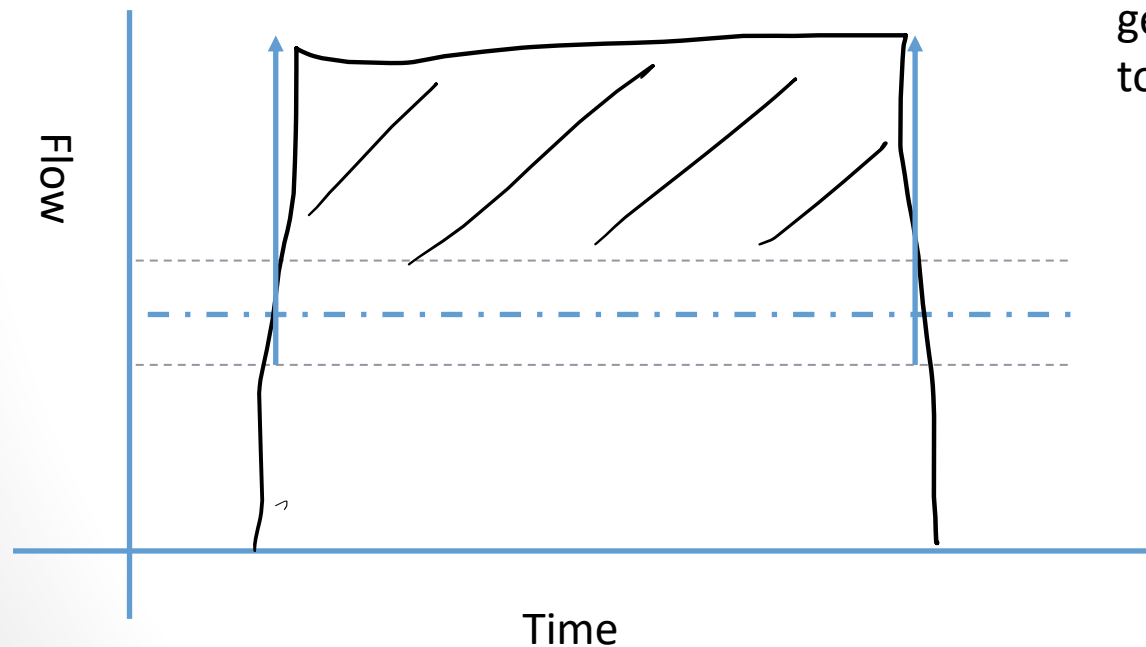
Flow trigger point

Tolerance

1. Puff

The vapour dose in electronically heated products is dependent on the length of time the heater is on (to a first approximation) not on the total volume of puff

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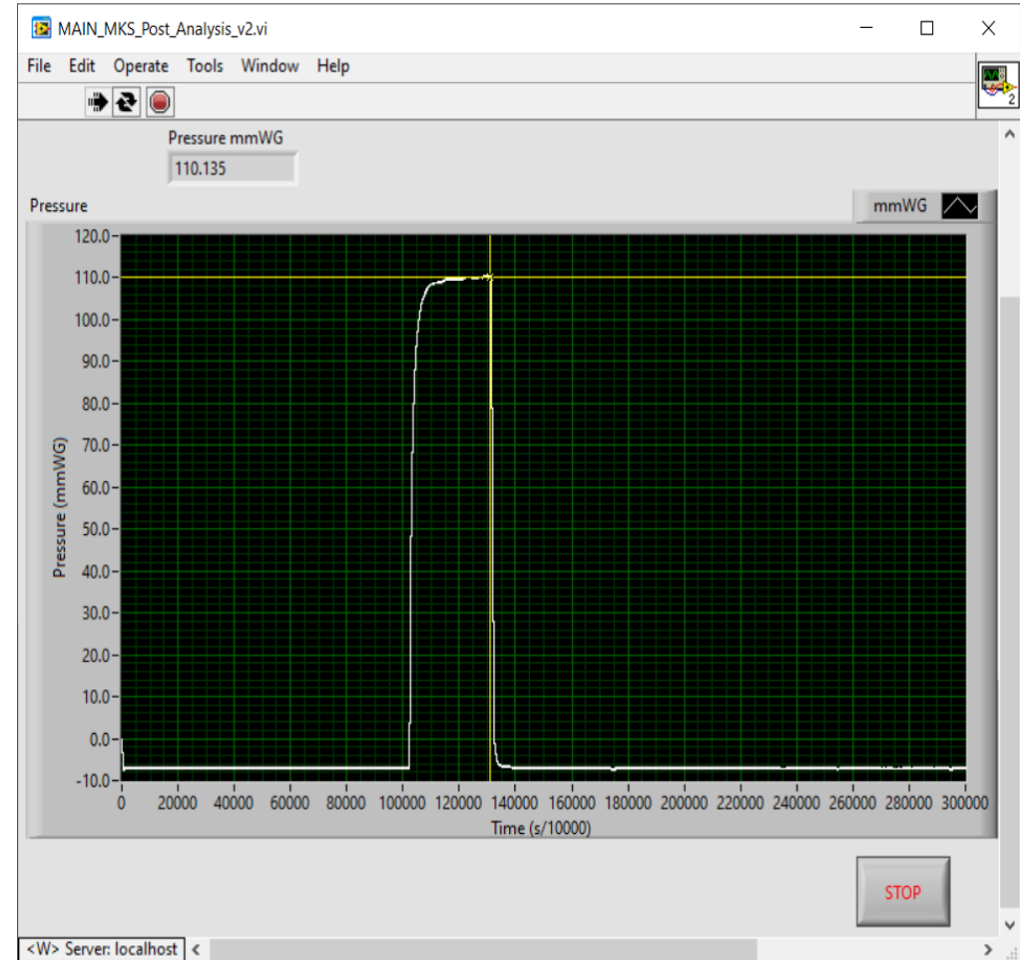
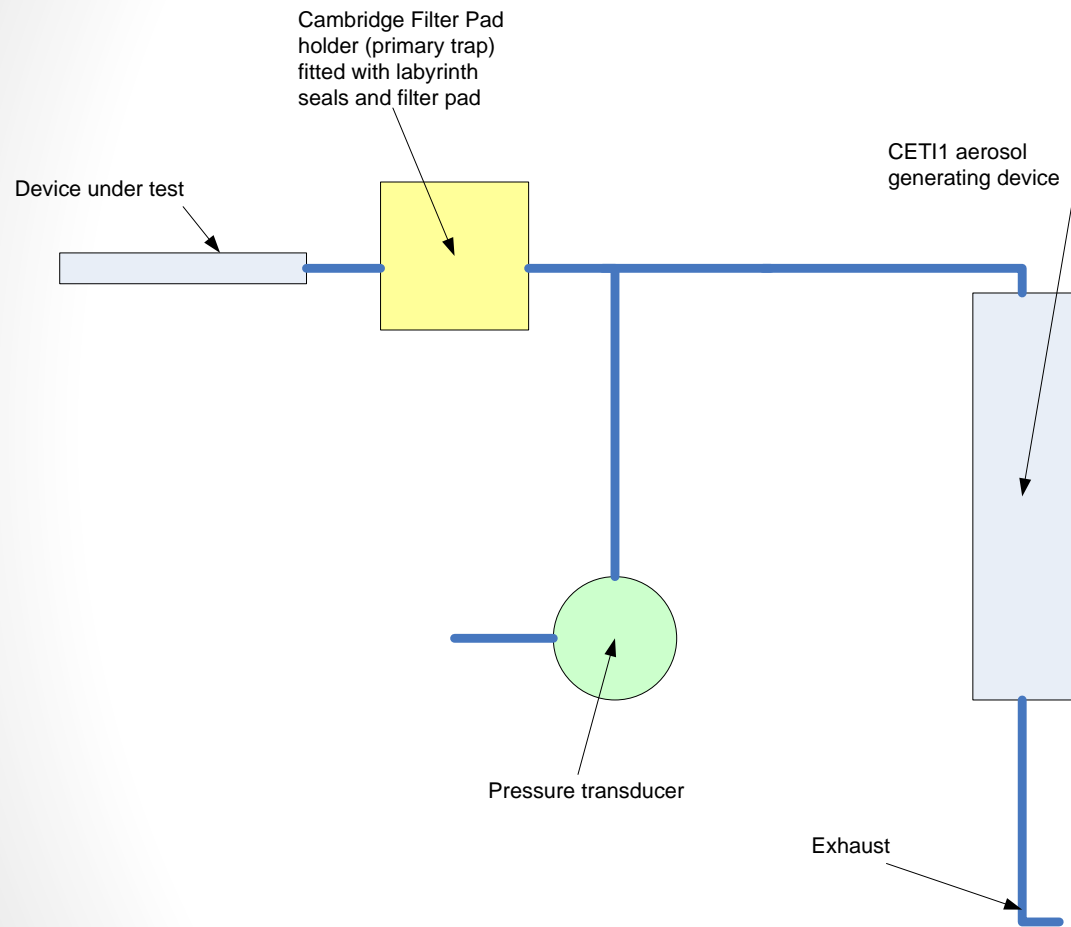


A “square wave” gives consistent aerosol generation irrespective of electronic component tolerances

Flow trigger point

Tolerance

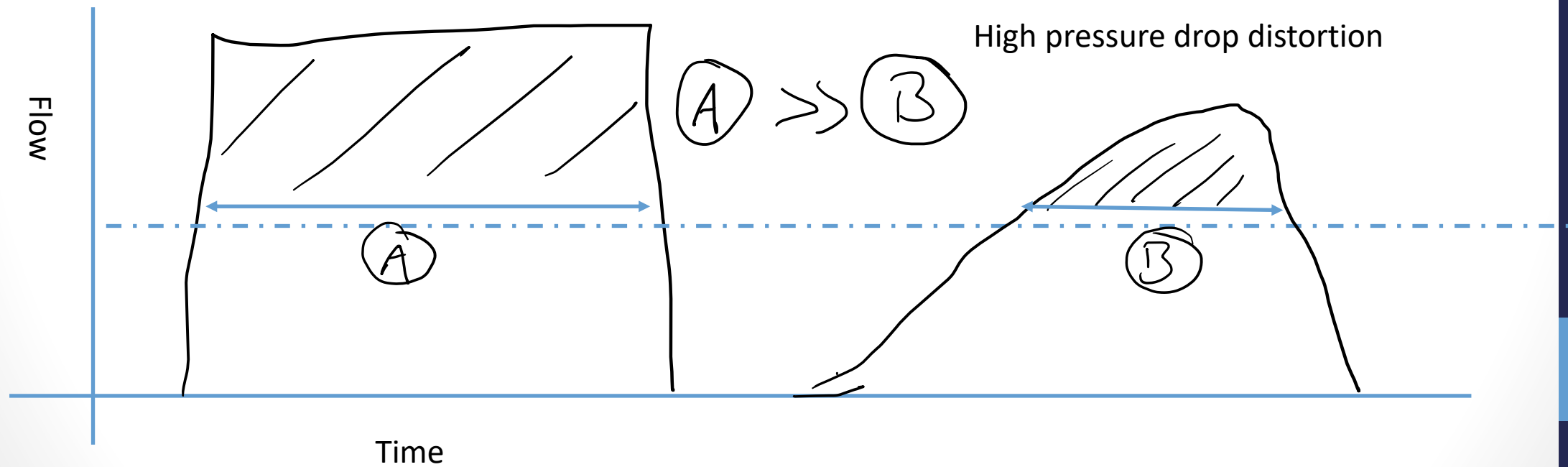
Dynamic monitoring



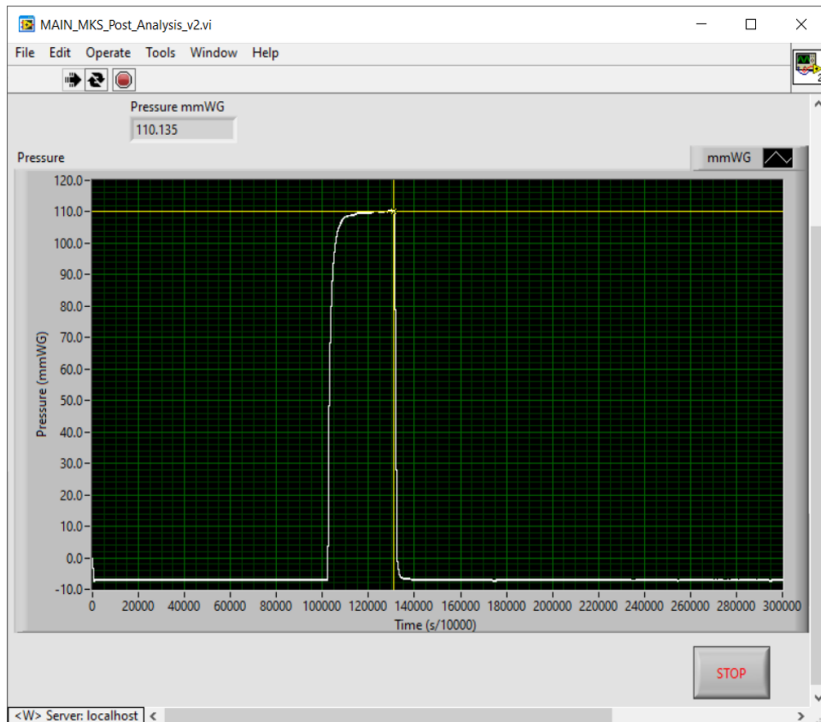
1. Puff

Distortion of the puff shape can occur through high pressure drop within the vaping device

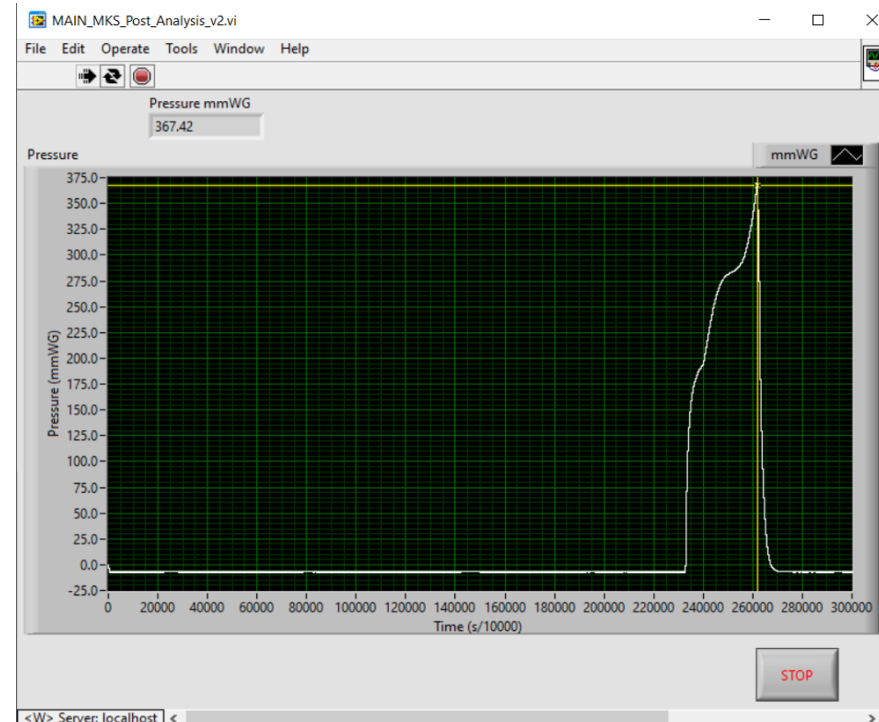
A limit should be set on pressure drop $\sim 200\text{mmWg}$.



- The THC oils had different levels of total cannabinoids. From 80% to 94% in preloaded cartridges
- As the total cannabinoid content increased the liquids became more viscous
- As the liquids became more viscous the harder it became to pull a puff and vape – see PD traces



Low viscosity ~ 110mmWg PD



High viscosity > 350mmWg PD

Mass Recovery

Table 1: Recovery weights from Select Essential Berry Gelato cannabis cartridge

Puff block	Puffs	Cartridge weight loss /g	Primary CFH weight gain /g	Secondary filter weight gain /g	Total weight recovered /g	Total recovery %	Primary recovery %
1	40	0.20719	0.2052	0	0.2057	99%	99%
2	35	0.08711	0.0845	0	0.0846	97%	97%
3	40	0.2278	0.2240	-1E-04	0.2242	98%	98%
4	40	0.1201	0.1162	0.0008	0.1181	98%	97%
5	40	0.0264	0.0246	-0.0011	0.0215	81%	93%
total		0.6686	0.6545	-0.0004	0.6541	98%	98%

?



Viscosity – Delivery Uniformity

Table 1: Delivery per puff for product with medium viscosity Product 16MV

Product 16MV			
puffs	weight captured per block (g)	delivery per puff (g)	total weight captured (g)
40	0.0634	0.001585	0.0634
80	0.0426	0.001065	0.1060
120	0.0572	0.001430	0.1632
160	0.0424	0.001060	0.2056
200	0.0594	0.001485	0.2650
average per puff delivery		0.001325	
std dev per puff delivery		0.000246	
COV		0.18566	

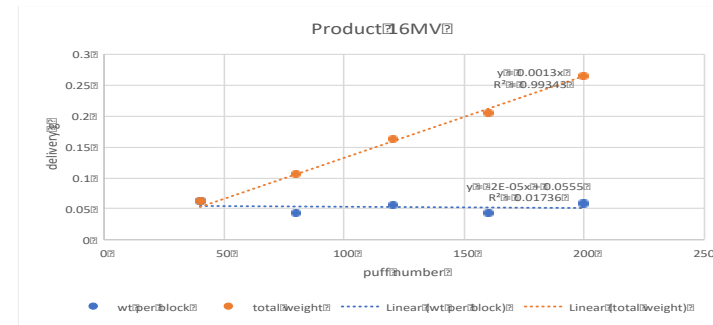


Figure 1: Plot of delivery from Product 16MV per puff and on an cumulative basis

Table 2: Delivery per puff for low viscosity cannabis oil Product 17LV

Product 17LV			
puffs	weight captured per block (g)	delivery per puff (g)	total weight captured (g)
40	0.0641	0.001602	0.0641
80	0.0423	0.001057	0.1064
110	0.035	0.001167	0.1414
150	0.0434	0.001085	0.1848
190	0.0354	0.000885	0.2202
average per puff delivery		0.001159	
std dev per puff delivery		0.000268	
COV		0.23123	

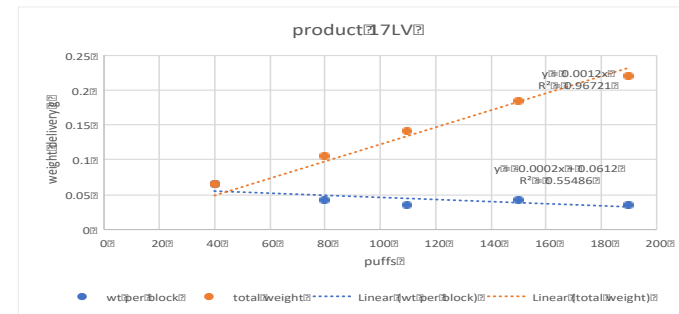


Figure 2: Plot of delivery from Product 17LV per puff and on an cumulative basis

Table 3: Delivery per puff for high viscosity cannabis oil Product 18HV

Product 18HV			
puffs	weight captured per block (g)	delivery per puff (g)	total weight captured (g)
40	0.0363	0.000907	0.0363
80	0.0323	0.000807	0.0686
120	0.0231	0.000577	0.0917
160	0.0313	0.000783	0.1230
200	0.0295	0.000737	0.1525
average per puff delivery		0.000762	
std dev per puff delivery		0.000121	
COV		0.15879	

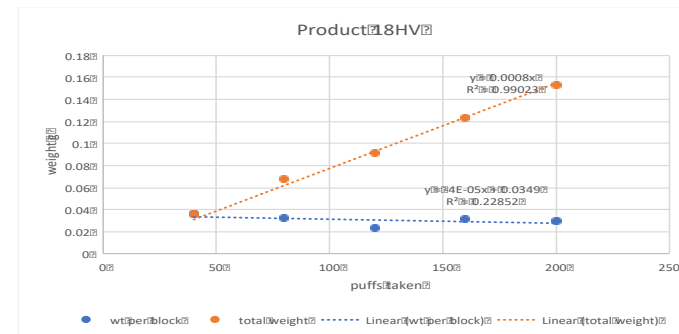
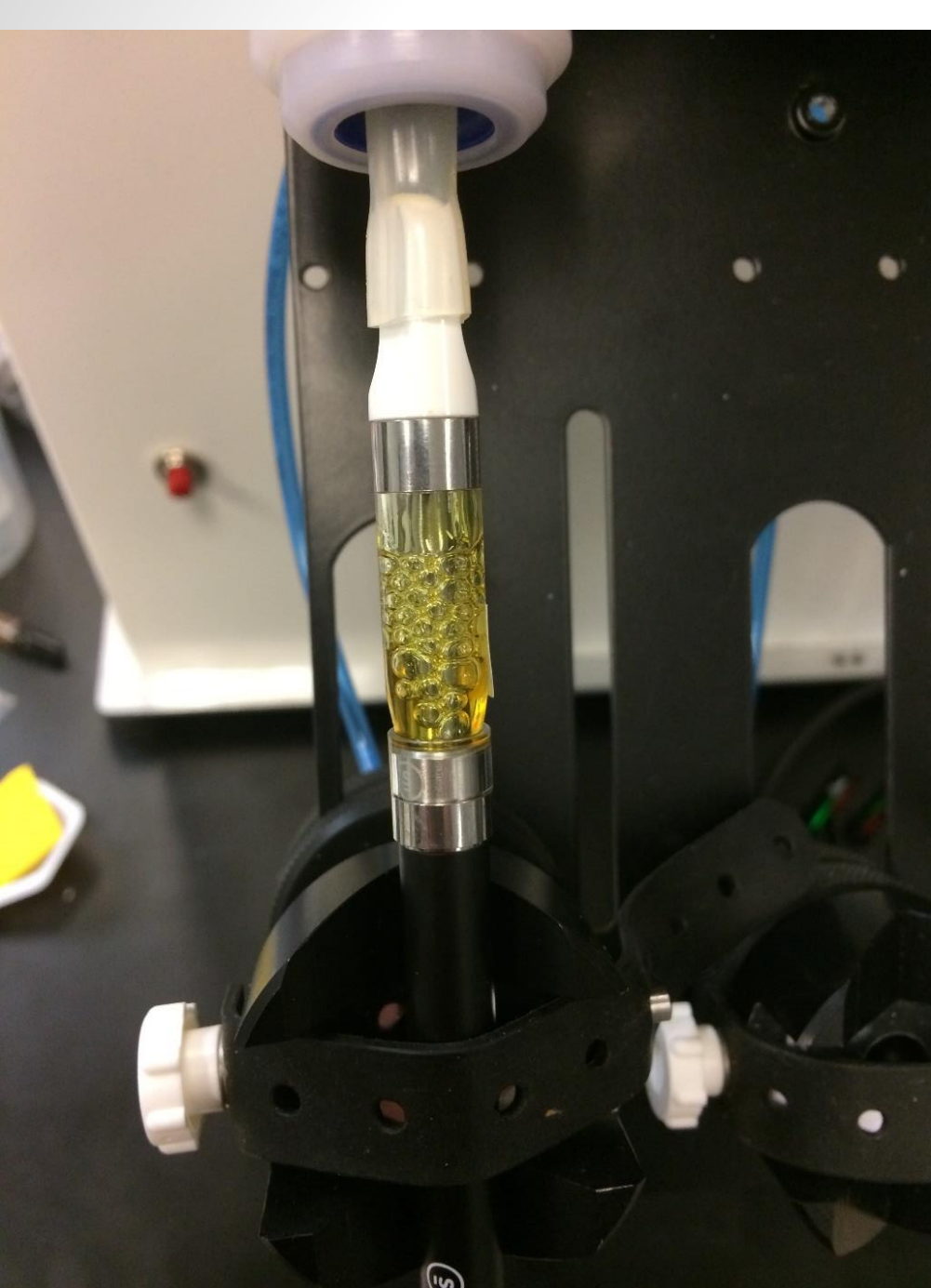


Figure 3: Plot of delivery from Product 18HV per puff and on an cumulative basis



High viscosity oils started to “freeze in” the liquid

3. Oil

To generate aerosol we can adopt 2 strategies

1. Keep the oil warm throughout the experiment
2. Dilute the oil with a solvent that does not change the oil chemistry

Heating to $\sim 50^\circ$ through an external radiant heater will allow 1mg/puff yields to be delivered in the aerosol phase (coil temperatures $\sim 220^\circ$ to 420°C). Oil is easily captured once volatilised

Dilution with IPA increases the delivery per puff depending on dilution – a “sweet spot” would be 30% dilution which delivers $\sim 4\text{mg}$ of aerosol per puff. However only 70% of the puff can be captured by a filter pad, the remaining is lost to the gas phase (probably the solvent is lost)

This is limited in applicability in that it cannot be used for “closed” systems

Dilution

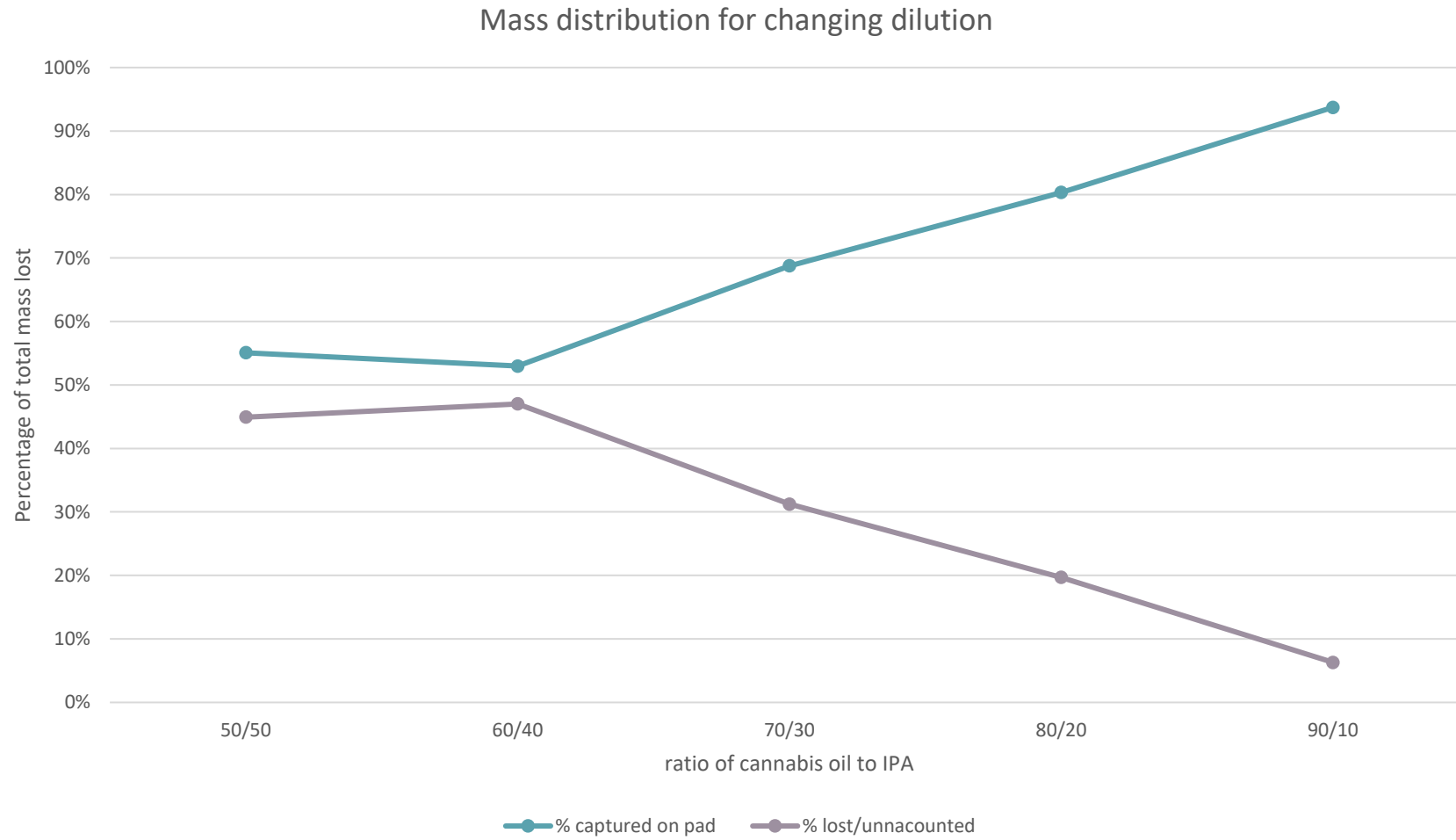
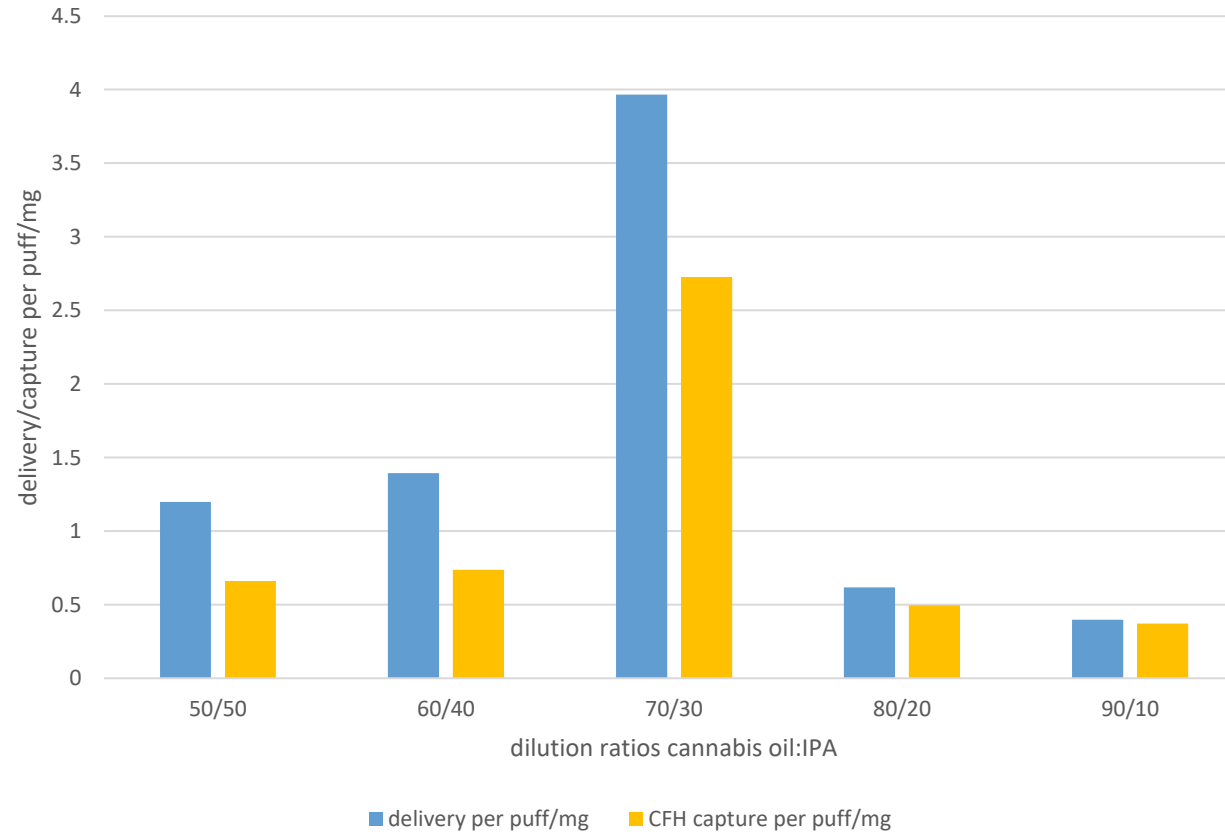


Table 1: mass lost by cartridge and gained by primary capture pad for GoSelect cartridges loaded with different cannabis oil/IPA mixtures

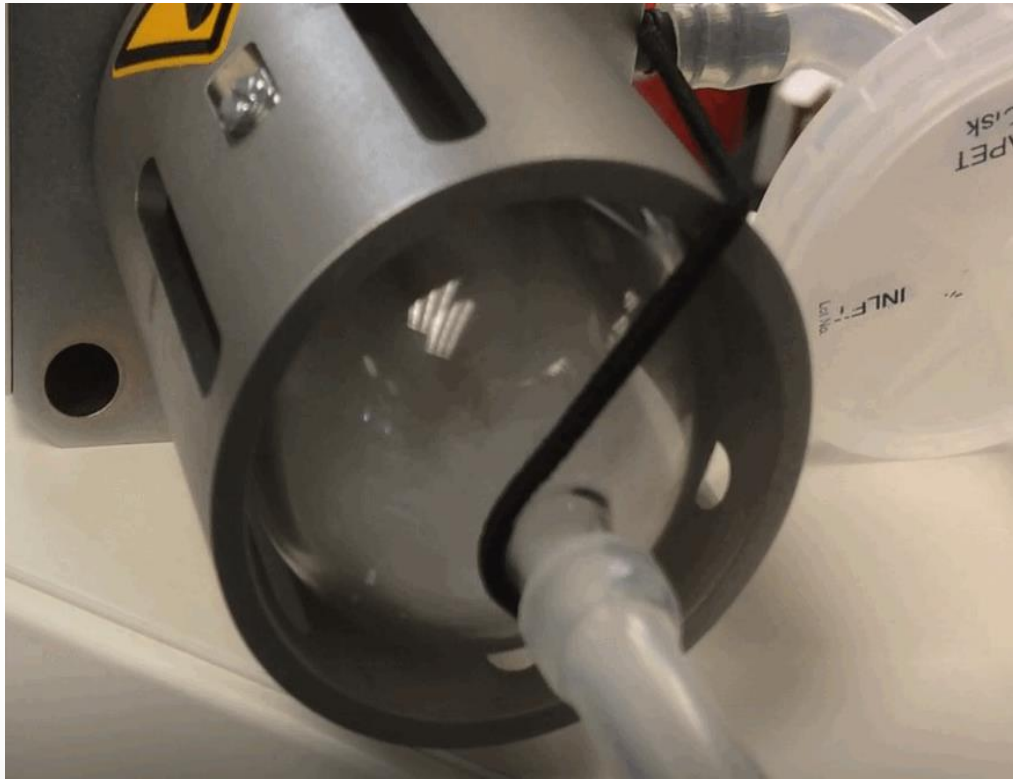
Mix THC:IPA	puffs	mass lost from cartridge/g	weight gain pad/g	mass discrepancy/g	% captured on pad	% lost/unaccounted
50/50	70	-0.0839	0.0462	-0.0377	55%	45%
60/40	100	-0.1393	0.0738	-0.0655	53%	47%
70/30	70	-0.2776	0.1909	-0.0867	69%	31%
80/20	60	-0.0371	0.0298	-0.0073	80%	20%
90/10	40	-0.0159	0.0149	-0.0010	94%	6%

?

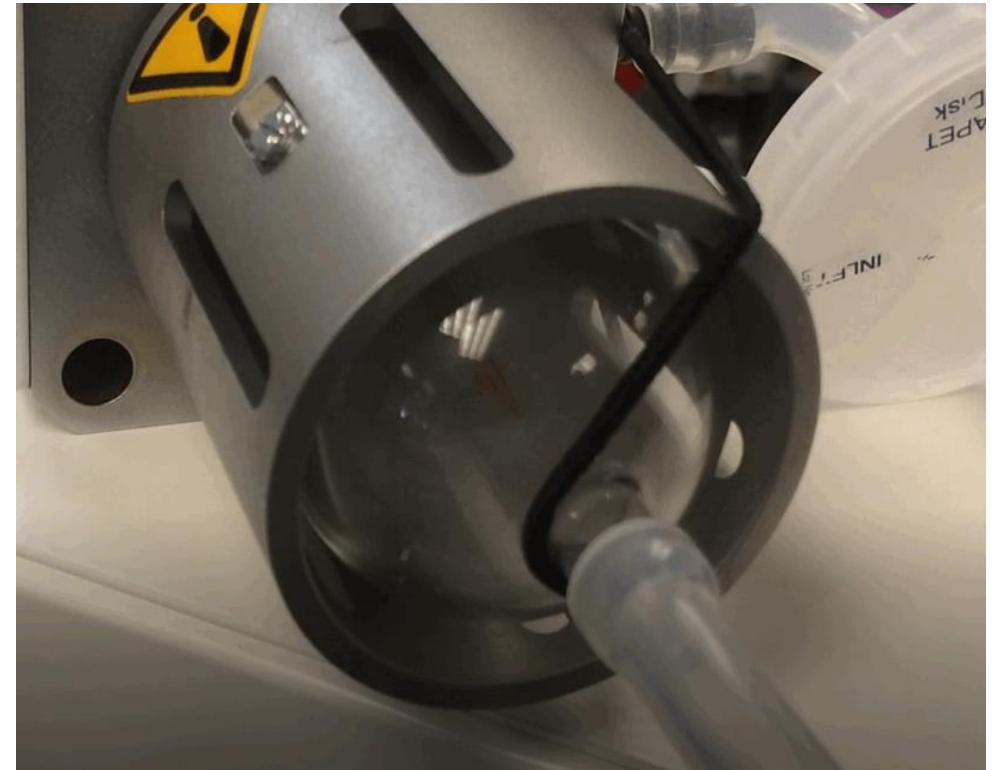
Per puff delivery and capture for dilutions



Visibility



Inside of the Electrostatic precipitator trap at the end of a puff, time zero. The inner electrode is totally obscured by the cannabis aerosol. Device was GoSelect Purple Punch 84.1% total cannabinoids 0.3g cartridge

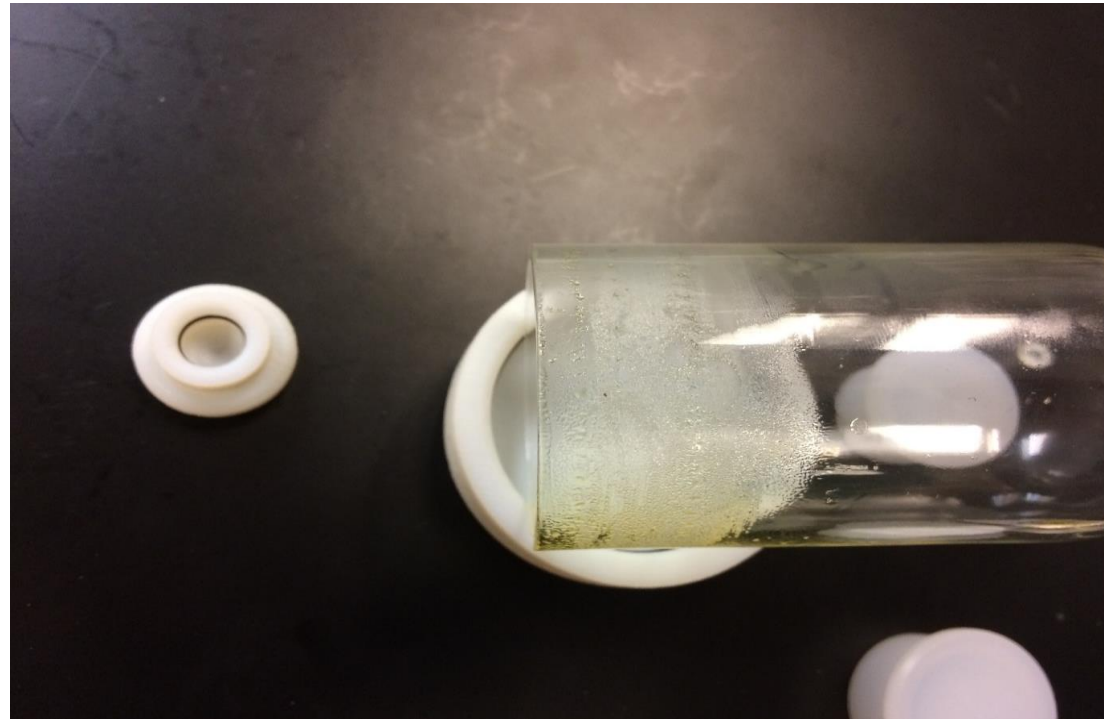


Inside of electrostatic precipitator 30 seconds after puff . The electrode is starting to emerge from the obscuring aerosol but some aerosol remains . This will be drawn through the trap as the next puff is drawn and will not be captured.

4. Aerosols

Electrostatic trapping is a preferred method for smoke analysis – well understood and reliable **But** for cannabis aerosols the recovery rate looks poor ~50%

This is similar to nicotine ENDS aerosols that are non polar. This has an impact on the efficiency of the lab not the accuracy of the method



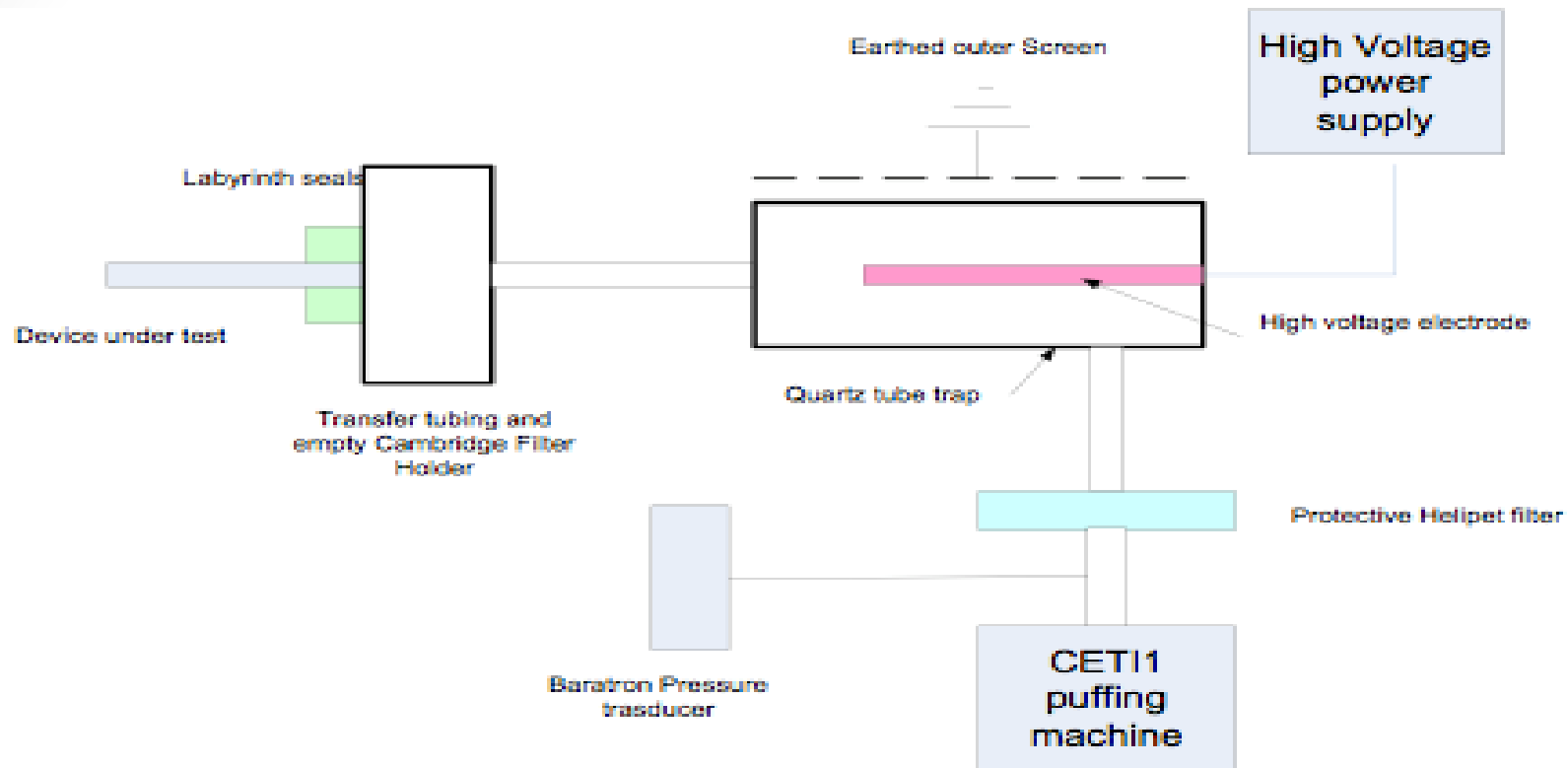


Table 1: Delivered mass of THC from two devices using 5/3/30 puffing regime

	Berry Gelato puff 1-30	Berry Gelato puff 1-60	Purple Punch 100 puffs
% Total Cannabinoids	92.10%		84.10%
mass loss from cartridge/g	0.1484	0.1110	0.0800
mass gained by EP trap/g	0.0433	0.0268	0.0322
Mass gained by transfer tubing/g	0.0125	0.0061	
Mass gained by protective filter/g	0.0450		
mass imbalance (unaccounted mass)/g	0.0476		
Delivery per puff/mg	4.95	3.70	0.80
EP recovery per puff/mg	1.443	0.8933	0.322
EP recovery efficiency	29%	24%	40%
Max recovery transfer tube and EP	1.86	1.10	
Max recovery rate	38%	30%	

Filter Pads for Collection



960 X CAMBRIDGE FILTER PAD - 44MM

Cambridge Filter Pad - 44mm Diameter

Boxed size of 960 individual filters pads

Description

Product Details

Cambridge Filter Pad - 44mm Diameter

Boxed size of 960 Filter Pads

Individual pads (41329) are also available



	As	Cd	Cr	Hg	Pb
CFP 44 mm	2.57	0.06	240.32	ND	1.80
CFP 55 mm	0.71	0.07	19.96	ND	3.68
CFP 92 mm	3.81	0.06	18.03	ND	2.30
#5 Pax Clear	3.04	0.08	17.56	ND	1.89
#6 Cliq Pineapple	2.32	0.06	13.83	ND	1.54
#6 Cliq Pineapple	2.21	0.06	13.99	ND	1.50
New Ultra Low Metal FP	0.04	0.00	0.95	ND	0.02

Quartz fibre filters

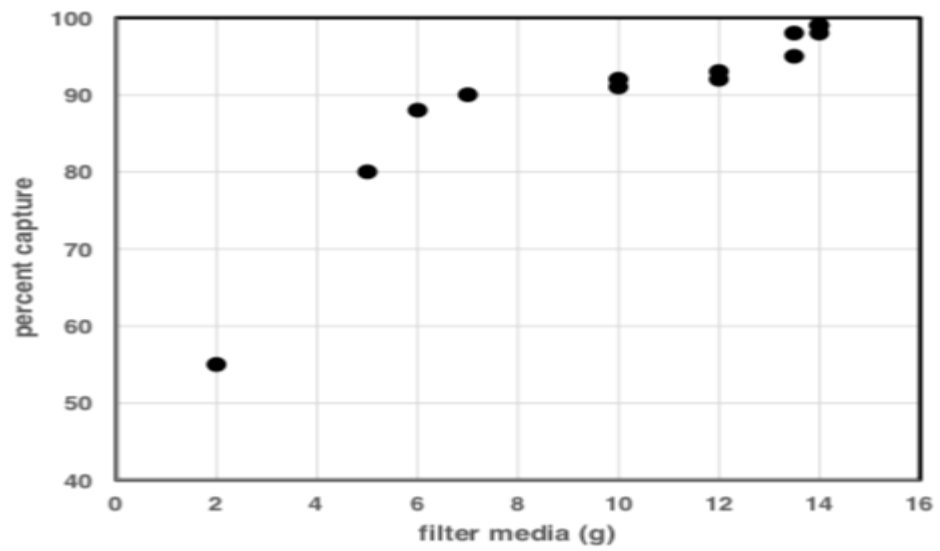
Supplier: [MACHEREY-NAGEL](#)

Ratings: (No Reviews)



Quartz fibre filters feature an extremely low content of metal traces recommended for the analysis of air-borne particles.

[Order Now](#)



Pathway Forward

Impinger Methods

- Slow
- Organic material not compatible with microwave digestion or running on ICP
- Very expensive and hard to clean

Smoke machines (EPT)

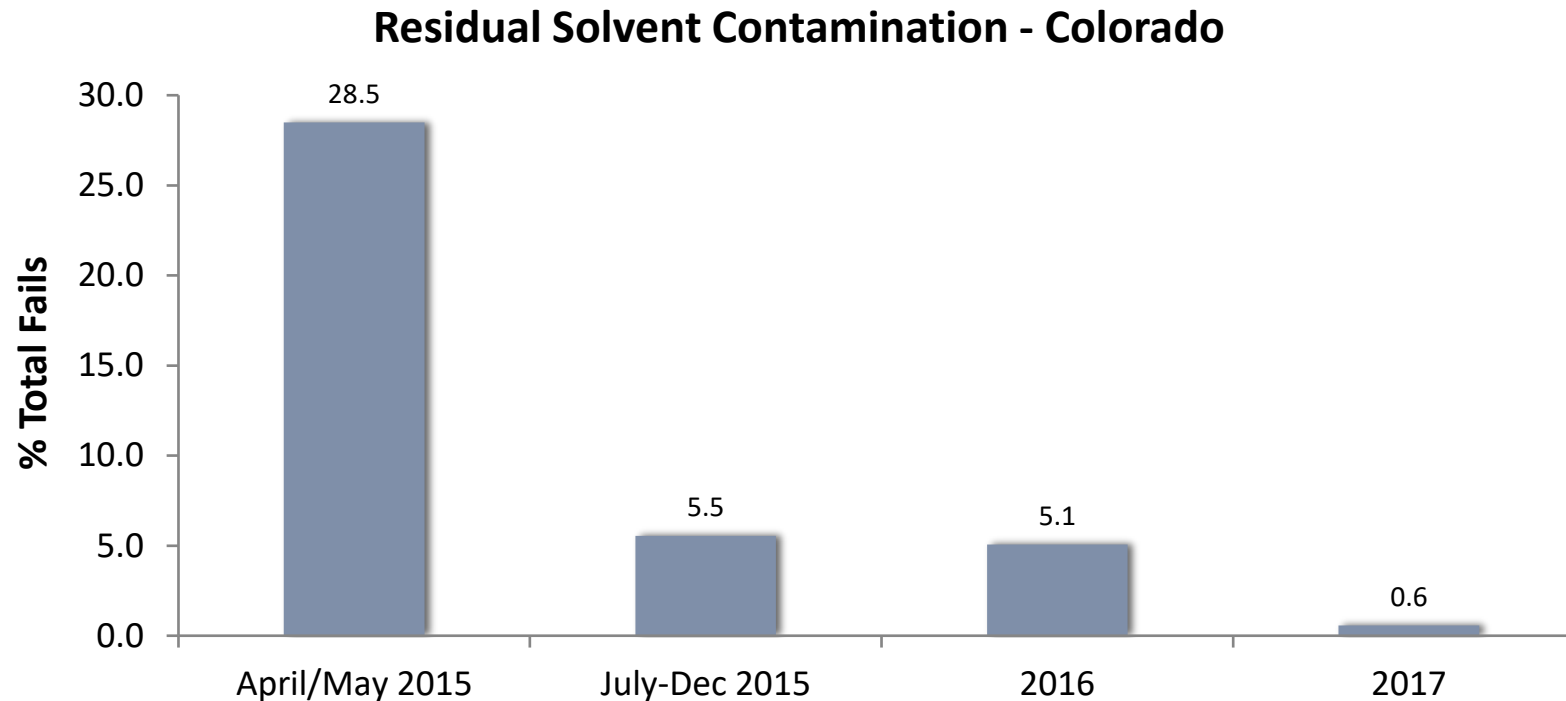
- No cannabis methods
- Viscosity issues?
- EPT vs Precipitation (cold) vs Filter Pad Capture?

Filter based methods

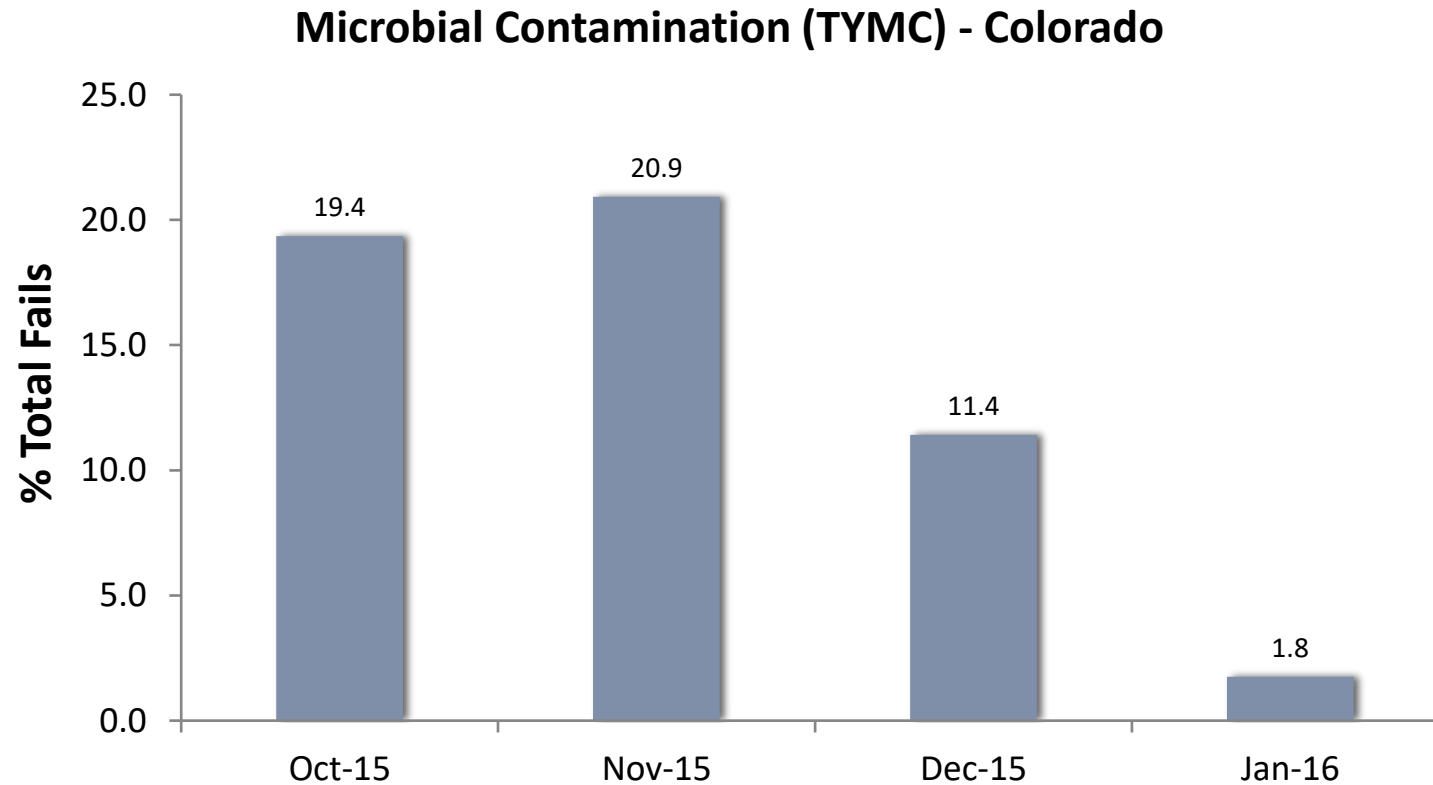
- Unproven
- Potentially cheap and simple
- Filters need to be custom made, in discussions



Fail Rates – Early Testing Colorado

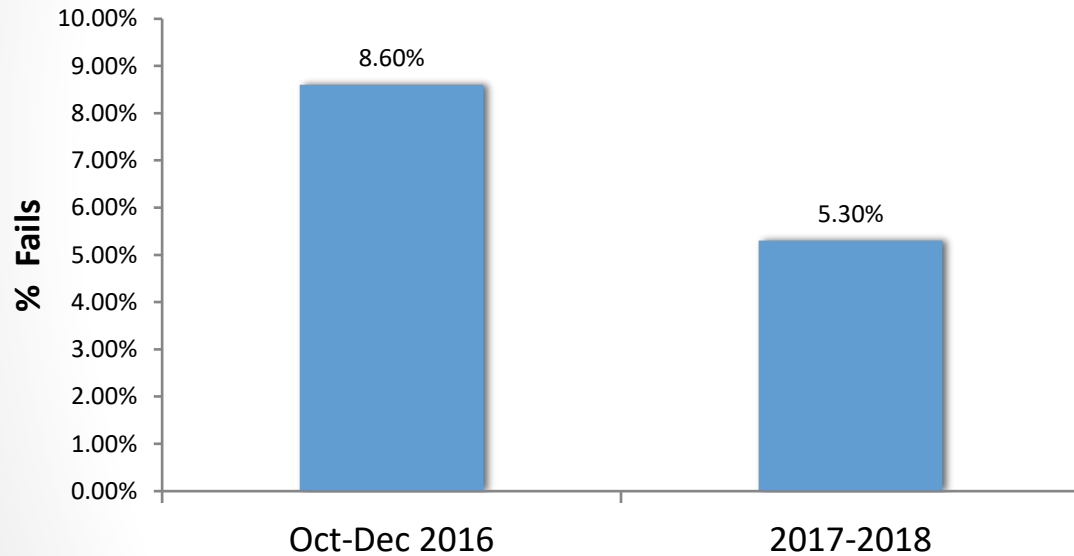


Fail Rates – Early Testing Colorado

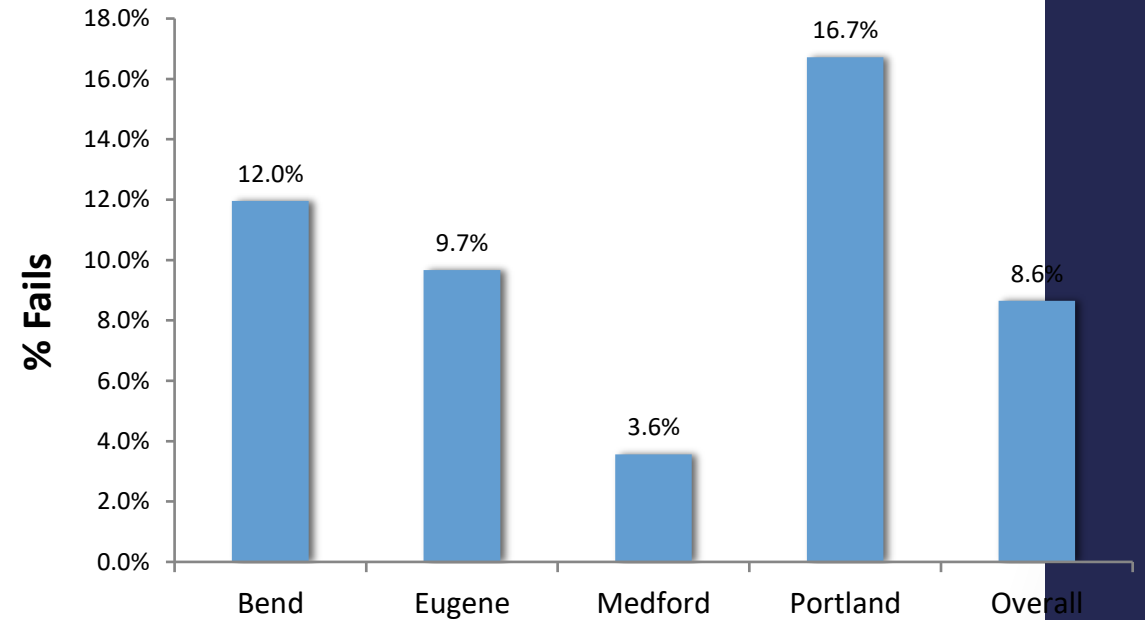


Fail Rates – Early Testing Oregon

Pesticide Fail Rates - Oregon

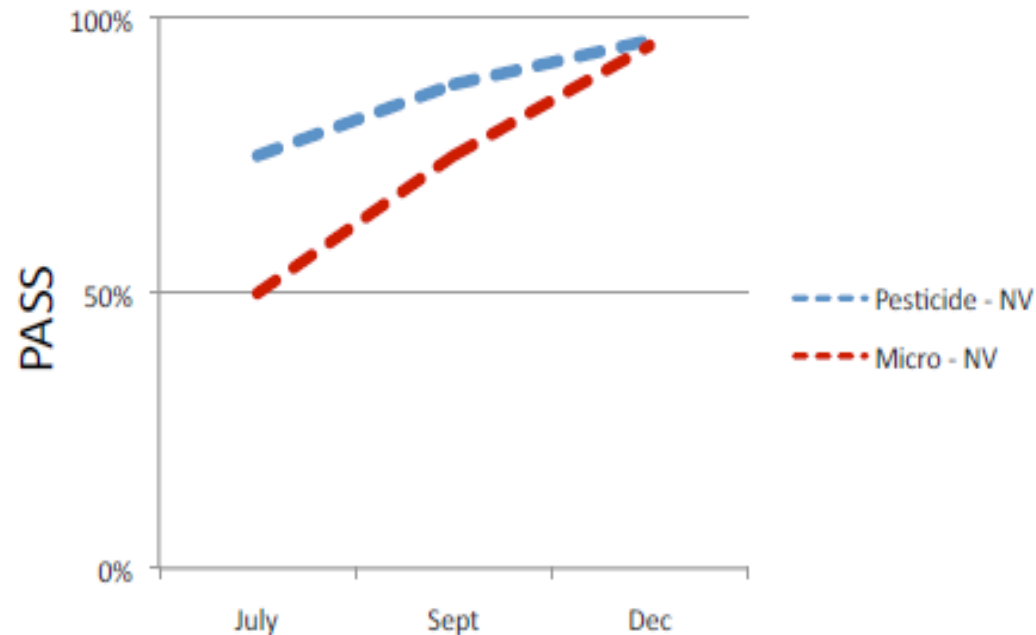


Pesticide Fail Rates - Oregon (2016)



Industry evolution

Testing Evolution in Nevada



Representation of trending P/F rates

- The impact on pass rates when testing regulations are instituted at time zero; versus being introduced years later....



Contact

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