Improving a Labs Turn Around Time and Pesticide Data Quality with Workflow Automation

Toby Astill, Ph.D.

Global Market Manager – Cannabis & Hemp

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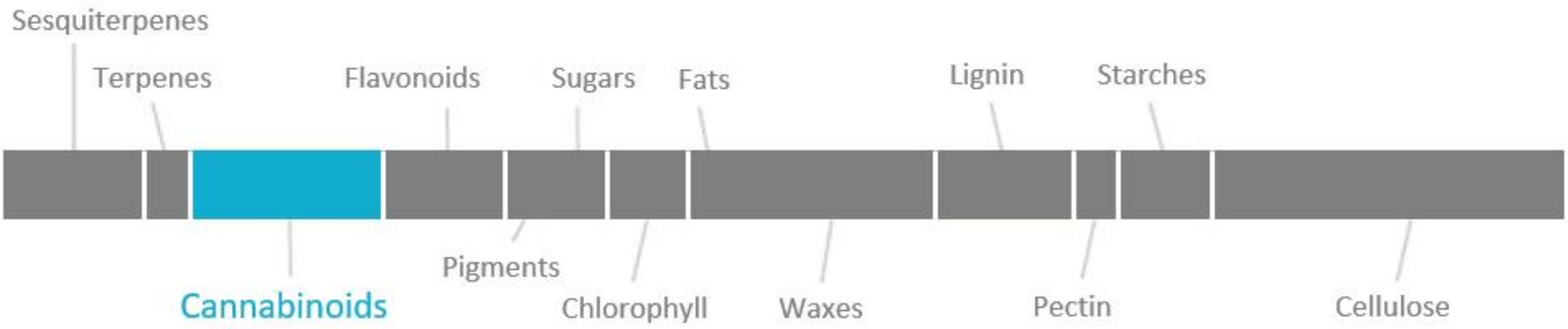
Cannabis Has Complex Chemistry



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Seed to Sale of Cannabis Products

Cultivation

Harvesting/
Trimming

Extraction

Distillation & Purification

Raw Ingredients

Infused Product

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Chemistry Overview

ChemDraw®

Cannabinoids

Cannabigerol, CBD, THC, Cannabichromene, Cannabinol, CBDA, THCA, CBDV, Tetrahydrocannabivarin

Terpenes

Phytol, Terpinolene, a-Bisabolol, Nerolidol, Menthol, Isoborneol, a-Pinene, a-Humulene, p-Cymene, Camphene, a-Terpinene, Eucalyptol, Linalool, ß-Caryophyllene, Valencene, Cedrene, Guaiol, Myrcene, Geraniol, Limonene, Caryophyllene Oxide, ß-Pinene, a-Phellandrene, Camphor, (+)-3-Carene

FLEXAR™ LC

Clarus® GC/MS with TurboMatrix™ Headspace

Residual Solvents

Ethyl acetate, Ethyl ether, 1,2-Dichloroethane, Heptane, Benzene, Chloroform, Butane, Ethanol, Methylene chloride, Propane, Hexane, Isopropyl alcohol, Ethylene oxide, Toluene, Total xylenes, Acetone, Acetonitrile, Trichloroethylene, Pentane, Methanol

THE CHEMISTRY OF CANNABIS

For years, PerkinElmer has worked with cannabis & hemp laboratories to develop industry-leading methods, technology, and exceptional return on investment. Whether your lab is well-established, or just starting up, having a single-source partner who can offer turnkey solutions that meet state and regional regulations is essential to a successful business.

Learn about our various testing methods and applications for cannabis analyses by visiting:
www.perkinelmer.com/cannabis
or calling: **1 (800) 762-4000**

QSight® 420 LC/MS/MS

NexION® 2000 ICP-MS

Heavy Metals

33 As Arsenic	48 Cd Cadmium
80 Hg Mercury	82 Pb Lead

Mycotoxins

Aflatoxin B1, Aflatoxin B2, Ochratoxin A, Aflatoxin G1, Aflatoxin G2

Pesticides

Piperonylbutoxide, Spiroxamine, Aldicarb, Daminozide, Dichlorvos, Dimethoate, Acephate, Spirotetramat, Methomyl, Prallethrin, Etofenprox, Fenoxycarb, Mevinphos, Acetamiprid, Thiacloprid, Tebuconazole, Myclobutanil, Naled, Oxamyl, Spiromesifen, Permethrin, Phosmet, Diazinon, Clofentezine, Bifenthrin, Imazalil, Methiocarb, Methyl Parathion, Paclobutrazol, Propoxur, Ethoprophos, Metalaxyl, Fludioxonil, Carbaryl, Kresoxim-methyl, Coumaphos, Carbofuran, Chlorfenapyr, Imidacloprid, Propiconazole, Chlorpyrifos, Malathion, Thiamethoxam, Flonicamid, Bifenazate, Acequinocyl, Fipronil, Azoxystrobin, Cyfluthrin, Etoxazole, Boscalid, Fenpyroximate, Chlorantraniliprole, Hexythiazox, Pyridaben, Captan, Spinovsad, Pentachloronitrobenzene, Trifloxystrobin, Chlordane, Dimethomorph, Spinetoram, Abamectin, Cypermethrine

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Part 1: Pesticide Analysis in Cannabis is Challenging

- Complex matrix associated
- Broad Interference from Cannabinoids present in wt % range.
- Terpenes and other non-cannabinoids compounds are present in high ppm
- Pesticides in 1.0 -1000 ppb

STANDARD OPERATING PROCEDURES

A Practical Approach to the Analysis of Pesticides & Mycotoxins in Cannabis Extract for California & Oregon QSight 420

HIGHLIGHTS

Equipment & Standards on page 2
Experimental Procedure on page 4
LC/MS/MS Configuration on page 7
Method Validation & Typical Results on page 10
Appx: Sample Recovery Data on page 16
Appx: Recommended Internal Standards on page 19

Recently, demand for the accurate quantification of pesticides in cannabis has increased, particularly among cannabis industry stakeholders and government regulatory institutions. This is due to both the complexity of preparing the cannabis matrix for pesticide analysis, and the increasingly stringent pesticide limits for cannabis products in certain states.

This document is intended to prepare your lab personnel to perform the series of analytical methods necessary for the complete analysis of pesticides in cannabis extracts and concentrates for California and Oregon. The methods described here will ensure that your lab attains the limits of quantitation specified for each relevant pesticide and mycotoxins below prescribed action limits by California and Oregon State.

The procedures included provide for the effective, reliable, and accurate quantitation of all pesticides listed by the *Bureau of Cannabis Control* (the proposed text of the regulations for California and Oregon). **The analytical methods target pesticides and mycotoxins in cannabis extract using the PerkinElmer LX-50 LC (*Liquid Chromatograph*) for effective separation and QSight® 420 tandem MS (*Mass Spectrometer*) for accurate quantitation.**

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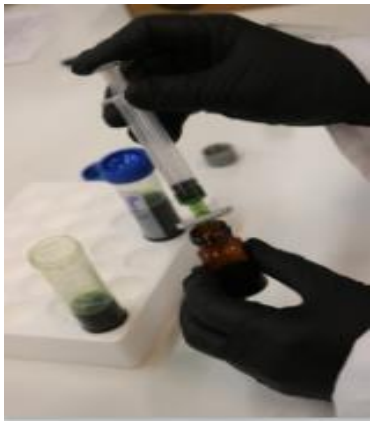
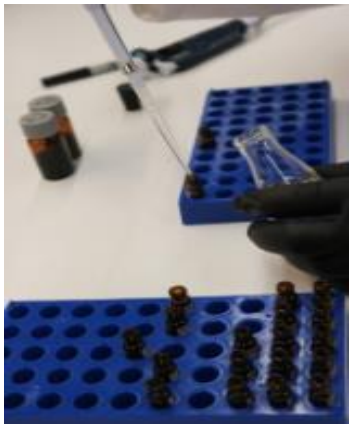
LC/MS Pesticide Method Prep





- No Quechers or No SPE Needed
- One instrument – QSight 420 LC/MS-MS

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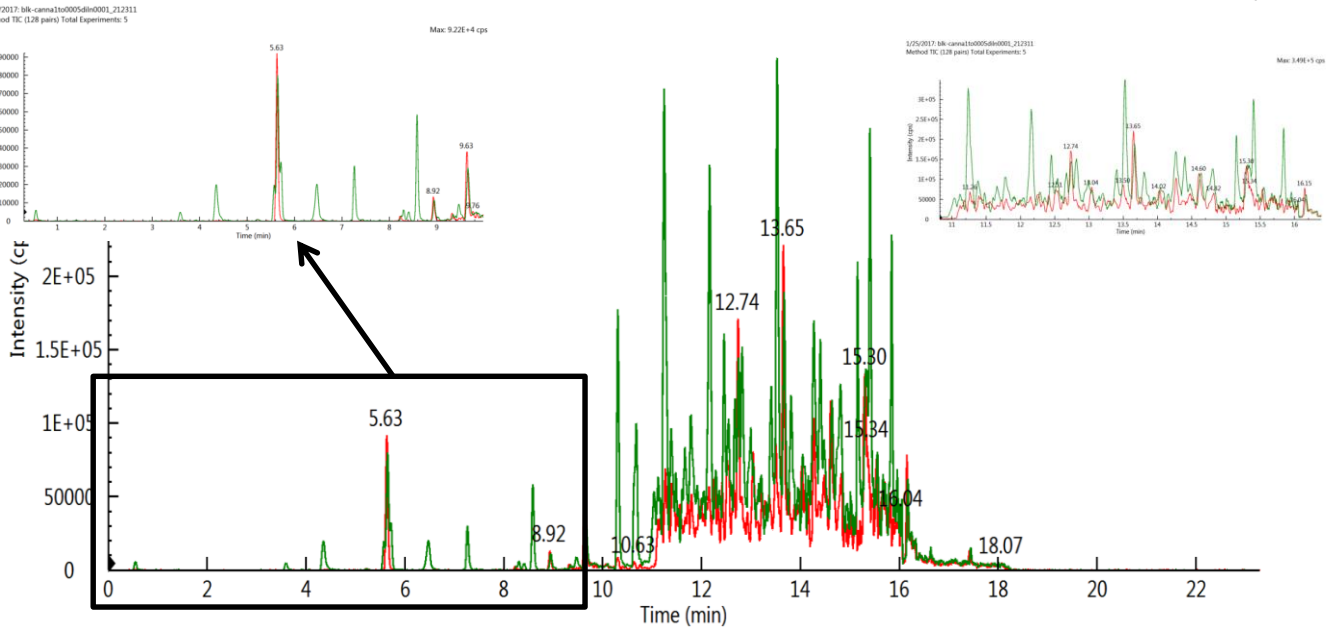
Influence of Cannabis Sample Matrix

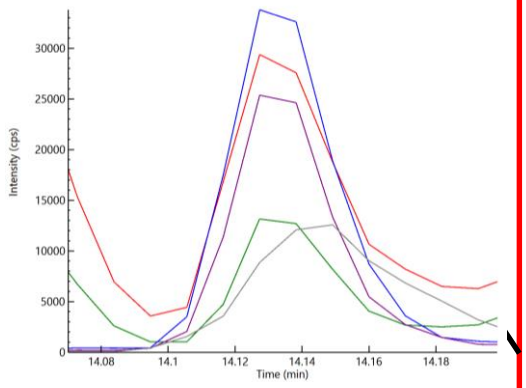
1/25/2017: blk-canna1to0005diln0001_212311
Method TIC (128 pairs) Total Experiments: 5

Cannabis extract (1 to 5 diluted) spiked with 25 ppb pesticide mix

Cannabis extract (1 to 5 diluted) Max: 2.36E+5 cps



TIC overlay shows easy detection of 25 ppb spike of pesticides mix in cannabis extract



Green – Distillate sample 1
Red – Distillate sample 2
Blue – Cannabis Flower
Purple – Hemp Edible
Grey – MCT oil with Concentrate

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Understanding Challenging Pesticides - PCNB

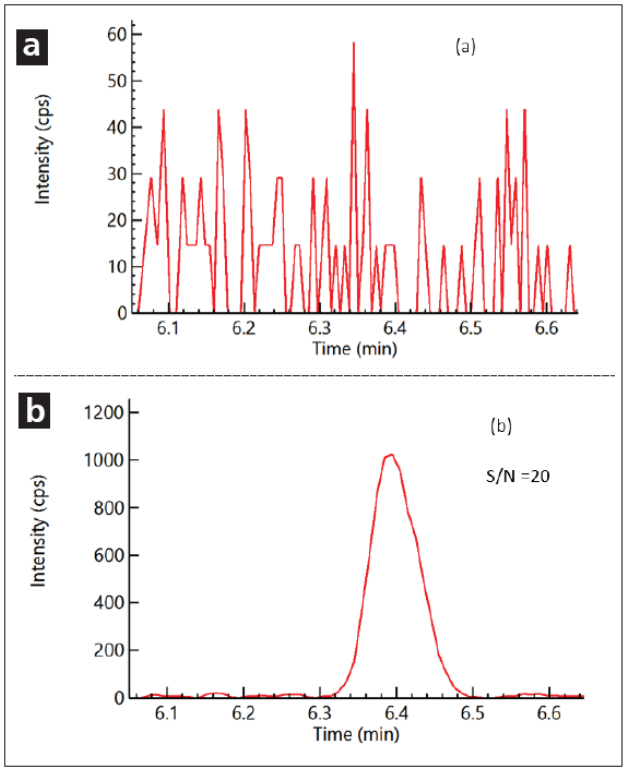
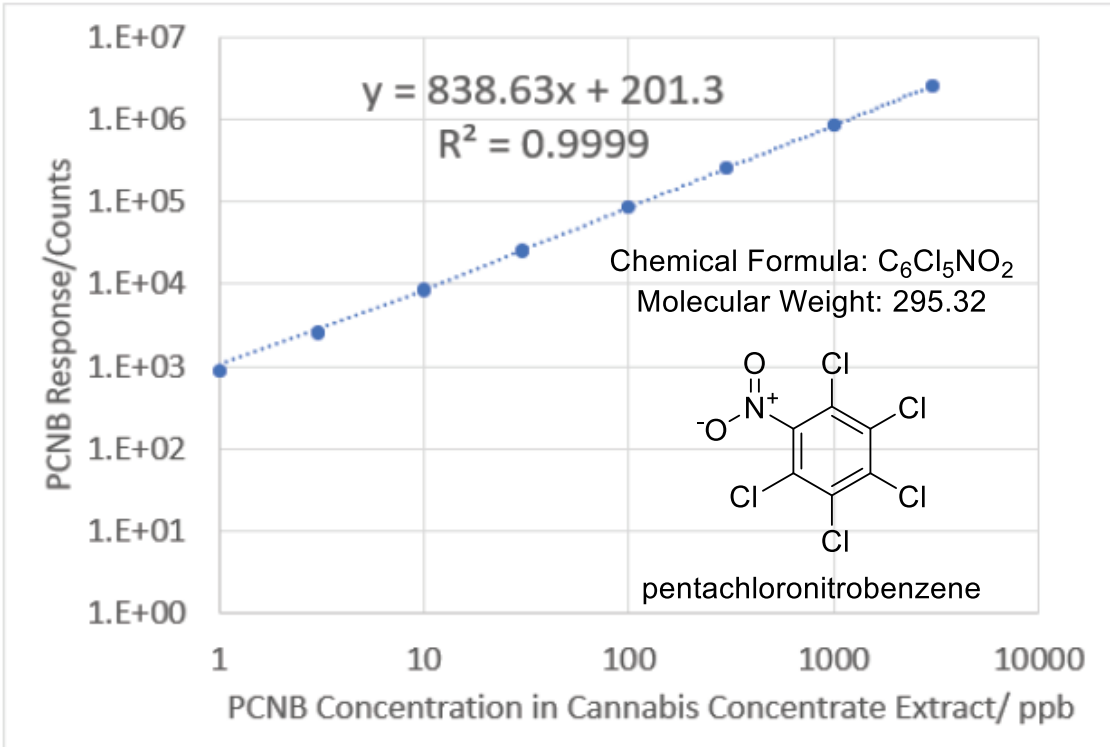


Figure 4. PCNB response in a blank cannabis concentrate matrix (a), and from spiked level of 0.1 µg/g in cannabis concentrate matrix (b).



- **Selective**
- **Linear**
- **Sensitive**

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Time

Data Quality

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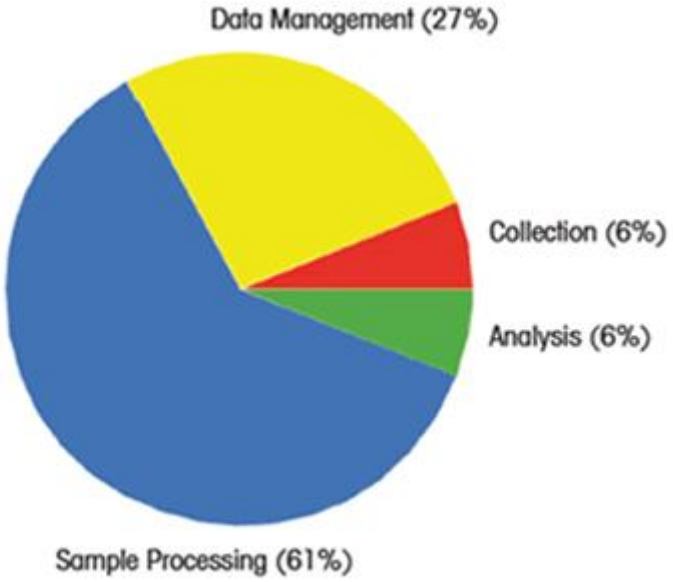
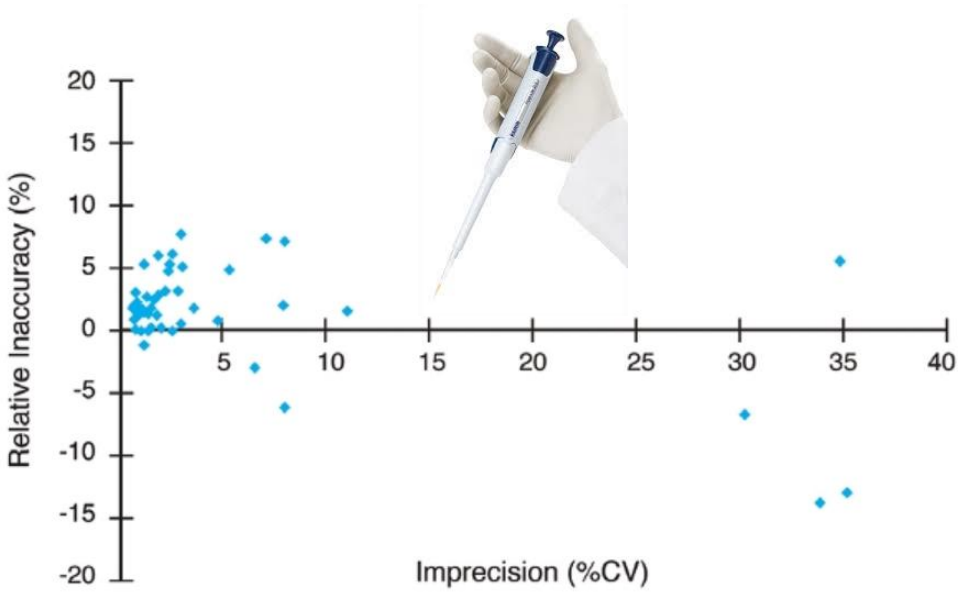
Sources of Experimental Variability

Time Spent in Lab



Pre-Training Pipette Skills Assessment[2]



1. https://www.mt.com/ca/en/home/perm-lp/product-organizations/labtec/Competence/sample_preparation.html
2. https://www.americanlaboratory.com/914-Application-Notes/156964-Improved-Pipetting-Technique-An-Out-of-the-Box-Solution-to-Reduce-Manual-Pipetting-Errors/

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Part 2: Adding Automation to improve Cannabis Testing

Janus Liquid Handler

GC/MS – Headspace

- **Residual Solvents**
- **Terpenes & Flavonoids**

HPLC

- **Potency**

LC-MS/MS

- **Pesticides**
- **Mycotoxins**

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Automation for cannabis testing Analytical Workflows

Potency

1-3 g ground material in 50 mL conical

↓

10-30 mL methanol

↓

Vortex 10 min

↓

Centrifuge 5-10 min

↓

Optional filter step over 0.22 µm (1-2 mL)

↓

Transfer 1:100 (10 µL filtrate + 990 µL methanol)

↓

Transfer to autosampler vial - HPLC

Pesticide Residue

1-3 g ground material in 50 mL conical

↓

Spike sample with 10 - 50 µL internal standard

↓

5-15 ml acetonitrile

↓

Vortex 10 min

↓

Centrifuge 5-10 min

↓

Optional filter step over 0.22 µm (1-2 mL) – transfer to glass vial

↓

Transfer 0.5 – 1 mL to autosampler vial and dilute 1:1 with acetonitrile – MS/MS

Mycotoxin

1-3 g ground material in 50 mL conical

↓

Spike sample with 10 - 50 µL internal standard

↓

5-15 mL acetonitrile

↓

Vortex 10 min

↓

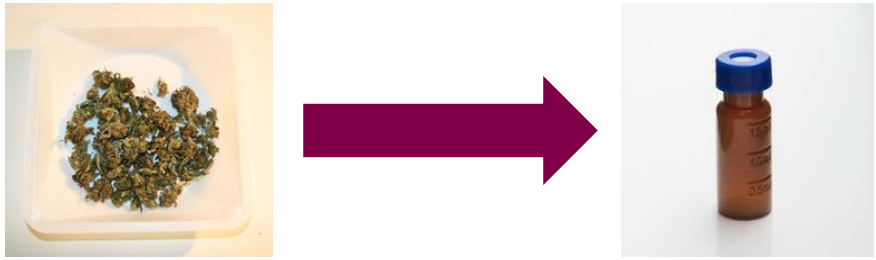
Centrifuge 5-10 min

↓

Optional filter step over 0.22 um (1-2 mL) – transfer to glass vial

↓

Transfer 0.5 – 1 mL to autosampler vial and dilute 1:1 with acetonitrile – MS/MS



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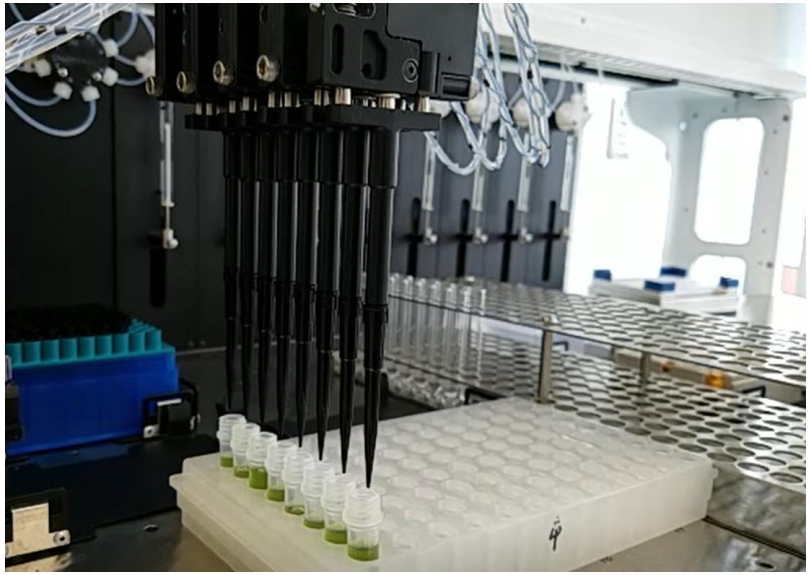
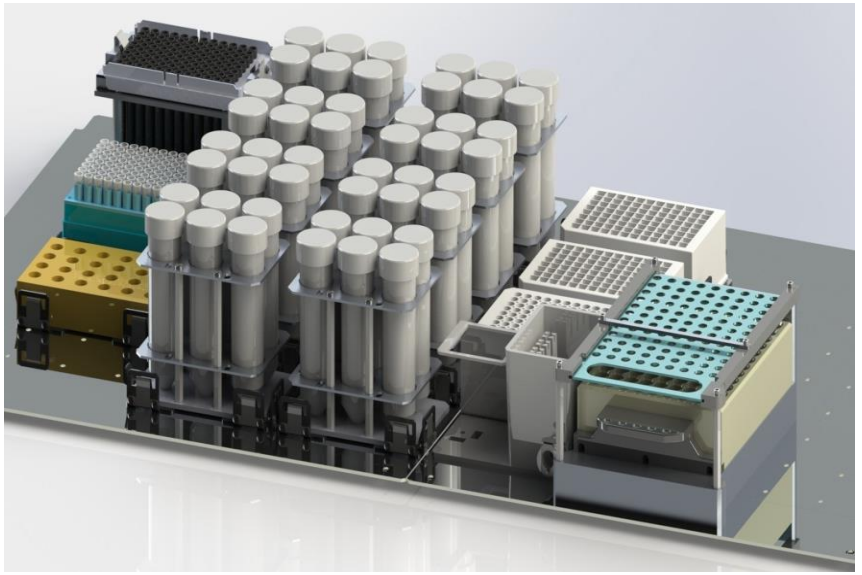
Automated sample prep for cannabis testing

Cannabis potency analysis · Pesticide analysis · Mycotoxin testing





- **Barcode Scanning**
- **High volume solvent addition**
- **Standard addition**
- **Serial Dilution**

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Time Savings

Pesticide and Mycotoxin

1 hour 7 min

48 Sample

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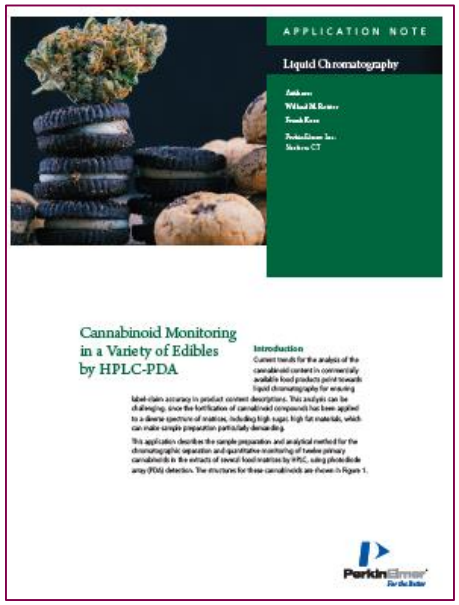
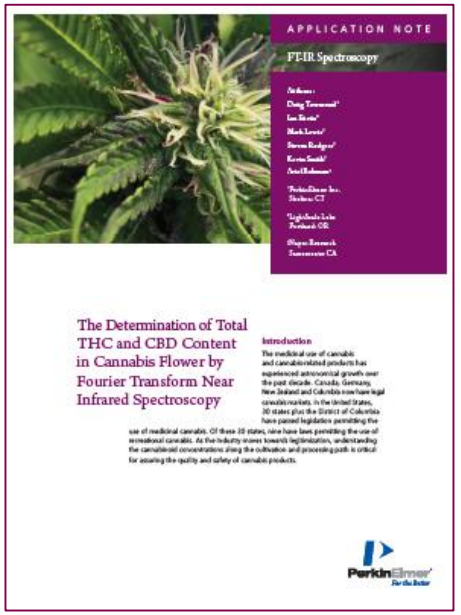
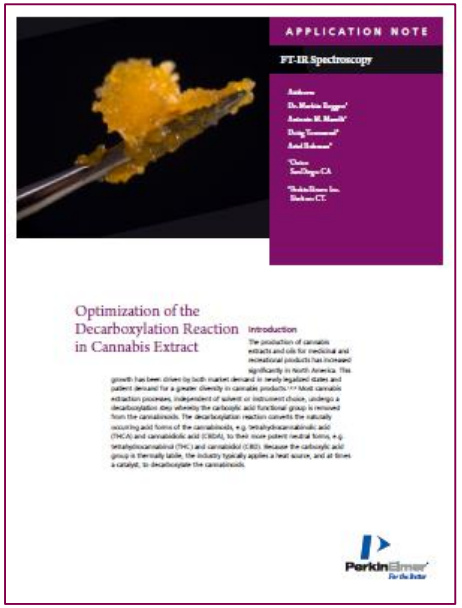
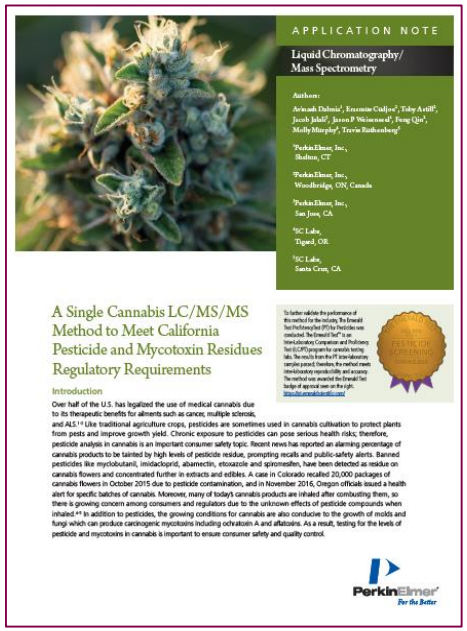
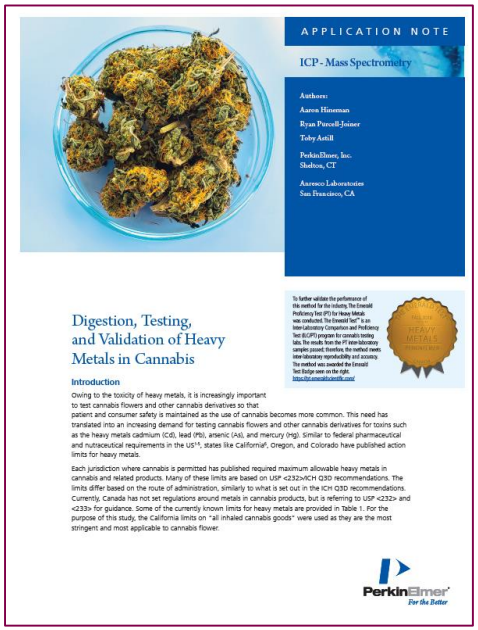
Available Application Collateral

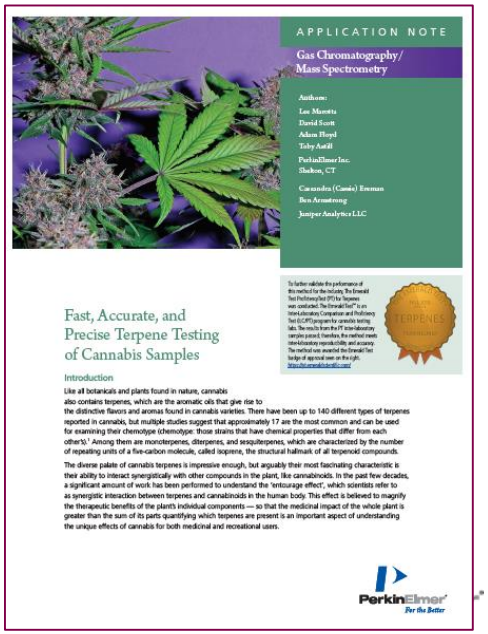












Summary

- Cannabis is a challenging matrix to test
  - Matrix heavily influences method
- LC/MSMS can be used to test for all pesticides in cannabis and hemp
- PerkinElmer's Automation improves data quality and TAT
- PerkinElmer is excited to work with the Scientific community to drive Cannabis & Hemp Science

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Q&A

Toby.Astill@perkinelmer.com

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