Simplifying Cannabis Pesticide, Mycotoxin and Potency Testing Workflows with Laboratory Automation to Meet ISO/GMP Standards

Toby Astill[1], Erasmus Cudjoe[1], Feng Qin[1], Sebastian Rakus[2], Avinash Dalmia[2]
[1]PerkinElmer, Woodbridge, Ontario, CA; [2]PerkinElmer, Waltham, MA, USA

1 Introduction

Currently, most laboratories, require multiple sample preparation steps to perform their pesticide residue assay. This workflow is time consuming, prone to errors due to the human variability, and poorly suited to high volume cannabis & CBD testing labs. To dramatically improve the labs 'Turn Around Time' PerkinElmer has developed a fully automated QS Works 420 pesticide and mycotoxin testing workflow. Herein, we present a fully automated and integrated analytical solution on the PerkinElmer Janus G3 420 sample preparation platform and QSight 420 LC/MS that provides sample traceability, complete SOP sample workup, analytical assay and compliance data reporting.

2 Janus 420 Automated Prep Station

PerkinElmer's JANUS® G3 420 workstation is optimized to automate **all** the pesticide SOP sample prep tests, including:

- Export Cannabis Flower sample weight from Micro Balance
- Spike Flower sample with 10 - 50 µL ONE Pesticide 420 and internal standard Kit
- Add Extraction solvent
- Transfer Sample Vial to Vortex Platform
- Transfer Sample Vial to Centrifuge
- Filter Sample and dilute 10X, 100X with acetonitrile for final transfer to 2ml LC/MS vial
- Export all sample prep data and LC/MS results to LIMs to finalize COA report

JANUS® G3 420 Workstation

5x Times Savings

Pesticide and Mycotoxin Preparation:

<2hrs for 48 Samples

3 Analytical Method

Chromatographic separation was carried out with a PerkinElmer® Quasar SPP Column (part#: N9306880) and the ONE Pesticide 420 CRM Reagent Kit which includes pesticide, mycotoxin, and internal standards.

ONE Pesticide 420 CRM Kit

Table 1: Experimental LC/MS conditions used for the pesticide residue analysis

LC CONDITIONS	
LC Column	Quasar SP Pesticides, 100 x 4.6 mm, 2.7µm
Mobile Phase A	2 mM ammonium formate with 0.1% formic acid in water
Mobile Phase B	2 mM ammonium formate with 0.1% formic acid in methanol
Mobile Phase Gradient	The optimized gradient elution method included analytical column re-conditioning was 18.5 min. The final method ensured separation of the bulk cannabis matrix from the analytes for improved quantitation.
Column Oven Temperature	30 ºC
Auto sampler Temperature	10 ºC
Injection Volume	3.0µL

4 Results and Discussion

All the pesticides studied showed good responses in cannabis samples with LOQs ranged from 0.0025-0.5 µg/g, which are well below the California action limit. Eight point-Calibration curves (Figure 1) were developed from both neat solution (solvent) and cannabis sample blank matrix (matrix-matched calibration). All the calibration curves showed good linearity with correlation coefficient (r^2) larger than 0.99 for all pesticides including the GC amenable pesticides.

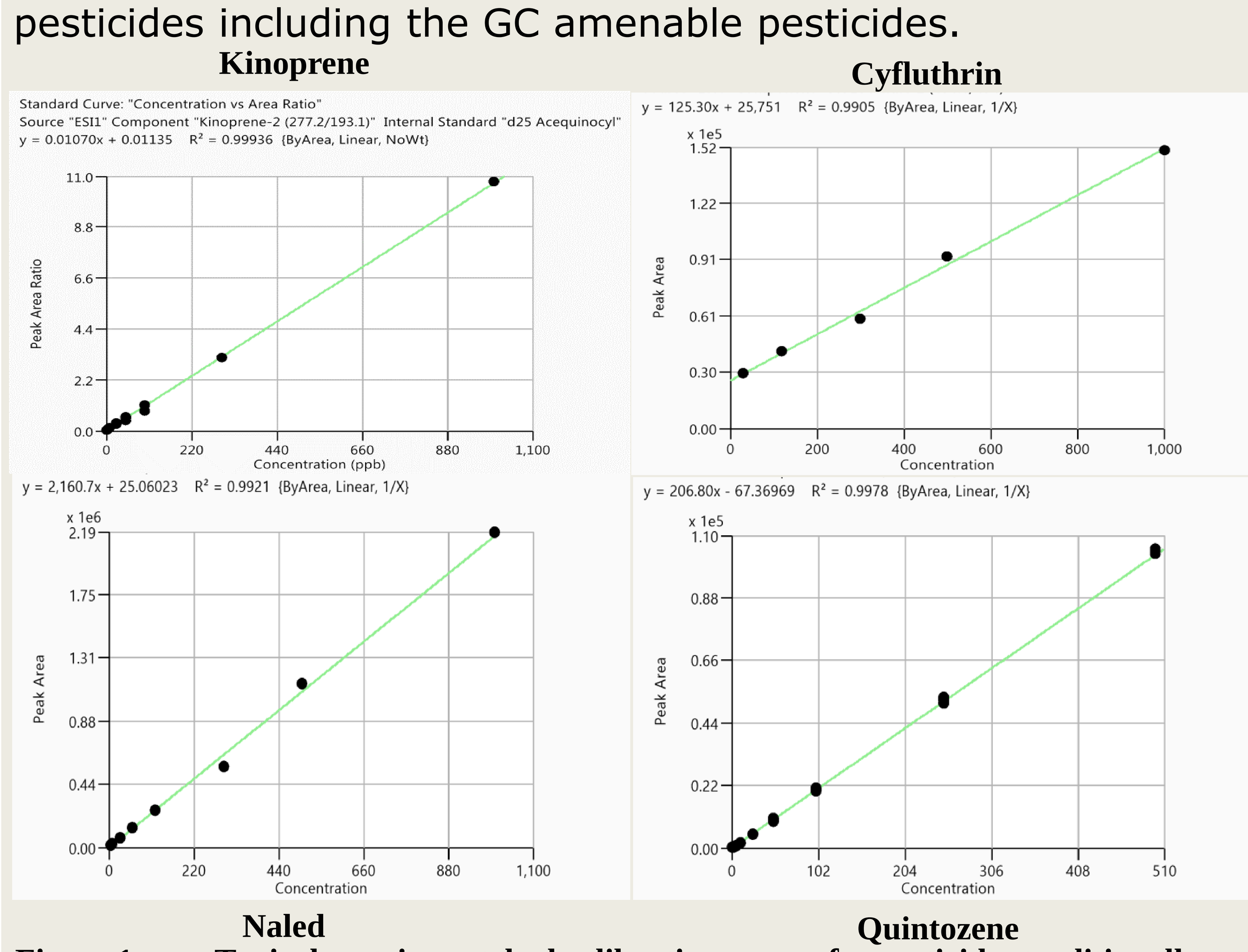


Figure 1. Typical matrix-matched calibration curves for pesticides traditionally GC amenable but carried out with PerkinElmer LC-MS/MS

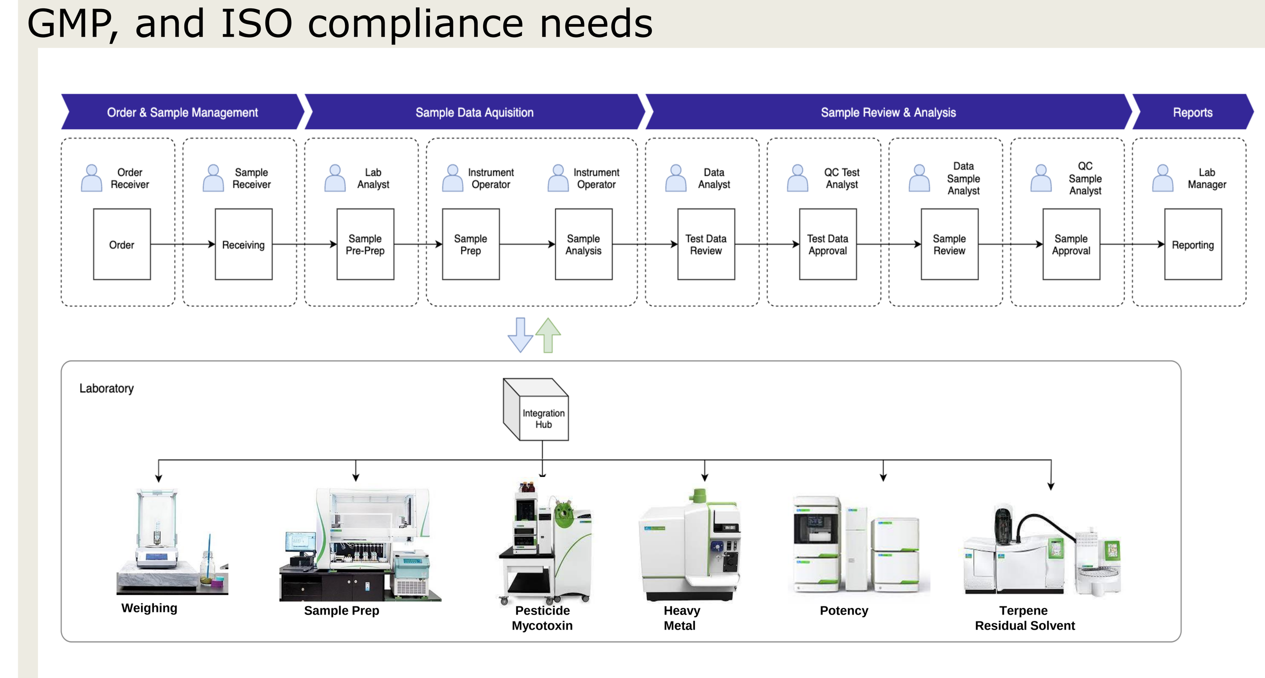
The limit of quantitation (LOQ) for each pesticide was determined in the cannabis matrix. LOQs for all pesticides met the California action limits in cannabis flower matrix(results shown in next section).

Table 2: **Recovery for Pesticides with LC-MS/MS in Cannabis.**
***: Pesticides typically analyzed by GC-MS/MS were analyzed by APCI or ESI on LC/MSMS**

Analyte	Average Recovery/%	RSD/%
Abamectin	78.1	8
Acephate	86.6	4
Acequinocyl	91.1	12
Acetamiprid	119.2	3
Aldicarb	110.7	9
Azoxystrobin	84.1	5
Bifenazate	123.4	2
Bifenthrin	109.9	4
Boscalid	83.1	6
Captan	101.9	18
Carbaryl	113.8	2
Carbofuran	114.2	5
Chlorantraniliprole	115.5	5
Chlordane*	129.1	8
Chlorfenpyr*	73	3
Chlorpyrifos	103.4	15
Clofentezine	97	10
Coumaphos	104.7	5
Cyfluthrin	98.3	13
Cypermethrin	97.5	9
Daminozide	77.2	5
Dimethomorph	92	6
DDVP (Dichlorvos)	110.3	3
Diazinon	84.7	5
Dimethoate	104.9	2
Ethoprophos	97.7	3
Etofenprox	111.6	2
Etoxazole	93.1	5
Fenoxycarb	105.6	3
Fenpyroximate	100	5
Fenhexamid	88.5	6
Fipronil	97	5
Flonicamid	112.7	3
Fludioxonil	119.4	5
Hexythiazox	93.3	5
Imazalil	116.4	4
Imidacloprid	107.1	3
Kresoxim-methyl	82.6	3
Malathion	108.4	2
Metalaxyl	107.5	4
Methiocarb	100.3	6
Methomyl	89.5	6
Methyl parathion*	105.8	2
Mevinphos	80	5
MGK-264	94.6	5
Myclobutanil	111.9	4
Mycotoxin B1	87	5
Mycotoxin B2	76.8	4
Mycotoxin G1	100.5	4
Mycotoxin G2	76.7	4
Naled	94.6	8
Ochratoxin A	96.4	5
Oxamyl	87.3	5
Paclobutrazol	116	2
PCNB*	105.9	4
Permethrins	103.4	3
Phosmet	107.3	6
Piperonyl Butoxide	90.5	13
Prallethrin	103.8	4
Propiconazole	123.5	4
Propoxur	106.2	3
Pyrethrins	123.1	3
Pyridaben	91.2	4
Spinetoram	82.3	5
Spinosads	74.6	5
Spiromesifen	90.5	5
Spirotetramat	106.8	2
Spiroxamine	108.9	6
Tebuconazole	109.6	6
Thiacloprid	105.9	3
Thiamethoxam	87.2	3
Trifloxystrobin	80.1	4

Note: The compounds analyzed using LC/MS/MS with APCI source are indicated using an asterisk (*). The rest of the compounds marked with no asterisk were determined using a LC/MS/MS with ESI source.

Data was managed across the entire workflow using SimplicityLab420 SaaS Cloud software. SimplicityLab 420 integrates data across all instruments to allow scientists to process more samples per shift and maintain the highest data quality. Data is exported and downloaded in a key sequence driven by the protocol, allowing individual platform elements to perform their tasks in a predefined controlled manner, it's then uploaded and stored in the cloud for remote access 24/7 or exported to METRC or the laboratory LIMs. The software reduces error and has architecture that supports CFR Part 11, GMP, and ISO compliance needs



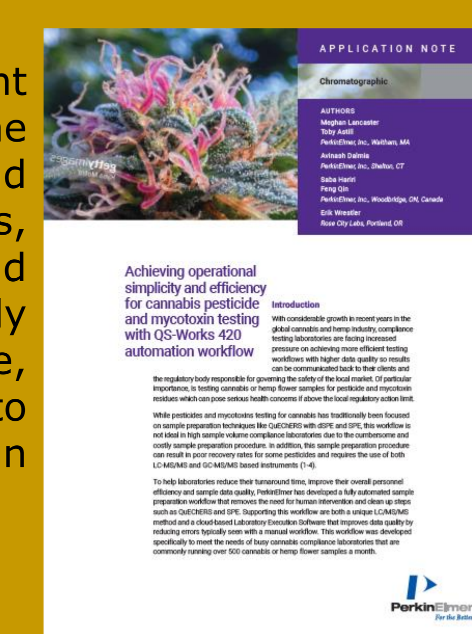
SimplicityLab420 SaaS SW Architecture Overview

6 Conclusion

This study demonstrates a unique, fully automated, quantitative, rapid, and reliable workflow solution for cannabis pesticide residue analysis. The addition of a JANUS 420 workstation offers the following:

- Removes the need for human intervention during the sample preparation, reducing potential for manual sample preparation handling errors
- Increases sample throughput per shift
- Pre-formatted ISO17034 Calibration and Internal Standard Kit
- Improves compliance with complete sample traceability

In addition, PerkinElmer's QSight 420 LC/MSMS method gives the analyst the ability to screen and quantitate all 96 pesticides, including the very hydrophobic and chlorinated compounds normally analyzed on a GC-MS/MS amenable, makes this method a novel way to screen and quantitate pesticides in cannabis with a single instrument.



For more information contact:

Toby.Astill@perkinelmer.com