Application Note

Cannabis Quality Testing, Food Safety

Agilent

Trusted Answers

Determination of Pesticides and Mycotoxins as Defined by California State Recreational Cannabis Regulations

A combined LC/MS/MS analysis method

Authors

Peter JW Stone, Jennifer Hitchcock, Jean-Francois Roy, and Christophe Deckers

Agilent Technologies, Inc.

Introduction

This Application Note details an LC/MS/MS analytical workflow developed by Agilent for the accurate measurement of the California State combined pesticide and mycotoxin action lists[1]. The workflow illustrates sample preparation and analysis techniques uniquely applied to cannabis flower through to data review and reporting.

Since the sanctioning of recreational cannabis use in various U.S. States in recent years, respective lawmakers have introduced unique State legislation. This State legislation details minimum acceptable levels of specific pesticides and mycotoxin content allowed in potential retail material. Table 1[1] summarizes the specific requirements for pesticide and mycotoxin limits in cannabis flower in California.

Table 1. The California list of pesticides and mycotoxins, and the defined action (not to exceed) levels. No Category I pesticide can be present at a concentration greater than the empirically determined limit of detection (LOD). This value is defined as *>LOD* in the table.

Target list	Action level, ng/g (ppb)
Avamectin B1a	100
Avamectin B1b	100
Acephate	100
Acequinocyl	100
Acetamiprid	100
Aldicarb	>LOD
Azoxystrobin	100
Bifenazate	100
Bifenthrin	3,000
Boscalid	100
Captan	700
Carbaryl	500
Carbofuran	>LOD
Chlorantraniliprole	10,000
Chlordane	>LOD
Chlorfenapyr	>LOD
Chlorpyrifos	>LOD
Clofentezine	100
Coumaphos	>LOD
Cyfluthrin	2,000
Cypermethrin	1,000
Daminozide	>LOD
Diazinon	100
DDVP (Dichlorvos)	>LOD

Target list	Action level, ng/g (ppb)
Dimethoate	>LOD
Dimethomorph 1	2,000
Dimethomorph 2	2,000
Ethoprop(hos)	>LOD
Etofenprox	>LOD
Etoxazole	100
Fenhexamid	100
Fenoxycarb	>LOD
Fenpyroximate	100
Fipronil	>LOD
Flonicamid	100
Fludioxonil	100
Hexythiazox	100
Imazalil	>LOD
Imidacloprid	5,000
Kresoxim-methyl	100
Malathion	500
Metalaxyl	100
Methiocarb	>LOD
Methomyl	1,000
Methyl parathion	>LOD
Mevinphos	>LOD
MGK-264	NA
Myclobutanil	100

Target list	Action level, ng/g (ppb)
Dibrom Naled	100
Oxamyl	500
Paclobutrazol	>LOD
Pentachloronitrobenzene	100
Permethrin	500
Phosmet	100
Piperonyl butoxide	3,000
Prallethrin	100
Propiconazole	100
Propoxur	>LOD
Pyrethrin I	500
Pyrethrin II	500
Pyridaben	100
Spinetoram J	100
Spinetoram L	100
Spinosin A	100
Spinosin D	100
Spiromesifen	100
Spirotetramat	100
Spiroxamine	>LOD
Tebuconazole	100
Thiacloprid	>LOD
Thiamethoxam	5,000
Trifloxystrobin	100

Experimental

Materials and reagents

Pesticide and mycotoxin standards: Pesticide mixes representative of respective U. S. States were obtained from LGC USA at a concentration of 100 µg/mL, as were the mixed aflatoxin standards (B1, B2, G1, and G2). The ochratoxin A standard was obtained at a concentration of 2 µg/mL.

Other reagents

- LC/MS grade methanol, Alfa Aesar (Ward Hill, Massachusetts, USA)
- Millipore deionized water >18.2 mOhm, MilliporeSigma (Burlington, Massachusetts, USA)
- Formic acid (97+ %), Sigma-Aldrich (St. Louis, MO, USA)
- Ammonium formate (99+ %), Sigma-Aldrich (St. Louis, MO, USA)

Instrumentation

UHPLC: Although any Agilent UHPLC configuration can be used for this analysis, the following instruments were used:

- Agilent 1290 Infinity binary pump (G4220A)
- Agilent 1260 Infinity II multisampler, thermostatted, with 100-µL loop and multiwash options (G7167A)
- Agilent 1260 Infintiy II multicolumn thermostat (G7116A with 6-port/2-position valve option #058)

To offset any extra time required for the injection program, it is recommended for high-throughput environments to perform overlapped injections. These injections should be initiated specifically at 10.5 minutes, and started from the MassHunter worklist run parameters settings by checking the overlapped injection radio button. For this process to operate optimally, it is important to set the autosampler configuration for a 100-µL loop and 100-µL metering device.

California MRM parameters are detailed in Appendix A for Agilent 6470 (G6470AA) and Agilent Ultivo (G6465BA) units. All fragmentor voltage (Frag) settings, respective collision energies (CE), and most abundant/appropriate MS/MS product ions per analyte were determined and obtained using the Agilent MassHunter Optimizer software.

Sample preparation protocol for LC/MS triple quadrupole analysis

1. One gram of chopped organic cannabis flower was transferred to a 50-mL polypropylene centrifuge tube.
2. Two ceramic homogenizers (p/n 5982-9313) or stainless-steel beads were also placed in the tube, which was then capped.
3. The tube was shaken mechanically for 2–5 minutes at high speed (vertical shaking on a Geno/Grinder-type machine) turning the plant content into fine powder.
4. (For prespiked samples only and recovery studies, the pesticide standard solutions were added to the 15 mL used in step 5 at the appropriate concentrations).
5. Fifteen milliliters of LC/MS-grade acetonitrile was added to the tube from step 3.

UHPLC method conditions

Parameter	Value
Column	Agilent InfinityLab Poroshell 120 Phenyl-Hexyl, 3.0 × 100 mm, 2.7 µm bead size (p/n 695975-312)
Guard column	Agilent InfinityLab Poroshell 120 Phenyl-Hexyl, 2.1 × 5 mm, 2.7 µm bead size (p/n 821725-914)
Column temperature	55 °C
Injection volume	10 µL (with injector program/pretreatment, see Table 2)
Autosampler temperature	4 °C
Needle wash	Flush port (100 % methanol), five seconds
Mobile phase	A) 5 mM ammonium formate/0.1% formic acid in water B) 0.1% formic acid in methanol
Gradient flow rate	0.5 mL/min
Gradient	Time (min) %B 0.00 30 1.00 30 2.00 75 8.00 96 9.00 100 9.50 100 9.51 30
Analysis and re-equilibration time	11 minutes
Total run time (sample to sample)	11 minutes

Table 2. Injector program/pretreatment.

Step	Action	Description
1	Draw	Draw 10 µL from location 1 with default speed using default offset (100 % deionized water)
2	Draw	Draw default volume from the sample with default speed using default offset
3	Wash	Wash needle in flush port for five seconds (100 % methanol)
4	Draw	Draw 10 µL from location 1 with default speed using default offset (100 % deionized water)
5	Mix	Mix 30 µL volume from air with maximum speed five times
6	Inject	Inject

Mass spectrometer configuration and conditions

Parameter	Value
Configuration	6470 or Ultivo triple quadrupole mass spectrometer equipped with Agilent Jet Stream (AJS) ESI source
Ion source conditions	
Ion mode	AJS ESI, positive and negative polarities
Capillary voltage	5,000 V
Drying gas (nitrogen)	13 L/min
Drying gas temperature	200 °C
Nebulizer gas (nitrogen)	55 psi
Sheath gas temperature	200 °C
Sheath gas flow	10 L/min
Nozzle voltage	500 V
Q1 and Q2 resolution	0.7 amu [autotune]
Delta EMV	0 V

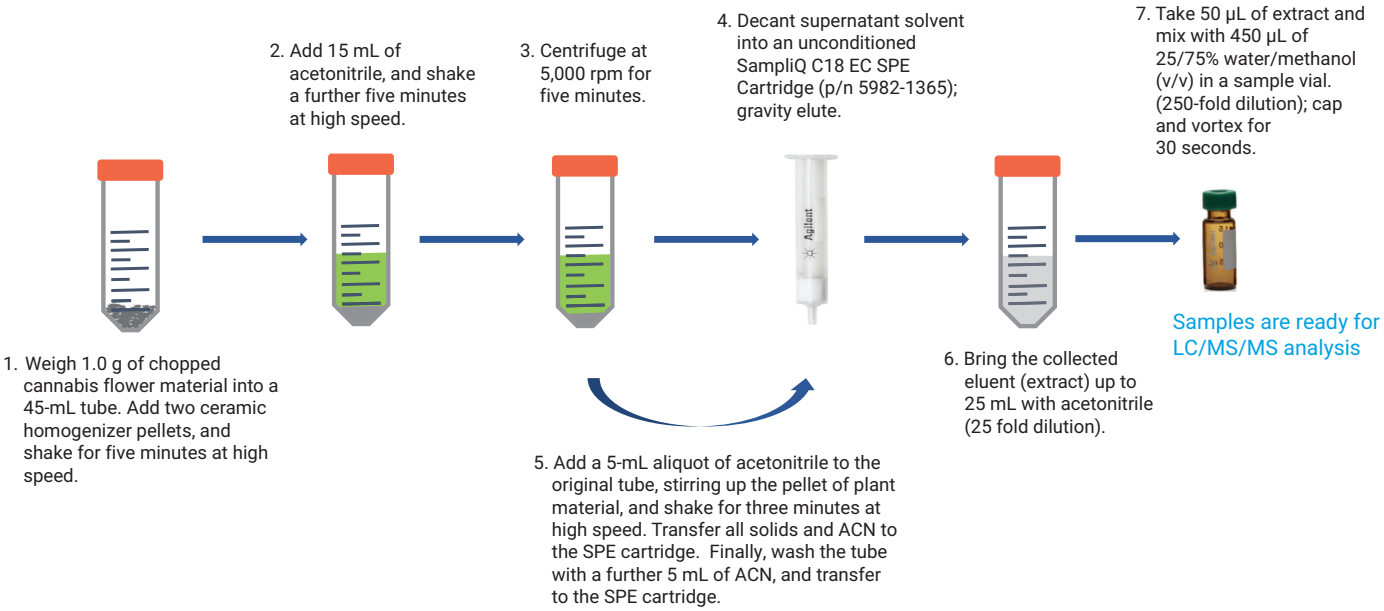
6. The tube and its contents were once more shaken mechanically for five minutes at high speed (vertical shaking on a Geno/Grinder). This shaking was for the extraction of pesticides and aflatoxins into the acetonitrile.
7. The tube was then centrifuged at 5,000 rpm for 10 minutes, and the supernatant transferred to a fresh vessel.
8. While the tube was centrifuged, the extraction manifold was prepared by placing a SampliQ C18 EC 6 mL, 500 mg solid phase extraction (SPE) cartridge (p/n 5982-1365) on the SPE manifold. To collect the cleaned-up eluent, a collection tube of 25 mL or more capacity was placed underneath the cartridge.
9. The supernatant from step 7 was decanted into the SampliQ C18 SPE cartridge. Flow through the cartridge was by gravity. When all solvent had completely passed through the C18 cartridge, the tube and plant pellet from step 7 was mixed with 5 mL of acetonitrile. The pellet was then agitated to bring it into a suspension once again, and was shaken for three minutes. The contents of the tube were then poured into the same C18 SPE cartridge, and the cleaned eluent collected. A further 5 mL of acetonitrile was added to the empty tube, vortexed for 30 seconds, and added to the SPE cartridge. This resulted in just under 25 mL volume of cleaned acetonitrile extract, which was made up to 25 mL using the graduations on the outside of the tube.
10. Fifty microliters of eluent from step 9 were added to 450 µL of water/methanol (25 %/75 % v/v) containing 0.1 % formic acid in a 2-mL sample vial, and capped.
11. This 10× dilution was vortexed for 20 seconds, and was then ready for LC/MS injection.
12. For samples, this solution was injected directly into the LC/TQ. For matrix calibrations or post extraction recovery studies, the desired amounts of pesticide and mycotoxins were spiked into the solution at this point.

The sample preparation steps outlined constitute a resultant 1/250 total dilution, and are outlined schematically in Figure 1.

Results and discussion

Matrix extract calibration standards were prepared down to low part per trillion (ppt) actual levels. This was so the lower limits of quantitation (LLOQ) could be determined and related back to the legislative requirements for California. This was necessary since the outlined sample preparation routine effectively dilutes the original plant material by effectively 250×. Given that California limits are effectively 100 ppb and higher, depending on the analyte concerned, the instrumental detection lower limits would need to be lower than 200 ppt for the most challenging analytes.

Figure 2 illustrates the California pesticide mix spiked into matrix, and each analyte overlaid together with aflatoxins B1, B2, G1, G2, and ochratoxin A at an actual concentration of 500 ppt, relating to an original pre-extraction concentration of 125 ppb.



These sample preparation steps constitute a resultant 1/250 total dilution.

Figure 1. LC/MS sample preparation—cannabis flower SPE cleanup and dilution (California).

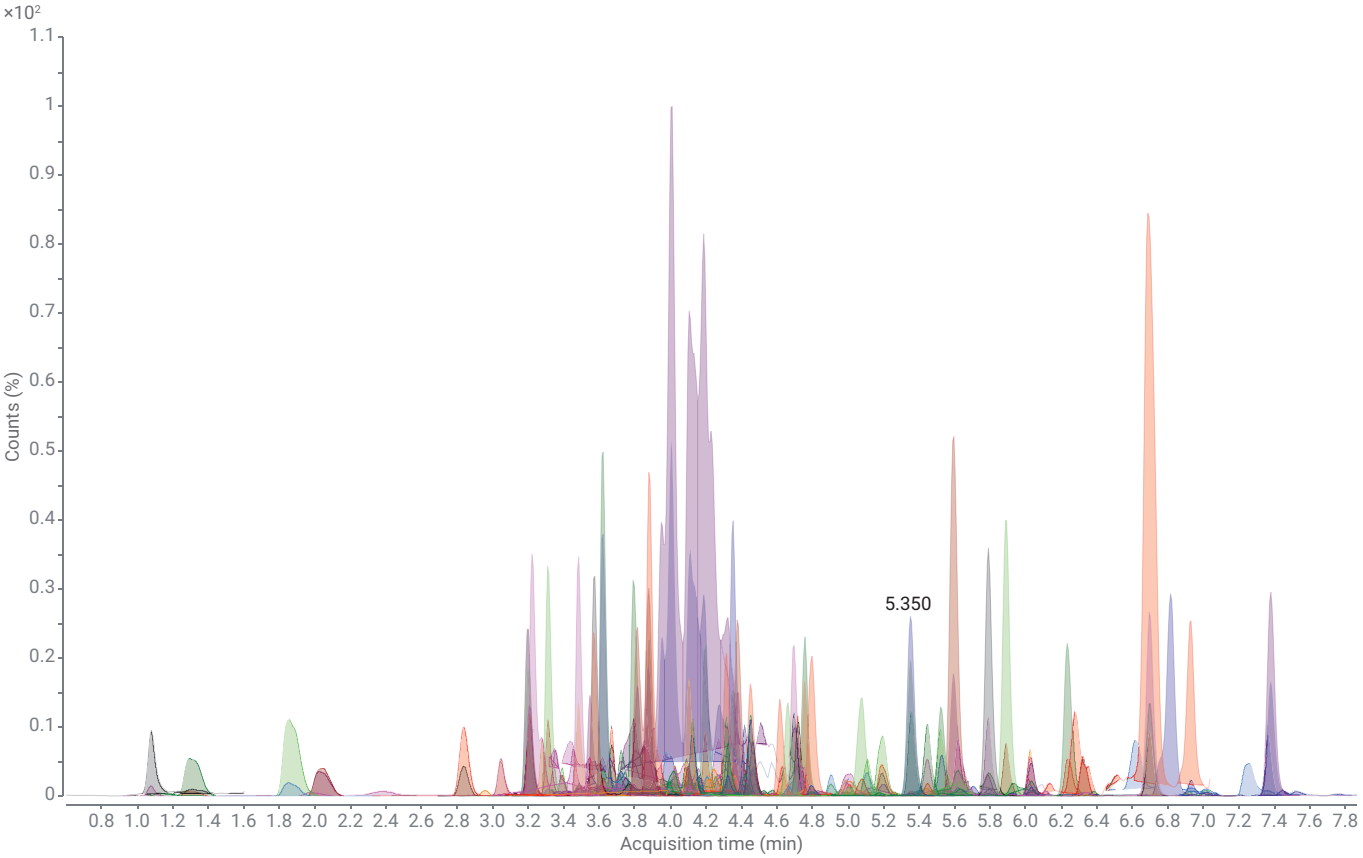


Figure 2. Overlaid chromatograms of California pesticides list and mycotoxins in extracted flower matrix, actual concentration 500 ppt (pre-extraction concentration = 125 ppb.)

Typical matrix calibration curves and LLOQ chromatography observed and obtained through this sample preparation routine are illustrated in Figures 3 and 4 (A, B, and C), respectively. Linear correlation values (R^2) for the spiked pesticides and mycotoxins were 0.990 or higher.

Table 3 outlines typical LLOQ results for pesticides obtained from multiple batches of cannabis flower prepared as outlined in the sample preparation section of this Application Note. The table for the California action list contains four analytes, which need to be analyzed using GC/MS/MS techniques due to a lack of functional groups required to invoke a true molecular ion adduct through LC/MS/MS. These are labeled in the LLOQ column as *GC/MS*, and Reference 2 outlines the techniques and methods required to analyze them. Table 4 summarizes the typical LLOQ values obtained for mycotoxins listed with Californian action levels.

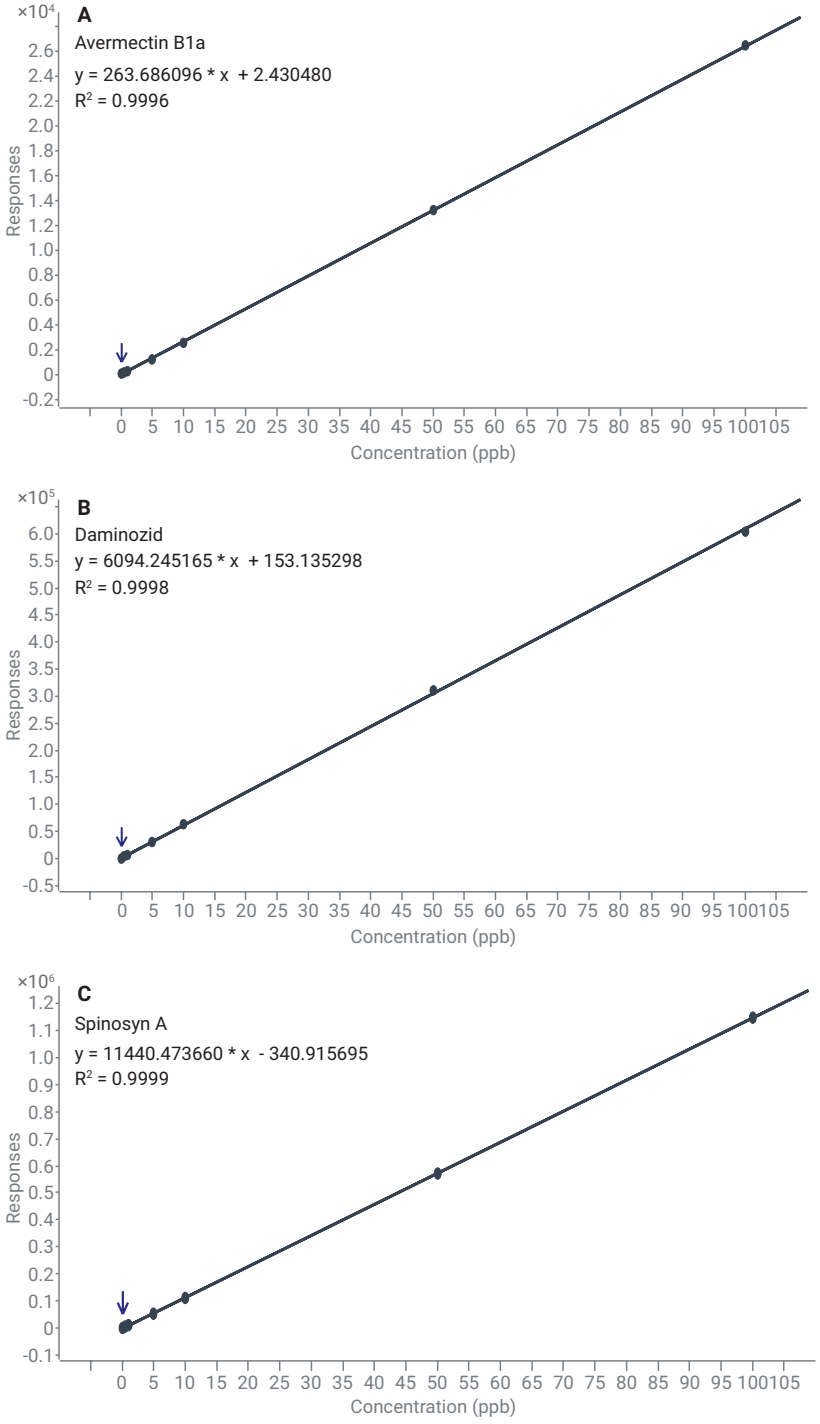


Figure 3. Example calibration curves, California list.

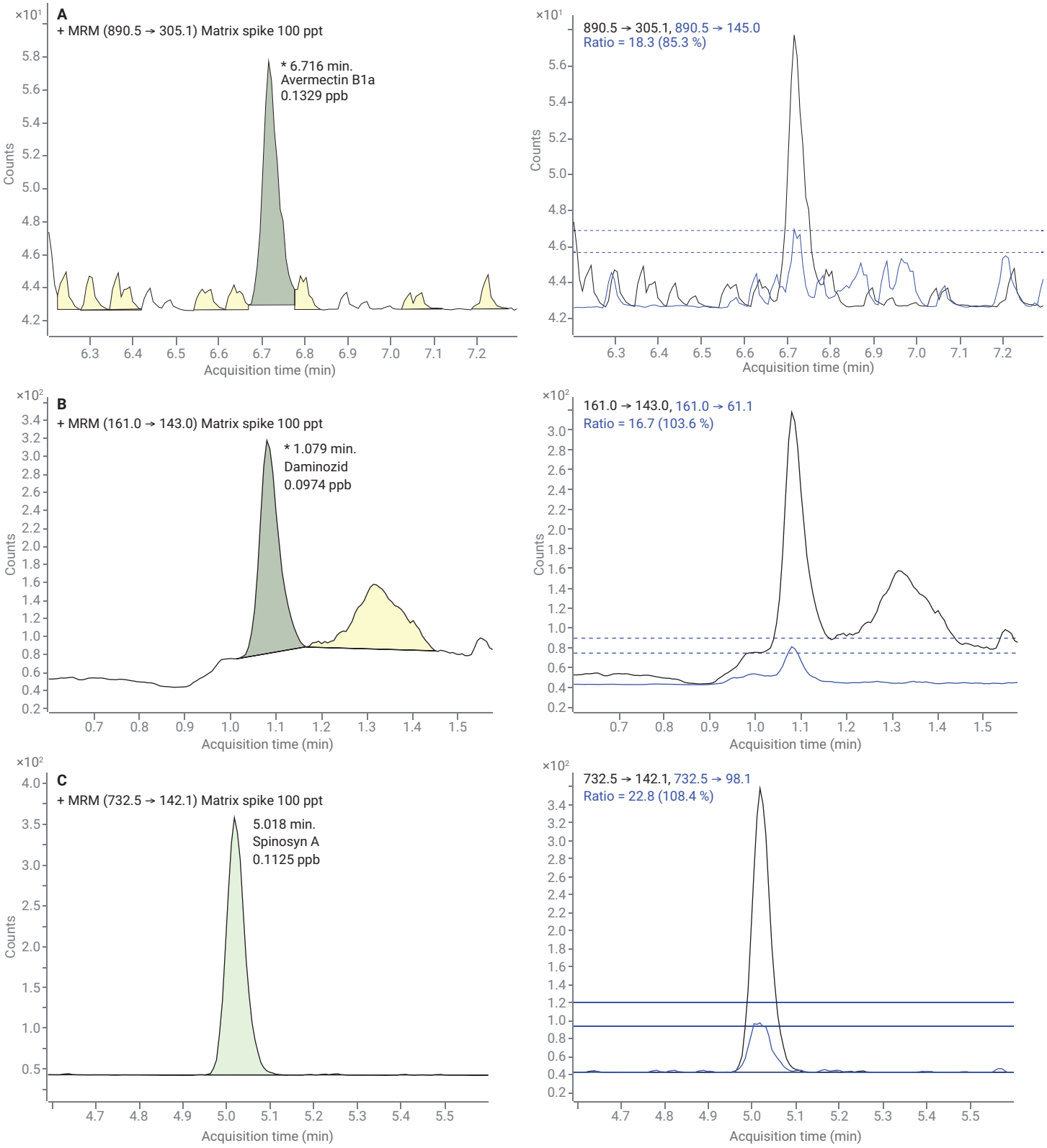


Figure 4. Examples of chromatography near LLOQ. A) Avermectin B1a, 0.1 ppb (ng/mL). B) Daminozide, 0.1 ppb (ng/mL). C) Spinosyn A, 0.1 ppb (ng/mL).

Table 3. Typical pesticide LLOQ results obtained as a mean from multiple (n = 5) batches of cannabis flower and prespiked into the sample extract before the SPE extraction and dilution routine described previously. Analytes typically responding more reliably through GC/MS are denoted in the LLOQ column as *GC/MS*.

California action list	CA action level (ppb)	LLOQ with 10 µL injection (ppb) original plant concentration
Avamectin B1a	100	50
Avamectin B1b	100	50
Acephate	100	25
Acequinocyl	100	2.5
Acetamiprid	100	2.5
Aldicarb	>LOD	5
Azoxystrobin	100	5
Bifenazate	100	50
Bifenthrin	3,000	5
Boscalid	100	50
Captan	700	GC/MS
Carbaryl	500	25
Carbofuran	>LOD	25
Chlorantraniliprole	10,000	25
Chlordane	>LOD	GC/MS
Chlorfenapyr	>LOD	100
Chlorpyrifos	>LOD	25
Clofentezine	100	2.5
Coumaphos	>LOD	5
Cyfluthrin	2,000	50
Cypermethrin	1,000	50
Daminozide	>LOD	25
Diazinon	100	2.5
Dichlorvos	>LOD	50
Dimethoate	>LOD	25
Dimethomorph 1	2,000	25
Dimethomorph 2	2,000	25
Ethoprop	>LOD	25
Etofenprox	>LOD	5
Etoxazole	100	25
Fenhexamid	100	50
Fenoxycarb	>LOD	5
Fenpyroximate	100	25
Fipronil	>LOD	5
Flonicamid	100	25
Fludioxonil	100	25

California action list	CA action level (ppb)	LLOQ with 10 µL injection (ppb) original plant concentration
Hexythiazox	100	5
Imazalil	>LOD	50
Imidacloprid	5,000	2.5
Kresoxim-methyl	100	5
Malathion	500	100
Metalaxyl	100	25
Methiocarb	>LOD	50
Methomyl	1,000	25
Methyl parathion	>LOD	GC/MS
Mevinphos	>LOD	50
MGK-264	NA	25
Myclobutanil	100	50
Dibrom Naled	100	50
Oxamyl	500	0.5
Paclobutrazol	>LOD	25
Pentachloronitrobenzene	100	GC/MS
Permethrin	500	50
Phosmet	100	5
Piperonyl butoxide	3,000	5
Prallethrin	100	25
Propiconazole	100	25
Propoxur	>LOD	25
Pyrethrin I	500	50
Pyrethrin II	500	50
Pyridaben	100	5
Spinetoram J	100	25
Spinetoram L	100	25
Spinosin A	100	5
Spinosin D	100	50
Spiromesifen	100	25
Spirotetramat	100	25
Spiroxamine	>LOD	25
Tebuconazole	100	25
Thiacloprid	>LOD	25
Thiamethoxam	5,000	25
Trifloxystrobin	100	2.5

Table 4. Typical mycotoxin LLOQ results obtained as a mean from multiple batches (n = 5) of cannabis flower and prespiked into the sample extract before the described SPE extraction and dilution routine.

CA action list (mycotoxins)	CA action level (ppb)	LLOQ with 10 µL injection original plant concentration (ppb)
Aflatoxin G1	Total amount of Aflatoxins not to exceed 20 ppb	3
Aflatoxin G2		3.5
Aflatoxin B1		3
Aflatoxin B2		3
Ochratoxin A	20	7

Sample preparation and autosampler pretreatment discussion

Recovery data were gathered for the sample preparation routine outlined in this Application Note, and displayed in Table 5. Prespiked negative cannabis flower and nonspiked negative flower were ground, solvent-extracted, and cleaned up using SPE as outlined in the sample preparation experimental section. The nonspiked extracts from this routine were then spiked at set levels, and the percentage recovery of the pre- and post spiked matrix-matched samples was calculated for every analyte with the following equation using single point calibrations:

$$\% \text{ Recovery} = \frac{\text{Pre} - \text{SPE spiked sample}}{\text{Post} - \text{SPE spiked sample}} \times 100$$

An important aspect of the sample preparation routine to note is the nature and composition of the diluent used in the final dilution step outlined in the experimental section stage 7 of Figure 1.

Many of the analytes in the California action list are highly nonpolar, and can precipitate out of solution when the aqueous content of the diluent is sufficiently high, yielding extremely poor recoveries for these analytes. For this reason, the composition of the final diluent was investigated from a recovery point of view. This investigation determined that the aqueous content

Table 5. Sample preparation percent recoveries observed for each California action pesticide from five separate batches (n = 5).

California pesticide list	Percent recovery at 60 ppb
Abamectin B1a	92.5
Abamectin B1b	113.4
Acephate	91.9
Acequinocyl	94.2
Acetamiprid	94.9
Aldicarb	93.7
Azoxystrobin	95.2
Bifenazate	98.8
Bifenthrin	98.0
Boscalid	104.2
Carbaryl	95.7
Carbofuran	94.9
Chlorantraniliprole	98.2
Chlorfenapyr	102.4
Chlorpyrifos	96.4
Clofentezine	100.8
Coumaphos	106.8
Cyfluthrin	97.7
Cypermethrin	96.3
Daminozide	88.4
DDVP (Dichlorvos)	97.3
Diazinon	97.1
Dimethomorph I	107.6
Dimethomorph II	108.2
Dimethoate	97.9
Ethoprop	103.0
Etofenprox	101.1
Etoxazole	98.6
Fenhexamid	129.5
Fenoxycarb	102.4
Fenpyroximate	103.2
Fipronil	90.6
Flonicamid	97.9
Fludioxonil	107.5
Hexythiazox	106.3
Imazalil	99.1
Imidacloprid	97.8
Kresoxim-methyl	103.7
Malathion	100.5
Metalaxyl	98.2
Methiocarb	102.7
Methomyl	96.5
Methyl parathion	110.4
Mevinphos	103.9
MGK-264	109.4
Myclobutanil	104.9
Oxamyl	97.0
Paclobutrazol	106.9
Permethrins*	96.8
Phosmet	101.9
Piperonyl butoxide	100.5
Prallethrin	98.1
Propiconazole	104.7
Propoxur	99.2
Pyrethrins†	70.8
Pyridaben	101.0
Spinetoram L	108.6
Spinetoram J	102.4
Spinosin A	101.2
Spinosin D	96.5
Spiromesifen	99.0
Spirotetramat	99.3
Spiroxamine	97.4
Tebuconazole	105.5
Thiacloprid	100.4
Thiamethoxam	97.2
Trifloxystrobin	100.8

Table 6. Average percent recoveries for each California mycotoxin (n = 5).

CA mycotoxin list	% Recovery at 4 ppb
Aflatoxin G1	102.8
Aflatoxin G2	102.7
Aflatoxin B1	104.8
Aflatoxin B2	102.3
Ochratoxin A	100.5

of that diluent could be no higher than 25 % v/v for the final dilution.

Injector pretreatment

For reversed-phase chromatography, such a high composition of organic solvent in the sample to be injected (in this case methanol at 75 % v/v) can and will result in splitting or smearing the early-eluting analyte peaks upon normal injection. To counter this effect and to keep peak shape and symmetry acceptable for all analytes in this method, an injector pretreatment routine is required, and is outlined in Table 2.

This pretreatment routine effectively dilutes the 75 % methanol in the sample when injected by sandwiching it between two equal 10 µL volumes of 0.1 % FA in water, mixing this together and effectively diluting it *in situ* to approximately 75/25 % aqueous/methanol. The chromatography gradient composition starts at 30 % methanol composition, and peak smearing/splitting is avoided, thus, acceptable peak shapes and symmetry is maintained across the complete chromatographic analysis.

Overlapped injections

To avoid the extra time needed for sample pretreatment in this manner, it is possible to preload samples at the re-equilibration period at the end of the chromatographic gradient by selecting the overlapped injection option. This option is available on all Agilent LC/MS/MS systems and configurations. For this methodology, it is recommended to invoke this function at or after 10.5 minutes to ensure that no retention time shift occurs in subsequent samples injected.

Review and reporting

Agilent LC/MS/MS and GC/MS/MS instruments employ the same MassHunter Quantitation software for data review and reporting. This optimizes lab productivity and operator's ease-of-use. To allow for review by exception, MassHunter Quantitation software enables quick and efficient batch processing using outlier settings per analyte. This approach automatically flags any sample or individual analyte, and draws the reviewer's attention to anything that may not be within designated limits. Figure 5 illustrates these outlier flags. The red color designates a value above accepted outlier limits, while blue denotes results below the required outlier limits.

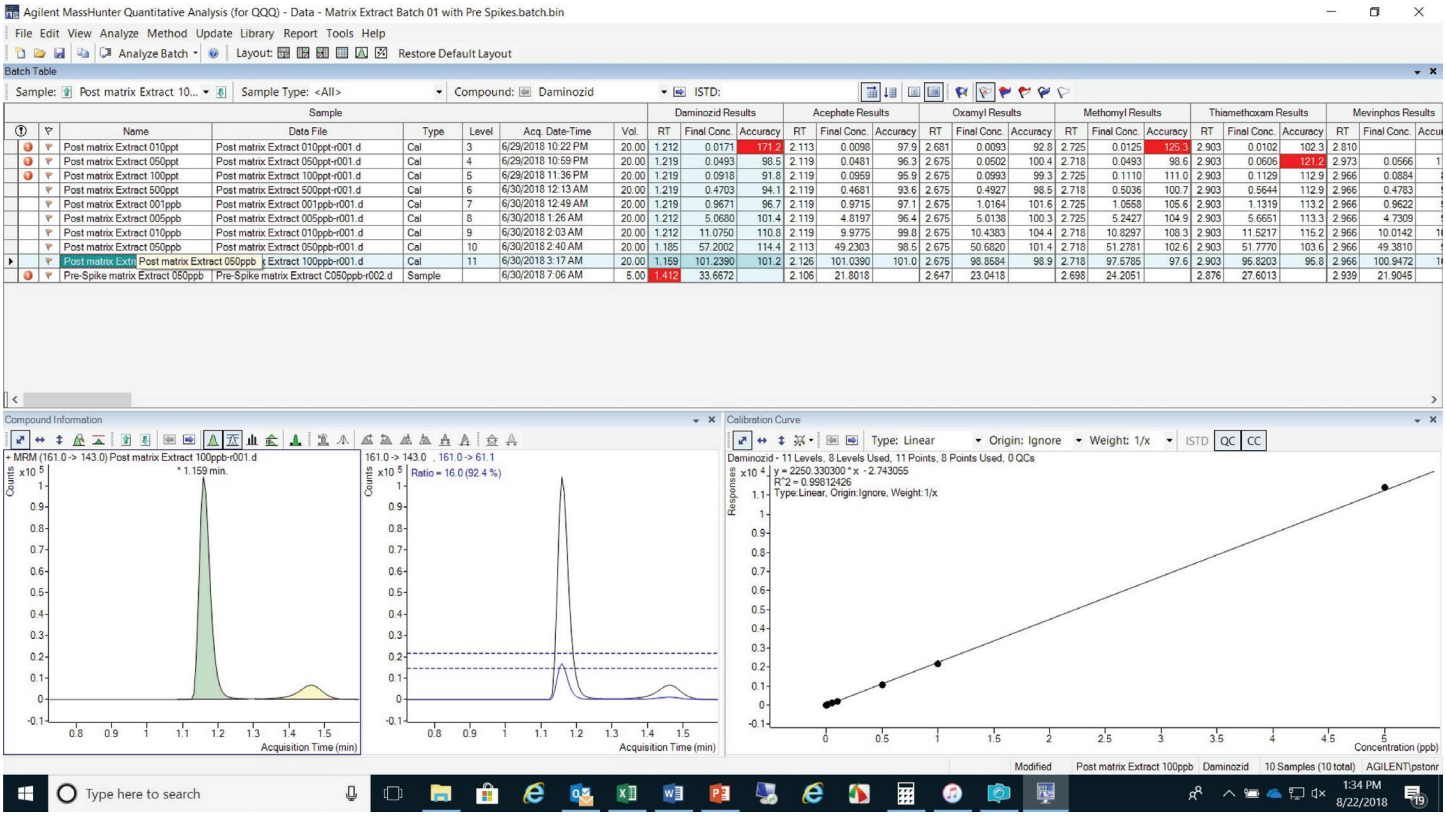
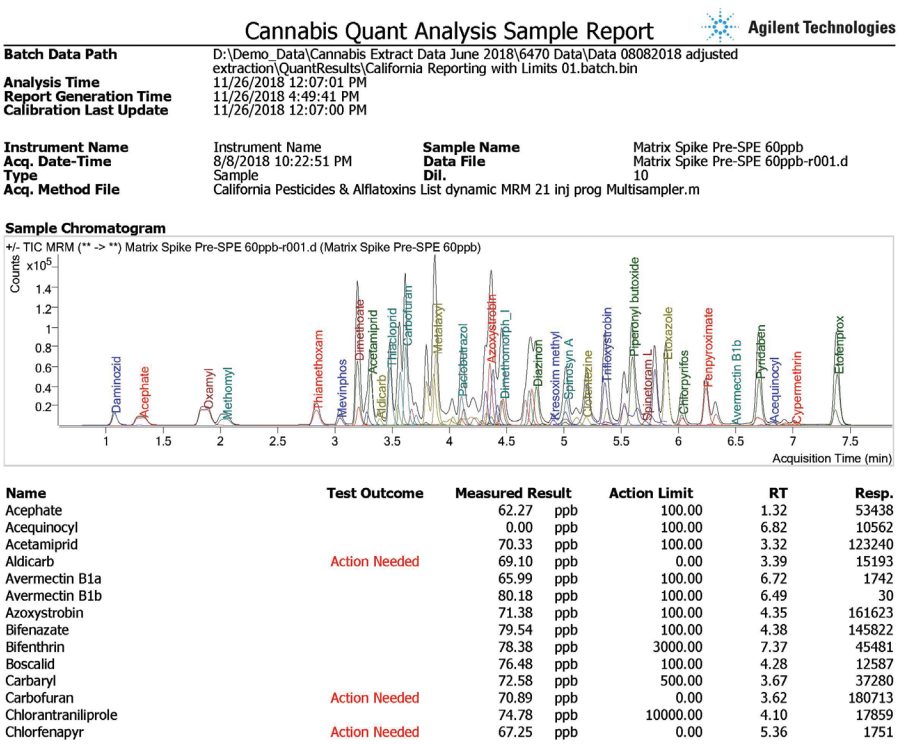


Figure 5. MassHunter Quantitative Analysis, review by exception batch review.

MassHunter offers the ability to tailor the analysis interface to the application with the Quant-My-Way functionality. Two preset configurations, or flavors, have been developed to meet the needs of cannabis method development, data processing and review, as well as reporting for LC/MS or GC/MS.

- First, the Scientist level has complete method setup, batch review, and reporting capabilities for each instrument technique (gas phase or liquid phase.)
- Second, the Analyst level has a simplified and uncluttered GUI, for use in the daily production environment. In this level, batch review and report generation are only allowed from predefined data review criteria, methods, and templates, which are set by the Scientist-designated personnel. Using these different GUI choices, a laboratory can more easily control how data are processed and reported in a more controlled environment.

Custom report templates that have been specifically designed for the cannabis analysis requirements of each geographic region are also available as an integral element of the MassHunter Quantitation software. Figure 6 shows an example of this.



Cannabis Quant Analysis Sample Report

Agilent Technologies

Batch Data Path	D:\Demo_Data\Cannabis Extract Data June 2018\6470 Data\Data 08082018 adjusted extraction\QuantResults\California Reporting with Limits 01.batch.bin
Analysis Time	11/26/2018 12:07:01 PM
Report Generation Time	11/26/2018 4:49:41 PM
Calibration Last Update	11/26/2018 12:07:00 PM

Instrument Name	Instrument Name	**Sample Name**	Matrix Spike Pre-SPE 60ppb
Acq. Date-Time	8/8/2018 10:22:51 PM	**Data File**	Matrix Spike Pre-SPE 60ppb-r001.d
Type	Sample	**Dil.**	10
Acq. Method File	California Pesticides & Alflatoxins List dynamic MRM 21 inj prog Multisampler.m		

Sample Chromatogram

Name	Test Outcome	Measured Result		Action Limit	RT	Resp.
Acephate		62.27	ppb	100.00	1.32	53438
Acequinocyl		0.00	ppb	100.00	6.82	10562
Acetamiprid		70.33	ppb	100.00	3.32	123240
Aldicarb	Action Needed	69.10	ppb	0.00	3.39	15193
Avermectin B1a		65.99	ppb	100.00	6.72	1742
Avermectin B1b		80.18	ppb	100.00	6.49	30
Azoxystrobin		71.38	ppb	100.00	4.35	161623
Bifenazate		79.54	ppb	100.00	4.38	145822
Bifenthrin		78.38	ppb	3000.00	7.37	45481
Boscalid		76.48	ppb	100.00	4.28	12587
Carbaryl		72.58	ppb	500.00	3.67	37280
Carbofuran	Action Needed	70.89	ppb	0.00	3.62	180713
Chlorantraniliprole		74.78	ppb	10000.00	4.10	17859
Chlorfenapyr	Action Needed	67.25	ppb	0.00	5.36	1751

Figure 6. Example of cannabis reporting templates.

Conclusions

This Application Note describes a robust LC/MS/MS method and sample preparation workflow that reliably meets at least 50 % the current California legislative safety action limits for pesticide and mycotoxin content for cannabis dried flower samples. It uses Agilent 6470 (G6470AA) and Agilent Ultivo (G6465BA) units, which yield similar results. This methodology complements other techniques, which are necessary for a handful of the action list items (captan, chlordane, PCNB, and methyl parathion). These analytes are more reliably analyzed using GC/MS/MS techniques such as that outlined in Agilent Application Note 5994-0568EN[3].

Sample preparation used a simple SPE filtration approach, recoveries from which were all between 70–130 %, as required by California legislation[1]. In addition, most recoveries were close to 100 % using the unique SampliQ C18 EC SPE cartridges and routines outlined in the experimental section. The unique ability to use an injector pretreatment routine for injection handling and manipulation of samples adds to the high percent recoveries while allowing for excellent chromatographic peak shapes across the entire analysis gradient.

A two-level graphical user interface approach has been created (if required), consisting of the Scientist and Analyst levels, for seamless data review and method creation using MassHunter Quantitation and batch processing software. This specifically allows a quality testing laboratory to assign access roles, and simplify workflows for data review and reporting within its workforce based on access level to methodology and ability levels.

Custom reporting templates are available as standard with MassHunter software, and are focused on regions or states, depending on local requirements.

References

1. Bureau of Marijuana Control Proposed Text of Regulations California Code of Regulations Title 16 Division 42. Bureau of Marijuana Control Chapter 5. Testing Laboratories.
2. Mordehai, A.; Fjeldsted, J. Agilent Jet Stream Thermal Gradient Focusing Technology. *Agilent Technologies Technical Overview*, publication number 5990 3494EN, **2009**.
3. Andrianova, A. A.; *et al.* Sensitive and Robust Detection of Pesticides in Dried Cannabis Plant Material Regulated in California, *Agilent Technologies Application Note*, publication number 5994-0568EN, **2019**.

Appendix A: Agilent 6470 (G6470AA) and Agilent Ultivo (G6465BA) transitions for pesticides and mycotoxins

Cell acceleration voltage (CAV) is irrelevant for Ultivo LC/MS instruments

Compound	Precursor Ion	Product Ion	Fragmentor (V)	CE (V)	Cell Acc (V)	Polarity
Acephate	184	143	60	5	4	Positive
Acephate	184	95	60	20	4	Positive
Acequinocyl	402.3	343.2	90	10	4	Positive
Acequinocyl	402.3	189.1	90	41	4	Positive
Acetamiprid	223	126.1	100	20	3	Positive
Acetamiprid	223	90.1	100	35	3	Positive
AflatoxinB1	313.1	285.1	130	20	3	Positive
AflatoxinB1	313.1	241.1	130	35	3	Positive
AflatoxinB2	315.1	287.1	130	25	3	Positive
AflatoxinB2	315.1	259.1	130	25	3	Positive
AflatoxinG1	329.1	311.1	130	20	3	Positive
AflatoxinG1	329.1	243.1	130	25	3	Positive
AflatoxinG2	331.1	285.1	130	25	3	Positive
AflatoxinG2	331.1	245.1	130	30	3	Positive
Aldicarb	116	89.1	50	4	3	Positive
Aldicarb	116	70.1	50	4	3	Positive
Avermectin B1a	890.5	567.1	160	8	4	Positive
Avermectin B1a	890.5	305.1	160	28	4	Positive
Avermectin B1a	890.5	145	160	45	4	Positive
Avermectin B1b	876.6	553.2	160	7	4	Positive
Avermectin B1b	876.6	291.1	160	15	4	Positive
Azoxystrobin	404	372.2	100	10	3	Positive
Azoxystrobin	404	344	100	25	3	Positive
Bifenazate	301.1	198.2	80	5	3	Positive
Bifenazate	301.1	170.1	80	15	3	Positive
Bifenthrin	440.1	181.1012	90	5	5	Positive
Bifenthrin	440.1	166	90	20	5	Positive
Boscalid	343	307.0633	140	12	5	Positive
Boscalid	343	271	140	28	5	Positive
Carbaryl	202	145	70	0	3	Positive
Carbaryl	202	127.1	70	25	3	Positive
Carbofuran	222.1	165.1	90	5	3	Positive
Carbofuran	222.1	123.1	90	20	3	Positive
Chlorantraniliprole	483.9	452.9	100	15	3	Positive
Chlorantraniliprole	483.9	285.9	100	10	3	Positive
Chlorfenapyr	409.2	59	130	20	3	Positive
Chlorfenapyr	409.2	31	130	45	3	Positive
Chlorpyrifos	349.9	197.9275	100	20	5	Positive

Compound	Precursor Ion	Product Ion	Fragmentor (V)	CE (V)	Cell Acc (V)	Polarity
Chlorpyrifos	349.9	96.9508	100	41	5	Positive
Clofentezine	303	138	90	10	3	Positive
Clofentezine	303	102.1	90	40	3	Positive
Coumaphos	363	307	125	15	4	Positive
Coumaphos	363	226.9	125	33	4	Positive
Cyfluthrin	453.3	193	90	13	2	Positive
Cyfluthrin	451.3	191	90	13	2	Positive
Cypermethrin	435.3	193	90	16	2	Positive
Cypermethrin	433.3	416.3	90	7	2	Positive
Cypermethrin	433.3	191	90	16	2	Positive
Daminozide	161	143	80	10	2	Positive
Daminozide	161	61.1	80	10	2	Positive
Diazinon	305.1	169.0794	100	20	5	Positive
Diazinon	305.1	153.1022	100	20	5	Positive
Dichlorvos	221	109	110	12	3	Positive
Dichlorvos	221	79	110	24	3	Positive
Dimethoate	230	199	70	5	3	Positive
Dimethoate	230	125	70	21	3	Positive
Dimethomorph_I	388.1	301.1	145	20	3	Positive
Dimethomorph_I	388.1	165	145	32	3	Positive
Dimethomorph_II	388.1	301.1	145	20	3	Positive
Dimethomorph_II	388.1	165	145	32	3	Positive
Ethoprophos	243	131	90	15	3	Positive
Ethoprophos	243	97	90	30	3	Positive
Etofenprox	394.2	177.2	90	10	3	Positive
Etofenprox	394.2	107.1	90	45	3	Positive
Etoxazole	360.1	141.0146	140	28	5	Positive
Etoxazole	360.1	113.0197	140	50	5	Positive
Fenhexamid	302.1	97.2	145	25	4	Positive
Fenhexamid	302.1	55.1	145	45	4	Positive
Fenoxycarb	302.1	116.1	100	5	3	Positive
Fenoxycarb	302.1	88.1	100	15	3	Positive
Fenpyroximate	422.1	366.2	130	15	3	Positive
Fenpyroximate	422.1	135.1	130	30	3	Positive
Fipronil	436.9	332	100	18	2	Negative
Fipronil	434.9	330	100	18	2	Negative
Fipronil	434.9	250.1	100	30	2	Negative
Flonicamid	230	203	120	15	2	Positive
Flonicamid	230	174	120	20	3	Positive
Fludioxonil	229	185	120	15	2	Positive
Fludioxonil	229	158	120	20	2	Positive
Hexythiazox	353	228.1	90	10	3	Positive

Compound	Precursor Ion	Product Ion	Fragmentor (V)	CE (V)	Cell Acc (V)	Polarity
Hexythiazox	353	168.1	90	25	3	Positive
Imazalil	297	201	120	15	3	Positive
Imazalil	297	159	120	20	7	Positive
Imidacloprid	256	209.1	90	16	2	Positive
Imidacloprid	256	175.1	90	20	2	Positive
Kresoxim methyl	314.1	267.1	80	0	3	Positive
Kresoxim methyl	314.1	222.2	80	10	3	Positive
Malathion	331.1	126.9	80	5	5	Positive
Malathion	331.1	99	80	10	5	Positive
Metalaxyl	280.1	220.2	100	10	3	Positive
Metalaxyl	280.1	160.1	100	20	3	Positive
Methiocarb	226.1	169.1	70	0	7	Positive
Methiocarb	226.1	121.1	70	15	3	Positive
Methomyl	162.9	106.1	60	5	3	Positive
Methomyl	162.9	88.1	60	0	3	Positive
Methyl-Parathion	264	232	140	18	2	Positive
Methyl-Parathion	264	125	140	24	2	Positive
Mevinphos	225	192.9	60	5	4	Positive
Mevinphos	225	126.9	60	17	4	Positive
MGK-264	276.2	210.1	100	12	4	Positive
MGK-264	276.2	98	100	28	4	Positive
Myclobutanil	289.1	125	110	35	3	Positive
Myclobutanil	289.1	70.1	110	15	7	Positive
Ochratoxin	404.1	238.9	130	26	3	Positive
Ochratoxin	404.1	220.9	130	32	3	Positive
Oxamyl	237	90.1	60	0	3	Positive
Oxamyl	237	72.1	60	15	3	Positive
Paclobutrazol	294.1	125	110	40	3	Positive
Paclobutrazol	294.1	70.1	110	20	7	Positive
Permethrin	391.1	355	120	5	3	Positive
Permethrin	391.1	183	120	5	3	Positive
Phosmet	317.9	160	80	10	3	Positive
Phosmet	317.9	133	80	40	3	Positive
Piperonyl butoxide	356.2	177.1	90	5	3	Positive
Piperonyl butoxide	356.2	119.1	90	35	3	Positive
Prallethrin	301.1	169	90	5	3	Positive
Prallethrin	301.1	105	90	20	3	Positive
Propiconazole	342.1	159	130	32	2	Positive
Propiconazole	342.1	69.1	130	16	2	Positive
Propoxur	210	168	60	5	5	Positive
Propoxur	210	111	60	10	5	Positive
Pyrethrin I	329.2	161	90	5	3	Positive
Pyrethrin I	329.2	143	90	20	3	Positive

Compound	Precursor Ion	Product Ion	Fragmentor (V)	CE (V)	Cell Acc (V)	Polarity
Pyrethrin I	329.2	133	90	20	3	Positive
Pyrethrin_II	373.2	161	102	2	3	Positive
Pyrethrin_II	373.2	133.1	102	24	3	Positive
Pyrethrin_II	373.2	77	102	98	3	Positive
Pyridaben	365.1	309.1	90	4	2	Positive
Pyridaben	365.1	147.2	90	20	2	Positive
Pyridaben	365.1	117.1	90	60	2	Positive
Spinetoram J	748.5	142.1	165	26	3	Positive
Spinetoram J	748.5	98.1	165	50	3	Positive
Spinetoram L	760.5	142.1	165	26	3	Positive
Spinetoram L	760.5	98.1	165	50	3	Positive
Spinosyn A	732.5	142.1	160	28	2	Positive
Spinosyn A	732.5	98.1	160	60	2	Positive
Spinosyn D	746.5	142.1	160	35	2	Positive
Spinosyn D	746.5	98	160	55	2	Positive
Spiromesifen	388.2	273	80	6	2	Positive
Spiromesifen	388.2	255	80	26	2	Positive
Spirotetramat	374.2	330.2	110	12	5	Positive
Spirotetramat	374.2	302.2	110	12	5	Positive
Spirotetramat	374.2	216.1	110	36	5	Positive
Spiroxamine	298.2	144.1	120	16	4	Positive
Spiroxamine	298.2	100.1	120	32	4	Positive
Tebuconazole	308.1	124.9	120	47	2	Positive
Tebuconazole	308.1	70	120	40	2	Positive
Thiacloprid	253	126	100	16	2	Positive
Thiacloprid	253	90	100	40	2	Positive
Thiamethoxam	292	211.1	80	8	2	Positive
Thiamethoxam	292	181.1	80	20	2	Positive
Trifloxystrobin	409.1	186	100	12	2	Positive
Trifloxystrobin	409.1	145	100	52	2	Positive

For more details concerning this Application Note, please contact Peter JW Stone at Agilent Technologies Inc., 5301 Stevens Creek Blvd, Santa Clara, CA, 95051, USA.

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