THOMSON INSTRUMENT COMPANY Solutions ™ At Work

series	cap color	membrane	pore size	part #
Standard Filter Vial	●	PTFE	0.2µm	35530

Time saving sample prep for the analysis of 54 pesticide & aflatoxin residues in Cannabis by LC-MS/MS

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Introduction

Pesticide analysis of cannabis leaves and finished goods is becoming increasingly important as many states are legalizing it for medicinal and recreational purposes. Dosing methods include smoking/vaporizing and edibles but cannabis is still a Schedule 1 illegal drug and therefore have no FDA testing guidelines. Trace levels of pesticides can be incurred during cultivation or inhaled from dried pesticides on the cannabis. This study evaluates the sample preparation aspect for LC-MS/MS analysis of a 50+ analyte panel of pesticides, fungicides and aflatoxins. QuEChERS was used to extract the analytes from the cannabis flowers, followed by centrifugation and Thomson Standard Filter Vial for sample clean-up.

Equipment:

- Sciex 6500 QQQ Mass Spectrometer
- Shimadzu LC-30AD Pumps
- Run Time: 15 minutes
- Flow Rate: 0.5 mL/min
- Injection Volume: 12 µL
- Column: Kinetex C18, 5µm, 3mm x 150mm
- Mobile Phase A: 0.1% FA in Water
- Mobile Phase B: 5mM Ammonium Formate, 0.1% Formic Acid in MeOH
- Centrifuge
- Thomson Standard|FV® 0.2µm PTFE (p/n 35530)*
- Thomson 48 position Vial Filter Press (p/n 35015-476)

*For some autosamplers it is important to adjust the needle depth of your autosampler when using Thomson filter vials to improve the reproducibility of injections

Sample Preparation of Cannabis Flowers

1. Weigh out 0.25g of the flower into a 50mL conical.
2. Add 7g of QuEChERS
3. Add 15mL of 1% Acetic Acid in Acetonitrile
4. Vortex for 30 minutes
5. Centrifuge for 5 minutes
6. Transfer 400µL into the outer shell of p/n 35530
7. Add 4µL of ISTD
8. Partially depress the plunger and vortex
9. Ready to analyze

Results

20+ compounds were extracted from cannabis flower with excellent recoveries utilizing a modified QueChERS method. The linear range for all the aflatoxins and ochratoxins are 0.5-50ng/mL; while the other analytes are 1.0-100ng/mL. Excellent linearity (see Table 2) and good recovery was achieved for all the compounds.

Table 1. Shows the LOQ, linear range, % CV, r2 and accuracy for each analyte

Analyte	LOQ (ng/mL)	Linear Range (ng/mL)	% CV	r2 Value	% Accuracy
Abamectin Group 1	1	1- 100	< 14.6	0.9932	93.4 - 105.5
Abamectin Group 2	1	1- 100	<25.4	0.98806	93.6 - 103.4
AFLATOXIN B2 1	0.5	0.5 - 50	<3.3	0.99837	93.7 - 105.7
AFLATOXIN B2 2	0.5	0.5 - 50	<4.9	0.99833	94.0 - 104.6
AFLATOXIN G2 1	0.5	0.5 - 50	<5.0	0.99829	93.1 - 105.2
AFLATOXIN G2 2	0.5	0.5 - 50	<5.4	0.9983	93.7 - 104.9
AFLATOXIN B1 1	0.5	0.5 - 50	<3.9	0.99805	92.2 - 105.9
AFLATOXIN B1 2	0.5	0.5 - 50	<4.0	0.99789	92.0 - 106.4
AFLATOXIN G1 1	0.5	0.5 - 50	<4.2	0.99853	94.1 - 104.6
AFLATOXIN G1 2	0.5	0.5 - 50	<4.5	0.99827	93.8 - 105.1
Bifenthrin 1	1	1- 100	<7.9	0.99699	92.6 - 105.6
Bifenthrin 2	1	1- 100	<6.2	0.99704	92.8 - 105.3
Chlormequat 1	1	1- 100	<1.4	0.99593	87.3 - 111.0
Chlormequat 2	1	1- 100	<4.5	0.99512	86.6 - 111.3
Daminozide 1	1	1- 100	<1.9	0.96303	66.0 - 131.6
Daminozide 2	1	1- 100	<4.5	0.99512	65.5 - 131.7
Dichlorvos 1	1	1- 100	<7.2	0.99369	86.0 - 112.4
Dichlorvos 2	1	1- 100	<7.2	0.99371	86.1 - 112.8
Imidacloprid 1	1	1- 100	<4.9	0.99904	97.4 - 101.3
Imidacloprid 2	1	1- 100	<5.5	0.99887	97.5 - 101.6
Malathion A 1	1	1- 100	<4.3	0.99574	86.9 - 108.7
Malathion A 2	1	1- 100	<3.7	0.99416	84.5 - 111.4
Myclobutanil 1	1	1- 100	<3.5	0.99808	91.6 - 105.2
Myclobutanil 2	1	1- 100	<4.8	0.99773	91.0 - 106.2
OCHRATOXIN A 1	0.5	0.5 - 50	<8.6	0.97237	67.4 - 120.0
OCHRATOXIN A 2	0.5	0.5 - 50	<18.5	0.96764	67.2 - 121.2
Paclobutrazol 1	1	1- 100	<5.7	0.99481	86.6 - 109.5
Paclobutrazol 2	1	1- 100	<3.8	0.99469	85.6 - 109.6
Permethrin, cis- 1	1	1- 100	<6.6	0.99813	95.5 - 103.2
Permethrin, cis- 2	1	1- 100	<6.5	0.99782	93.6 - 102.8
Permethrin, trans- 1	1	1- 100	<8.1	0.99723	92.9 - 102.9
Permethrin, trans- 2	1	1- 100	<7.3	0.99694	91.8 - 105.2
Piperonyl butoxide 1	1	1- 100	<8.4	0.99523	93.2 - 106.3
Piperonyl butoxide 2	1	1- 100	<8.9	0.99526	93.1 - 106.3
Propiconazole 1	1	1- 100	<3.8	0.99759	90.1 - 105.4
Propiconazole 2	1	1- 100	<2.8	0.99722	89.6 - 106.7
Pyrethrins Cinerin I 1	1	1- 100	<13.0	0.99779	98.6 - 101.9
Pyrethrins Cinerin I 2	1	1- 100	<20.5	0.99494	96.4 - 103.3
Pyrethrins Cinerin II 1	1	1- 100	<8.3	0.99651	90.3 - 105.5
Pyrethrins Cinerin II 2	1	1- 100	<12.7	0.99351	88.2 - 110.2
Pyrethrins Jasmolin I 1	1	1- 100	<12.9	0.99702	94.6 - 103.7
Pyrethrins Jasmolin I 2	1	1- 100	<21.5	0.99449	96.2 - 103.5
Pyrethrins Jasmolin II 1	1	1- 100	<22.7	0.99355	93.8 - 103.3
Pyrethrins Jasmolin II 2	1	1- 100	<10.0	0.99751	94.5 - 103.7
Pyrethrins Pyrethrin I 1	1	1- 100	<17.6	0.99626	97.4 - 101.7
Pyrethrins Pyrethrin I 2	1	1- 100	<5.0	0.99906	96.4 - 102.4

Analyte	LOQ (ng/mL)	Linear Range (ng/mL)	% CV	r2 Value	% Accuracy
Pyrethrins Pyrethrin II 1	1	1- 100	<3.2	0.99853	92.9 - 104.2
Pyrethrins Pyrethrin II 2	1	1- 100	<38.3	0.98319	91.9 - 106.9
Spinosyn A 1	1	1- 100	<4.0	0.99913	95.2 - 102
Spinosyn A 2	1	1- 100	<3.2	0.99931	96.1 - 103.0
Spinosyn D 1	1	1- 100	<3.9	0.99897	94.9 - 103.2
Spinosyn D 2	1	1- 100	<5.4	0.9987	94.8 - 103.4
Spiromesifen 1	1	1- 100	<16.6	0.99223	95.8 - 105.0
Spiromesifen 2	1	1- 100	<13.8	0.99457	95.4 - 104.1
Uniconazole 1	1	1- 100	<4.7	0.99774	91.1 - 104.8
Uniconazole 2	1	1- 100	<8.0	0.99667	89.5 - 105.5

Table 2. % recovery of a subset of the analytes in Table 1

Analyte	RE MIX (PPB)	Spike Conc. (ppb)	% Recover
Abamectin	8.12	10	81.2%
AFLATOXIN B2	4.84	5	96.9%
AFLATOXIN G2	4.96	5	99.1%
AFLATOXIN B1	4.89	5	97.9%
AFLATOXIN G1	4.92	5	98.4%
Bifenthrin	8.36	10	83.6%
Chlormequat	9.38	10	93.8%
Daminozide	8.74	10	87.4%
Dichlorvos	9.43	10	94.3%
Imidacloprid	8.78	10	87.8%
Malathion A	10.00	10	100.0%
Myclobutanil	9.62	10	96.2%
Naled	8.23	10	82.3%
OCHRATOXIN A	4.58	5	91.6%
Paclobutrazol	9.59	10	95.9%
Permethrin, cis-	8.80	10	88.0%
Permethrin, trans-	8.80	10	88.0%
Piperonyl butoxide	10.02	10	100.2%
Propiconazole	9.94	10	99.4%
Pyrethrins Cinerin I	9.64	10	96.4%
Pyrethrins Cinerin II	9.43	10	94.3%
Pyrethrins Jasmolin I	8.99	10	89.9%
Pyrethrins Jasmolin II	9.98	10	99.8%
Pyrethrins Pyrethrin I	9.09	10	90.9%
Pyrethrins Pyrethrin II	9.51	10	95.1%
Spinosyn A	8.27	10	82.7%
Spinosyn D	8.39	10	83.9%
Spiromesifen	9.97	10	99.7%
Uniconazole	9.37	10	93.7%

Conclusion

Using a modified QuEChERS approach on difficult matrices allows for many compounds to be included in multiresidue pesticide screens that would have otherwise been excluded due to matrix suppression or false negative results. This modified QuEChERS – Filter Vial method saves time, reduces solvent waste and cost over the traditional approach, QuEChERS – SPE. This validated method for the compounds in Table 2 has good linearity and recovery without having to use more expensive time consuming clean-up techniques. This approach is an extremely cost effective way to ensure problem analytes on difficult matrices can be included in a screen. The Thomson Standard Filter vials save time and money when replacing SPE and traditional syringe filtration techniques.