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LCMS-8060 and GCMS-TQ8050

Analysis of Pesticides and Mycotoxins in Cannabis

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Pesticides in Cannabis

Summary: A high sensitivity method for detection of pesticides and mycotoxins in cannabis was developed.

Background: Medicinal and recreational cannabis use has increased dramatically in several states. State rules require testing of cannabis entering their markets, including testing for pesticides and mycotoxins. Stringent requirements for pesticide testing have been adopted which require high sensitivity LCMS and GCMS analysis. Robustness and speed are also required for accurate and economical cannabis analysis.

Method: Authentic standards mixes were obtained from ChemService Inc., and kept at −20 °C and protected from light when not in use. Dried cannabis flower was provided by licensed cannabis testing laboratories, where all work was conducted in compliance with state law.

Homogenized dried cannabis flower (0.5 g) was extracted with 5 mL acetonitrile for LCMS and 10mL acetonitrile for GCMS, with vortexing and sonication. After centrifugation to remove solid material, the supernatant filtered for LCMS analysis, or for GCMS, a 3 mL aliquot was treated with SupelQue Verde dSPE (Supelco). LCMS detection was performed using an LCMS-8060, a high speed, high sensitivity triple quadrupole mass spectrometer in MRM mode. MRM transitions and retention times for each analyte were established using authentic standards. GCMS analysis was carried out in a similar way, using a TQ-8050 triple quadrupole mass spectrometer in MRM mode. Analysis conditions for each method are shown in the tables below.

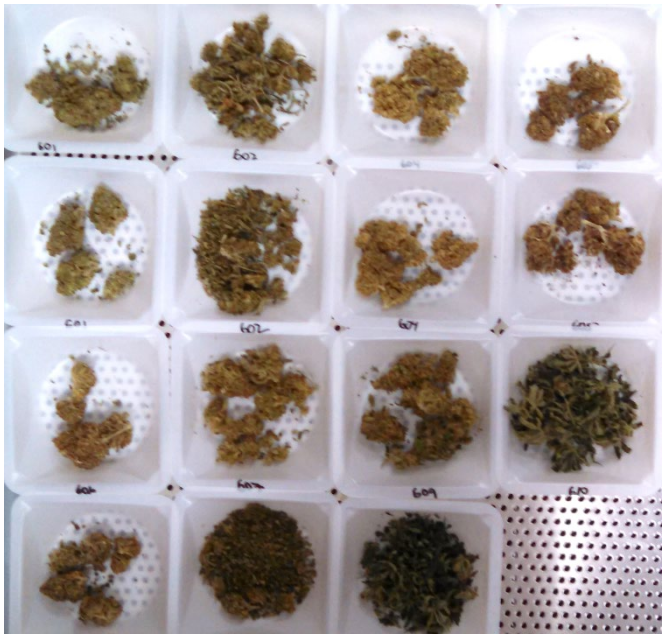

Figure 1 Typical dried flower samples

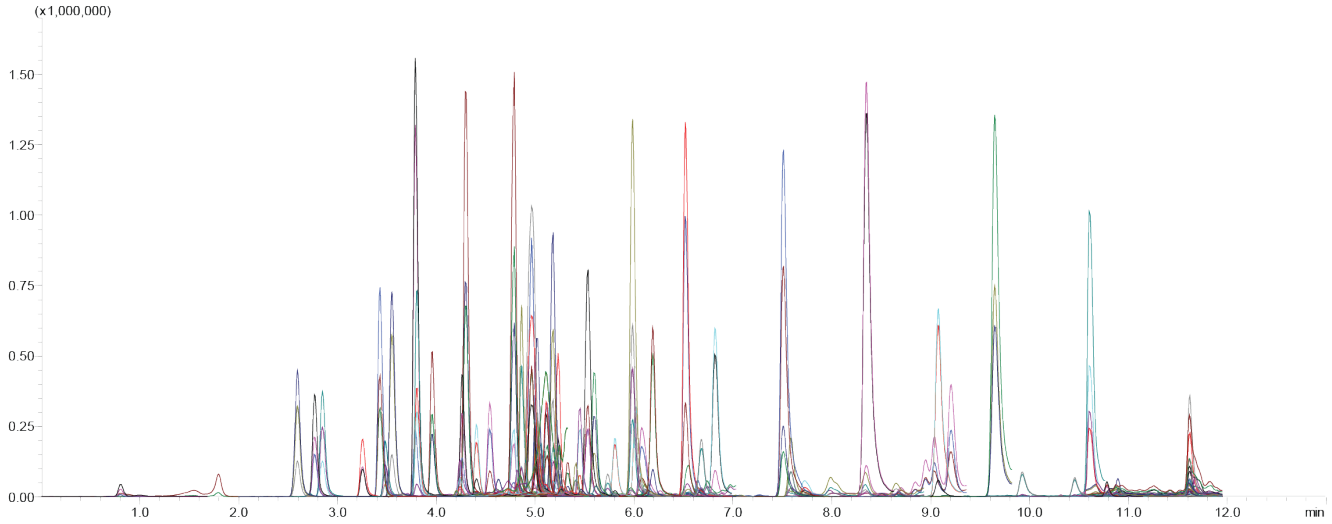

Figure 2 Representative LCMS chromatogram of pesticides and mycotoxins spiked in matrix at the 150 ng/g (dried flower basis) level

Column	Restek Raptor ARC-18 (2 x 150 mm)
Pump A	5 mM Ammonium formate with 0.1% formic acid in water
Pump B	Methanol
Time Program	10% B (0 min); 75% B (4 min); 87% B (9.5 min); 100% B (10 min); 100% B (12 min); 10% B (12.01-15 min)
Flow Rate	0.5 mL/min
Injection Volume	1 µL
Oven Temperature	40 °C
Ionization Mode	ESI (Positive/Negative)
Probe Voltage	+0.5kV/−0.5kV
Nebulizing Gas	3 L/min
Drying Gas	15 L/min
Heating Gas	15 L/min
Interface Temperature	100 °C
DL Temperature	100 °C
Block Heater Temperature	100 °C

Table 1 LCMS conditions

Inlet	250 °C; single taper gooseneck splitless liner with glass wool; Splitless injection, sampling time 1 minute
Column	SH-Rxi-5Sil-MS 15 m x 0.25 mm, 0.25 µm with 5 m guard; Helium carrier gas; Constant linear velocity 40.6 cm/sec
Oven Program	100 °C, hold 0.5 min; 40 °C/min to 200 °C; 15 °C/min to 275 °C; 40 °C/min to 330 °C, hold 2 min
Ion Source Temperature	230 °C

Table 2 GCMS conditions

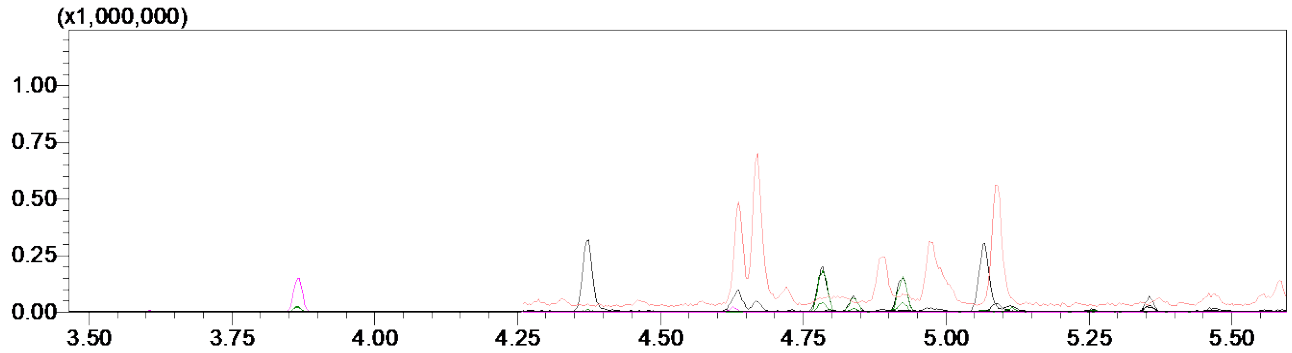

Figure 3 Representative GCMS chromatogram of pesticides spiked in matrix at the 200 ng/g (dried flower basis) level

	LOQ (ng/g flower)	Method		LOQ (ng/g flower)	Method		LOQ (ng/g flower)	Method
Abamectin	30	LCMS	Dinotefuran	2	LCMS	Oxamyl	2	LCMS
Acephate	20	LCMS	Dodemorph	4	LCMS	Paclobutrazol	2	LCMS
Acequinocyl	60	LCMS	Endosulfan-sulfate	4	LCMS	Pentachloronitrobenzene	<20	GCMS
Acetamiprid	<2	LCMS	Ethoprophos	2	LCMS	Permethrin	10	LCMS
Aldicarb	<2	LCMS	Etofenprox	4	LCMS	Phenothrin	10	LCMS
Allethrin	50	LCMS	Etoxazole	<2	LCMS	Phosmet	10	LCMS
Azoxystrobin	4	LCMS	Fenhexamid	20	LCMS	Piperonyl butoxide	5	LCMS
Bifenazate	2	LCMS	Fenoxycarb	2	LCMS	Pirimicarb	2	LCMS
Bifenthrin	4	LCMS	Fenpyroximate	10	LCMS	Prallethrin	10	LCMS
Boscalid	4	LCMS	Fensulfothion	5	LCMS	Propiconazole	60	LCMS
Buprofezin	<2	LCMS	Fenthion	100	LCMS	Propoxur	2	LCMS
Captan	<500	GCMS	Fenvalerate	100	LCMS	Pyraclostrobin	10	LCMS
Carbaryl	10	LCMS	Fipronil	2	LCMS	Pyrethrins	100	LCMS
Carbofuran	<2	LCMS	Flonicamid	25	LCMS	Pyridaben	2	LCMS
Chlorantraniliprole	2	LCMS	Fludioxonil	2	LCMS	Resmethrin	35	LCMS
Chlordane	20	GCMS	Fluopyram	2	LCMS	Spinetoram	2	LCMS
Chlorfenapyr	20	GCMS	Hexythiazox	15	LCMS	Spinosad	<2	LCMS
Chlorpyrifos	10	LCMS	Imazalil	10	LCMS	Spirodiclofen	10	LCMS
Clofentazine	4	LCMS	Imidacloprid	4	LCMS	Spiromesifen	20	LCMS
Clothianidin	4	LCMS	Kresoxim-methyl	4	LCMS	Spirotetramat	2	LCMS
Coumaphos	4	LCMS	Malathion	2	LCMS	Spiroxamine	2	LCMS
Cyantraniliprole	2	LCMS	Metalaxyl	2	LCMS	Tebuconazole	2	LCMS
Cyfluthrin	500	LCMS	Methiocarb	4	LCMS	Tebufenozide	5	LCMS
Cypermethrin	60	LCMS	Methomyl	<2	LCMS	Teflubenzuron	15	LCMS
Cyprodinil	10	LCMS	Methoprene	50	LCMS	Tetrachlorvinphos	4	LCMS
Daminozide	15	LCMS	Methyl parathion	20	GCMS	Tetramethrin	4	LCMS
Deltamethrin	30	LCMS	Mevinphos	4	LCMS	Thiacloprid	<2	LCMS
Diazinon	<2	LCMS	MGK-264	500	LCMS	Thiamethoxam	<2	LCMS
Dichlorvos	15	LCMS	Myclobutanil	10	LCMS	Thiophanate-methyl	5	LCMS
Dimethoate	<2	LCMS	Naled	2	LCMS	Trifloxystrobin	<2	LCMS
Dimethomorph	5	LCMS	Novaluron	15	LCMS			

Table 3 Limits of quantitation for each analyte

	LOQ (ng/g flower)	Method
Aflatoxin B1	2	LCMS
Aflatoxin B2	2	LCMS
Aflatoxin G1	2	LCMS
Aflatoxin G2	2	LCMS
Ochratoxin A	8	LCMS

Table 4 Limits of quantitation for each mycotoxin

Results and Discussion: Calibration curves were prepared by spiking authentic standards in pooled matrix blank extracts having previously tested negative for pesticides. The calibration curves ranged from 1 ng/g to over 2500 ng/g (dried flower basis) and were linear over the tested range. A 1/x weighting factor was used for calibration curves.

Peak identification was made using retention time and ion ratio matching, and the required S:N was 10:1 for the LOQ level.

The present method conditions represent a significant improvement in performance compared to previous methods. The newly optimized LC column with a longer length and adjusted gradient increase the separation of analytes from matrix interferences and also results in less signal suppression. The new, low-temperature interface conditions and spray voltage in the LCMS method also increased the signal intensity significantly for several challenging analytes. Finally, the ‘Verde’ dSPE improved the result for the GCMS method by removing most of the cannabinoids in the flower sample, which keeps the GCMS cleaner.

Conclusion: A rapid, robust, and high sensitivity method for analysis of pesticides and mycotoxins in cannabis has been developed.

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