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Trusted Answers

Sensitive and Robust Detection of Pesticides Regulated in California in Dried Cannabis Plant Material

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Abstract

At the present time, many U.S. state-regulated pesticide lists for cannabis can be analyzed exclusively by LC/MS/MS. Notable exceptions include California, Florida, and Nevada, where GC/MS/MS is also required. The number of states requiring GC/MS is expected to grow as more compounds and lower detection limits are required.

Quantifying pesticides at low levels in cannabis is possible with the Agilent standardized sample preparation procedure for cannabis combined with Agilent's 8890 GC System and 7010B Triple Quadrupole Mass Spectrometer. The Agilent MassHunter Pesticide and Environmental Pollutant MRM Database includes information on pesticides from the California target list, and retention time locking enables simple and rapid system setup. At the same time, longer column life and less frequent source cleaning can be achieved with midcolumn backflushing.

Using this approach, 43 GC-amenable pesticides regulated by the Bureau of Cannabis Control in California met the established limits of quantitation (LOQs) with LOQs for 36 pesticides at ≤0.08 ppb in-vial (≤10 ppb in dried cannabis plant material, assuming 100 % pesticide recovery), and all 43 pesticides at LOQs ≤0.8 ppb in-vial (≤100 ppb in cannabis plant material).

Introduction

By the end of 2018, the recreational use of cannabis became legal in nine states and Washington, DC in the U.S., and in all provinces in Canada. The global movement for cannabis legalization drives the demand for cannabis analytical testing methods for the detection of pesticide residues. Licensed retailers are required to have the cannabis goods analyzed for regulatory compliance testing. In the U.S., cannabis regulations vary from state to state, with California currently having the largest target list of pesticides tested in recreational cannabis[1]. The Canadian target list mandated by Health Canada generally exhibits lower required LOQs than any U.S. state[2]. The described methodology for pesticide residues in cannabis addresses the analytical challenge with a defined sample preparation procedure and state-of-the-art GC and LC triple quadrupole mass spectrometry[3].

The analysis of pesticides in cannabis is challenging due to high concentrations (percentage by weight) of endogenous chemicals such as cannabinoids and terpenes that interfere even with MS/MS processes. High-boiling matrix that elutes after the analytes requires extended bakeout times to prevent carryover and ghost peaks in subsequent runs. The highest boiling contaminants can deposit in the head of the column, requiring more frequent column trimming and adjustment of MRM and data analysis time windows due to retention time shifting. Following the Agilent recommended sample preparation procedure, the use of midcolumn backflush and retention time locking (RTL), quantifying pesticides at state-regulated limits is easily achieved.

This Application Note focuses on the triple quadrupole GC/MS analysis of GC-amenable pesticides regulated by the California Bureau of Cannabis Control, highlighting the pesticides that are commonly found to present a challenge for LC/MS analysis: for example, captan, chlordane, and pentachloronitrobenzene. The California LOQs were successfully met for 43 pesticides. The rest of the pesticides regulated in California are analyzed at the action level by LC/MS, as reported elsewhere[3].

Experimental

The Agilent system was configured to achieve the highest sensitivity, and minimize potential problems with pesticide analysis in high-matrix cannabis samples. Most importantly:

- **High-efficiency source (HES)** delivers confident trace analysis for ultratrace level applications by creating of up to 20x more ions.

- **Midcolumn backflushing** was used after the MS data were collected, the oven was held at the final temperature in post run mode, and the carrier gas flow through the first column was reversed. This reversed flow carried any high boilers that were in the column at the end of data collection out of the head of the column and into the split vent trap. The capability to reverse the flow is provided by the Agilent purged ultimate union (PUU). The PUU is a tee that is inserted, in this case, between two identical 15 m columns. During the analysis, a small makeup flow of carrier gas from Agilent's 8890 GC Pneumatic Switching Device (PSD) module is used to sweep the connection. During backflushing, the makeup flow from the PSD is raised to a much higher value, sweeping high boilers backwards out of the first column and forwards from the second. For this configuration, the backflushing time was 1.3 minutes.

- **Pulsed splitless injection** maximizes the transfer of analytes from the inlet onto the GC column. A pulsing pressure of 50 psi allowed for an injection volume of 3 μL of ACN, thus enabling lower limits of detection (LODs) in cannabis samples.

- **The PSD** is an Agilent 8890 GC pneumatics module optimized for backflushing applications. At high pressures during backflushing, the fixed restrictor can have hundreds of mL/min of wasted flow. The PSD will stay at the user-defined setpoint (default 3 mL/min) even at high pressures, which significantly reduces the required gas flow. Also, when the PSD is present in a midcolumn backflush configuration, the setup for pulsed splitless mode is simplified as the column flow for both column 1 and column 2 will be increased respectively during the pulse.

- **The Agilent MassHunter Pesticide and Environmental Pollutant MRM Database** includes up to eight MRM transitions per analyte, allowing users to simplify building acquisition methods for pesticide analysis. The database includes retention times for retention time-locked methods.

- **Dynamic MRM mode** creates a capability to tackle large multi-analyte assays and to accurately quantitate narrow peaks by an automated and most-efficient dwell time distribution.

- **Retention time locking** allows a new column or instrument to have retention times that match the MRM database or an existing method. This simplifies method maintenance and system setup.

- **The Agilent MassHunter Optimizer for GC/TQ** enables an automated optimization of MRM transitions, including precursor and product ion choice, and collision energy optimization. The Optimizer was used to optimize the MRM transitions for kinoprene, a pesticide from the Canadian target list.

Figure 1 shows the system configuration used for this application.

Tables 1 and 2 list the instrument operating parameters. MRM transitions for all pesticides, except for kinoprene and the deuterated internal standards, were provided by the MassHunter Pesticide and Environmental Pollutant MRM Database.
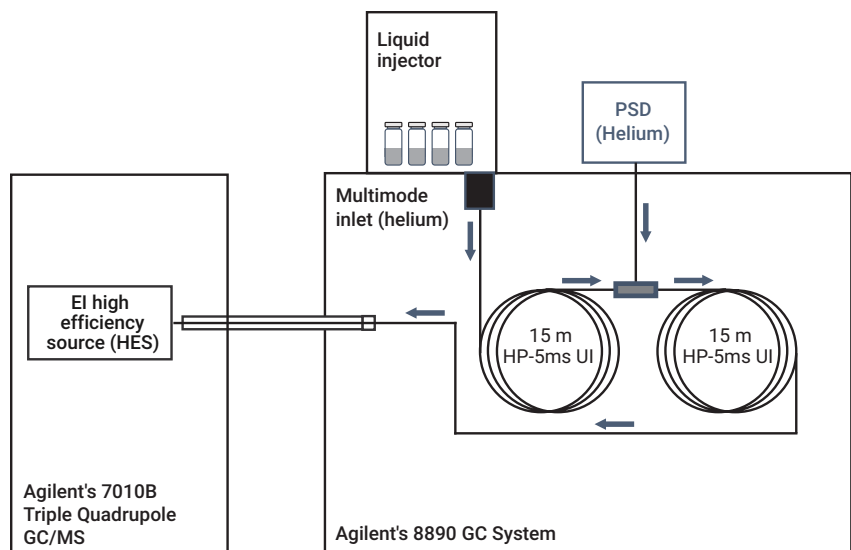

Figure 1. System configuration.

Table 1. GC and MS conditions for the pesticide analysis.

Agilent's 8890 GC System with fast oven, autoinjector, and tray	
Inlet	Multimode inlet (MMI)
Mode	Pulsed splitless
Injection Pulse Pressure	50 psi for 0.75 minutes
Purge Flow To Split Vent	50 mL/min at 0.7 minutes
Septum Purge Flow Mode	Switched
Injection Volume	3.0 µL
Injection Type	Reversed two-layer sandwich
L1 Airgap	0.2 µL
L2 Injection Volume	0.4 µL
L2 Airgap	0.2 µL
Inlet Temperature	280 °C
Carrier Gas	Helium
Inlet Liner	Agilent 4 mm single taper, with glass wool (p/n 5190-2293)
Oven	
Initial Oven Temperature	60 °C
Initial Oven Hold	1 minute
Ramp Rate 1	40 °C/min
Final Temperature 1	170 °C
Final Hold 1	0 minutes
Ramp Rate 2	10 °C/min
Final Temperature 2	310 °C
Final Hold 2	3 minutes
Total Run Time	20.75 minutes
Post Run Time	1.3 minutes
Equilibration Time	0.5 minutes

Column 1	HP-5MS UI, 15 m × 0.25 mm, 0.25 µm (p/n 19091S-431UI)
Control Mode	Constant flow
Flow	1.091 mL/min
Inlet Connection	MMI
Outlet Connection	PSD (PUU)
Post Run Flow (Backflushing)	−12.906 mL/min
Column 2	HP-5MS UI, 15 m × 0.25 mm, 0.25 µm (p/n 19091S-431UI)
Control Mode	Constant flow
Flow	1.291 mL/min
Inlet Connection	PSD (PUU)
Outlet Connection	MSD
Post Run Flow (Backflushing)	13.429 mL/min
MS	
Model	Agilent's 7010B Triple Quadrupole GC/MS
Source	High-efficiency source
Vacuum Pump	Performance Turbo
Tune File	Atunes.eihs.tune.xml
Mode	dMRM
Solvent Delay	3 minutes
EM Voltage Gain Mode	10
Quadrupole Temperature (MS1 and MS2)	150 °C
Source Temperature	280 °C
Transfer Lne Temperature	280 °C
He Quench Gas	2.25 mL/min
N_2 Collision Gas	1.5 mL/min

Table 2. MRM transitions used for the quantifier and qualifiers.

Name	RT (min)	Quantifier	Qualifier 1	Qualifier 2	Qualifier 3
Dichlorvos-d6	4.646	115.0 → 83.0	151.0 → 115.0	193.0 → 99.0	82.9 → 47.0
Dichlorvos	4.673	185.0 → 93.1	144.9 → 109.0	109.0 → 79.0	
Mevinphos	5.598	127.0 → 94.9	127.0 → 109.0	109.0 → 78.9	
Acephate	5.650	94.0 → 47.0	94.0 → 64.0	136.0 → 94.0	
Oxamyl	6.297	115.0 → 72.0	98.0 → 69.0	98.0 → 58.0	162.0 → 114.9
Propoxur	6.833	110.0 → 63.0	110.0 → 64.0	152.0 → 110.0	
Ethoprophos	7.019	157.9 → 97.0	157.9 → 114.0	138.9 → 97.0	
Naled	7.248	145.0 → 109.0	109.0 → 79.0	185.0 → 93.0	
Dimethoate	7.776	125.0 → 47.0	125.0 → 79.0	87.0 → 46.0	
Carbofuran	7.847	164.2 → 149.1	149.1 → 121.1	149.1 → 77.1	
Pentachloronitrobenzene	8.227	248.8 → 213.8	141.9 → 106.9	176.9 → 141.9	
Diazinon	8.285	137.1 → 84.0	137.1 → 54.0	199.1 → 93.0	
Spiroxamine	9.084	100.0 → 58.1	101.0 → 59.0	126.0 → 84.0	
Parathion-methyl	9.160	263.0 → 109.0	109.0 → 79.0	125.0 → 79.0	125.0 → 47.0
Carbaryl	9.237	144.1 → 116.1	144.1 → 89.0	115.1 → 89.0	
Metalaxyl	9.337	234.0 → 146.1	234.0 → 174.1	220.0 → 192.1	
Methiocarb	9.580	168.0 → 109.1	168.0 → 153.1	153.0 → 109.1	
Malathion	9.734	126.9 → 99.0	157.8 → 125.0	173.0 → 99.0	
Kinoprene	9.740	149.0 → 77.0	149.0 → 91.0	221.0 → 109.2	
Chlorpyrifos	9.959	313.8 → 257.8	198.9 → 171.0	196.9 → 169.0	
MGK-264	10.441	164.0 → 98.1	164.0 → 80.1	111.0 → 82.0	
Fipronil	10.648	366.8 → 212.8	254.9 → 228.0	350.8 → 254.8	367.0 → 213.0
Captan-d6	10.705	84.0 → 81.0	84.0 → 53.0	112.1 → 84.0	154.0 → 84.1
Captan	10.755	149.0 → 70.0	149.0 → 79.1	151.0 → 79.0	151.0 → 80.0
Chlordane-*trans*	11.045	375.0 → 266.0	271.7 → 236.9	372.8 → 265.8	
Paclobutrazol	11.099	236.0 → 125.0	236.0 → 167.1	125.1 → 89.0	
Chlordane-*cis*	11.318	375.0 → 266.0	271.7 → 236.9	372.8 → 265.8	
Fludioxonil	11.557	248.0 → 127.1	248.0 → 182.0	248.0 → 154.1	
Myclobutanil	11.711	179.0 → 125.1	179.0 → 90.0	150.0 → 123.0	
Kresoxim-methyl	11.829	116.0 → 89.0	116.0 → 63.0	131.0 → 89.0	
Chlorfenapyr	12.055	247.0 → 227.0	249.0 → 112.0	328.0 → 247.0	
Trifloxystrobin	12.920	116.0 → 89.0	116.0 → 63.0	131.0 → 89.0	172.0 → 95.0
Propiconazole I	13.108	172.9 → 109.0	172.9 → 145.0	172.9 → 74.0	
Propiconazole II	13.247	172.9 → 109.0	172.9 → 145.0	172.9 → 74.0	
Tebuconazole	13.279	250.0 → 125.0	125.0 → 99.0	125.0 → 89.0	
Spiromesifen	13.711	272.0 → 254.2	272.0 → 209.2	273.0 → 255.1	
Bifenthrin-d5	13.954	181.0 → 165.2	187.1 → 171.1	141.0 → 91.0	170.1 → 119.1
Bifenthrin	13.977	181.0 → 165.2	181.0 → 166.2	166.2 → 165.2	
Permethrin	15.719	163.0 → 127.0	182.9 → 155.1	183.1 → 153.1	183.1 → 168.1

Pyridaben	15.761	147.2 → 117.1	147.2 → 132.2	147.2 → 105.1	
Coumaphos	15.840	361.9 → 109.0	210.0 → 154.1	225.9 → 198.1	225.9 → 163.1
Cyfluthrin	16.244	163.0 → 127.0	162.9 → 90.9	206.0 → 150.0	
Boscalid	16.561	140.0 → 76.0	140.0 → 112.0	111.9 → 76.0	
Cypermethrin	16.563	163.0 → 127.0	163.0 → 91.0	181.0 → 152.1	
Ethofenprox	16.770	163.0 → 107.1	163.0 → 135.1	135.0 → 107.0	
Azoxystrobin-d4	18.426	348.2 → 333.1	348.2 → 183.1	348.2 → 172.0	348.2 → 156.0
Azoxystrobin	18.445	344.1 → 171.9	344.1 → 182.9	344.1 → 329.0	
Dimethomorph	18.433	300.9 → 165.0	302.9 → 164.9	386.8 → 300.9	

Matrix-matched calibration standards were prepared in extracts of dried cannabis flower grown without pesticides. Figure 2 shows the Agilent recommended sample preparation procedure.

Results and Discussion

Forty-three GC-amenable pesticides met LOQs established in California

The LOQs required in California were successfully met for 43 pesticides. Forty-two had LOQs ≤0.8 ppb in-vial (≤100 ppb in dried cannabis plant material), and 36 of the 42 pesticides had LOQs ≤0.08 ppb in-vial (≤10 ppb in dried cannabis plant material, where ppb in plant material corresponds to ng/g). Captan, *cis*- and *trans*-chlordane, and pentachloronitrobenzene (highlighted in Table 3) commonly present a challenge for LC/MS analysis, whereas they met the established LOQs with this method.

1. Weigh 1.0 g of chopped cannabis flower into a 50 mL tube. Add two ceramic homogenizer pellets, and shake for five minutes at 3,000 rpm.

2. Add 15 mL of acetonitrile, and shake for five minutes at 3,000 rpm.

3. Decant the supernatant solvent into an unconditioned SampliQ C18 EC cartridge (p/n 5982-1365). Keep the 50 mL tube with the pellets for Step 4. Gravity elute into a clean 50 mL tube.

4. Add 5 mL of acetonitrile, and shake for five minutes at 3,000 rpm.

5. Decant the supernatant solvent into the SampliQ C18 EC cartridge used in Step 3. Gravity elute. Keep the 50 mL tube with the pellets for Step 6.

6. Rinse the centrifuge tube with 5 mL of acetonitrile, and pass through supernatant into the same SPE cartridge used in Steps 3 and 5.

7. Bring the collected eluent (extract) up to 25 mL with acetonitrile (25-fold dilution).

8. Mix 100 µL of extract with 400 µL of acidified 50:50 hexane:acetone (v:v) (125-fold dilution*). Vortex for 10 seconds, and cap. **Samples are now ready for GC/MS/MS analysis.**

Figure 2. Schematic representation of sample preparation procedure for GC/MS/MS analysis. * Dilution solvent acidified with 0.1 % formic acid.

Table 3. Selected GC-amenable pesticides that meet LOQs established in California.

Compound	RT (min)	Reporting limit in California (ppb in dried cannabis plant material)	Reporting limit in Canada (ppb in dried cannabis plant material)	LOQ* (8890/7010) (ppb in dried cannabis plant material)	LOQ (ppb in vial)	High calibration limit (ppb in vial)	R^2
Acephate	5.650	100	20	100	0.8	10	0.9978
Azoxystrobin	18.445	100	20	10	0.08	5	0.9945
Bifenthrin	13.977	3,000	Under development (100)	100	0.8	50	0.9971
Boscalid	16.561	100	20	6.25	0.05	5	0.9960
Captan	10.755	700	N/A	100	0.8	25	0.9997
Carbaryl	9.237	500	50	10	0.08	25	0.9986
Carbofuran	7.847	100	20	10	0.08	5	0.9947
Chlordane-*cis*	11.318	100	N/A	6.25	0.05	5	0.9996
Chlordane-*trans*	11.045	100	N/A	6.25	0.05	5	0.9995
Chlorfenapyr	12.055	100	Under development (100)	6.25	0.05	5	0.9948
Chlorpyrifos	9.959	100	Under development (10)	6.25	0.05	5	0.9982
Coumaphos	15.840	100	20	6.25	0.05	5	0.9985
Cyfluthrin	16.244	2,000	Under development (3,750)	6.25	0.05	5	0.9976
Cypermethrin	16.563	1,000	Under development (3,750)	10	0.08	25	0.9981
Diazinon	8.285	100	Under development (10)	6.25	0.05	5	0.9985
Dichlorvos	4.673	100	100	6.25	0.05	5	0.9933
Dimethoate	7.776	100	20	6.25	0.05	5	0.9940
Dimethomorph	18.433	2,000	N/A	6.25	0.05	25	0.9992
Ethoprophos	7.019	100	20	6.25	0.05	5	0.9977
Etofenprox	16.770	100	Under development	10	0.08	5	0.9997
Fipronil	10.648	100	60	6.25	0.05	5	0.9907
Fludioxonil	11.557	100	20	6.25	0.05	5	0.9933
Kinoprene	9.740	N/A	Under development	100	0.8	25	0.9998
Kresoxim-methyl	11.829	100	Under development (10)	6.25	0.05	5	0.9980
Malathion	9.734	500	20	6.25	0.05	25	0.9998
Metalaxyl	9.337	2,000	20	6.25	0.05	25	0.9997
Methiocarb	9.580	100	20	6.25	0.05	5	0.9965
Methyl-parathion	9.160	100	Under development	6.25	0.05	5	0.9924
Mevinphos	5.598	100	50	6.25	0.05	5	0.9927
MGK-264	10.441	100	Under development	6.25	0.05	5	0.9982
Myclobutanil	11.711	100	20	100	0.8	10	0.9964
Naled	7.248	100	Under development	18.75	0.15	5	0.9935
Oxamyl	6.297	500	3,000	312.5	2.5	50	0.9995
Paclobutrazol	11.099	100	20	6.25	0.05	5	0.9932
Pentachloronitrobenzene	8.227	100	N/A	6.25	0.05	5	0.9994
Permethrin	15.719	500	Under development (500)	6.25	0.05	25	0.9993
Propiconazole I	13.108	100	Under development (10)	6.25	0.05	5	0.9920
Propiconazole II	13.247	100	Under development (10)	6.25	0.05	5	0.9937
Propoxur	6.833	100	20	6.25	0.05	5	0.9955
Pyridaben	15.761	100	50	6.25	0.05	5	0.9992
Spiromesifen	13.711	100	3000	6.25	0.05	5	0.9953
Spiroxamine	9.084	100	Under development	6.25	0.05	5	0.9987

| Tebuconazole | 13.279 | 100 | Under development (10) | 6.25 | 0.05 | 5 | 0.9963 |
| Trifloxystrobin | 12.920 | 100 | 20 | 100 | 0.8 | 10 | 0.9968 |

* LOQs in dried cannabis plant material were determined assuming a 100 % pesticide recovery. Internal standard (ISTD) matrix-matched calibration was performed for dichlorovos, captan, bifenthrin, and azoxystrobin. ISTDs were added by sandwich injection. External standard (ESTD) matrix-matched calibration was performed for the rest of the analyzed pesticides. The high end of the calibration range was chosen based on the required LOQ for the pesticide.

Successful GC/MS analysis of pesticides known to be challenging in LC/MS analysis

Figure 3 shows GC/MS MRM chromatograms at LOQ level for pentachloronitrobenzene, chlordane-*trans*, chlordane-*cis*, and captan. These pesticides are commonly challenging for LC/MS analysis. With the GC/MS method described here, these pesticides had LOQs lower than those established in California.

Reported LOQs were based on 10 replicate sequential injections at a given concentration level with %RSD <20. Instrument detection limits (IDLs) in fg on-column for each pesticide were determined by performing 10 replicate sequential injections of the matrix-matched calibration standard at the LOQ level, as shown in Figure 3. Calculations of IDL were made according to Formula 1. The value of t-test statistics for 10 measurements at a 99 % confidence interval (t_α) was 2.821. Table 3 presents the determined IDLs.

$IDL = (t_\alpha)(RSD)(amount\ of\ standard\ on\text{-}column)/100\ \%$

Formula 1.

A

Pentachloronitrobenzene
LOQ 0.05 ppb in vial (6.25 ppb in cannabis)
RSD 3.1 %
IDL in post spiked matrix 13 fg on-column

LOQ required in CA: 100 ppb

B

Captan
LOQ 0.8 ppb in vial (100 ppb in cannabis)
RSD 8.6 %
IDL in post spiked matrix 584 fg on-column
LOQ required in CA: 700 ppb

C

1. Chlordane-*trans*
LOQ 0.05 ppb in vial (6.25 ppb in cannabis)
RSD 12.0 %
IDL in post spiked matrix 51 fg on-column
LOQ required in CA: 100 ppb

2. Chlordane-*cis*
LOQ 0.05 ppb in vial (6.25 ppb in cannabis)
RSD 18.4 %
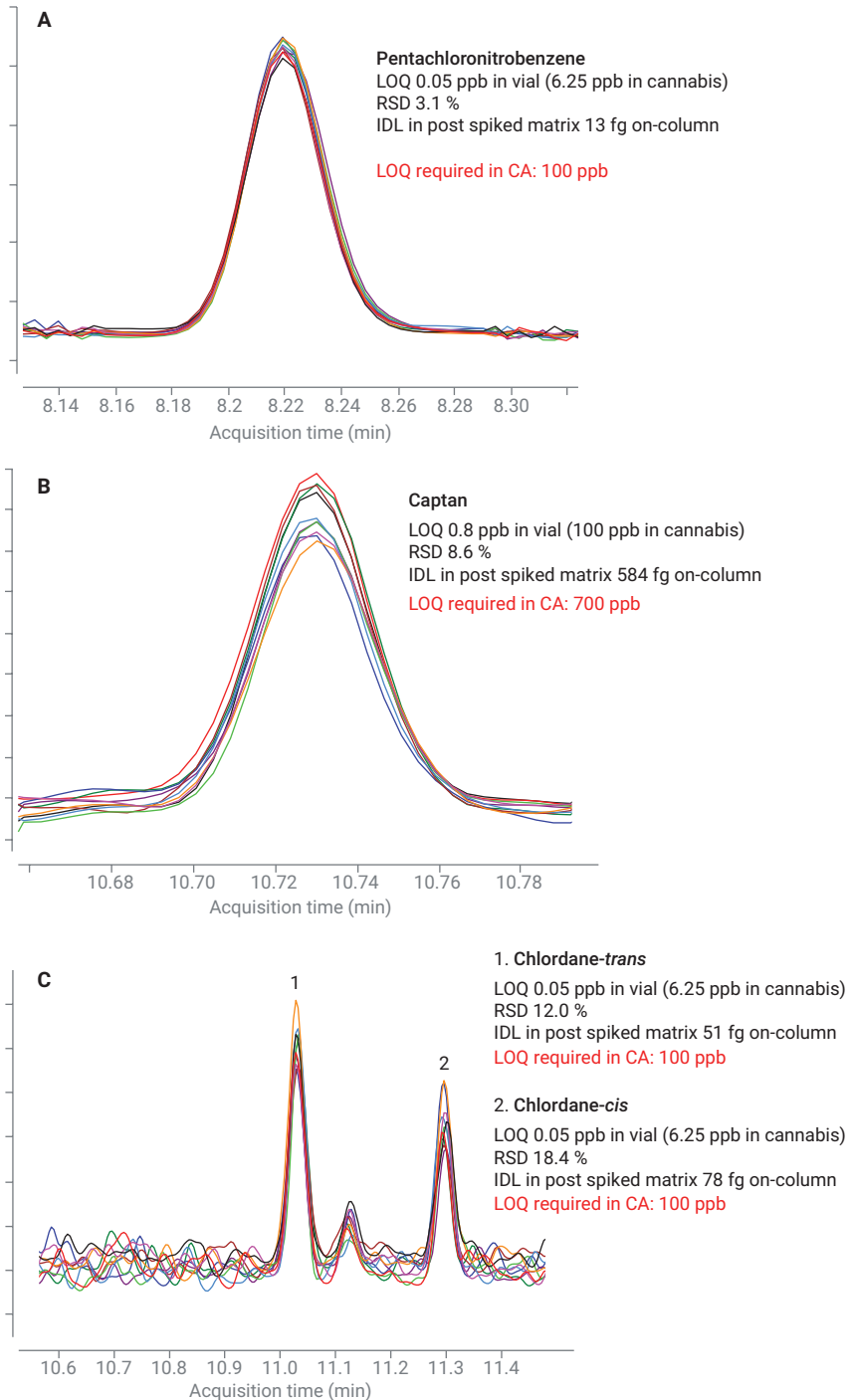IDL in post spiked matrix 78 fg on-column
LOQ required in CA: 100 ppb

Figure 3. MRM chromatograms of 10 sequential injections for for pentachloronitrobenzene (quintozene), captan, chlordane-*trans*, and chlordane-*cis* at LOQ level.

All 43 pesticides that met California required LOQs exhibited good linearity over the calibration range. Table 3 reports R^2 values, and Figure 4 shows select calibration curves.
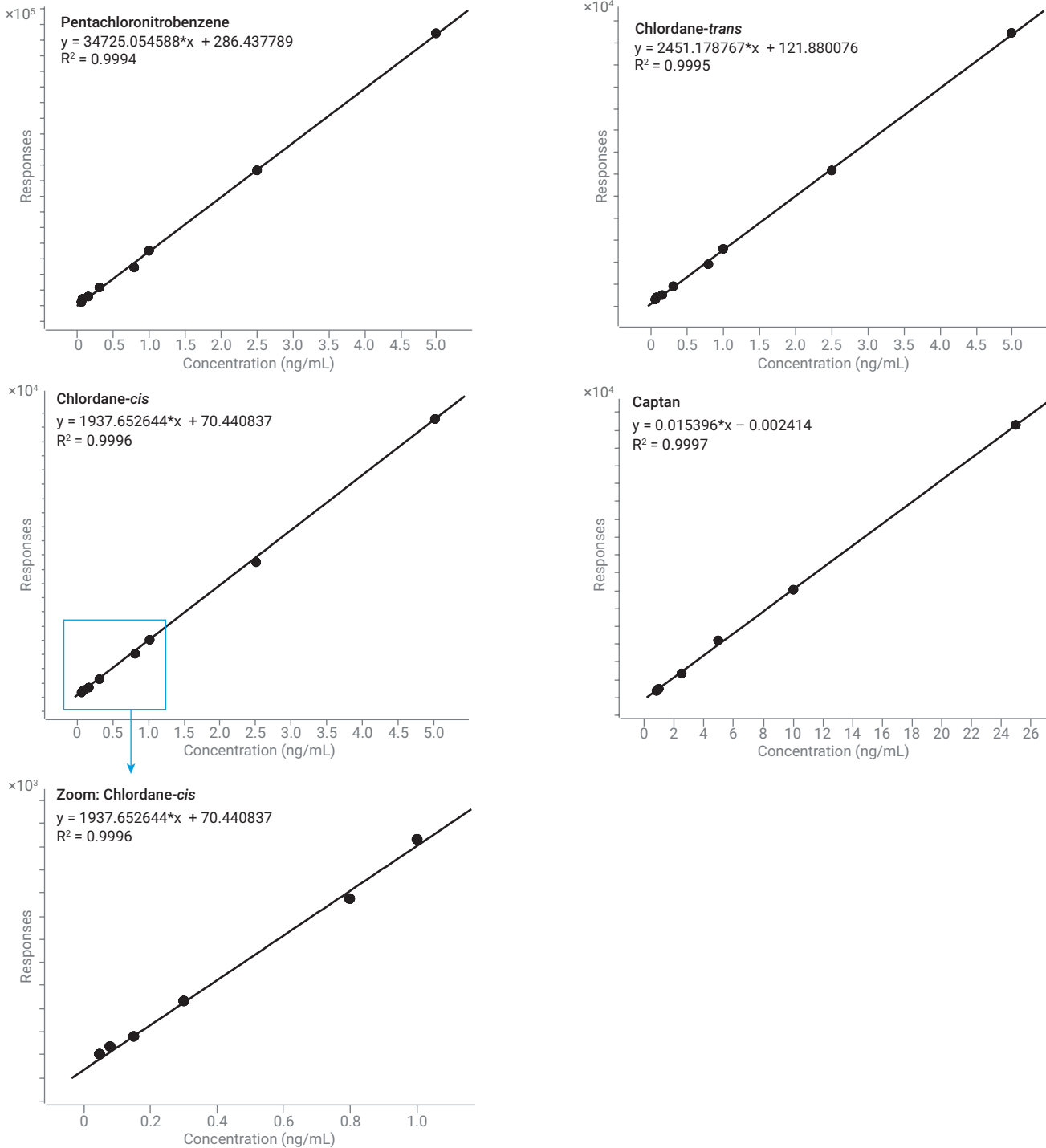

Pentachloronitrobenzene
y = 34725.054588*x + 286.437789
R^2 = 0.9994

Chlordane-*trans*
y = 2451.178767*x + 121.880076
R^2 = 0.9995

Chlordane-*cis*
y = 1937.652644*x + 70.440837
R^2 = 0.9996

Captan
y = 0.015396*x − 0.002414
R^2 = 0.9997

Zoom: Chlordane-*cis*
y = 1937.652644*x + 70.440837
R^2 = 0.9996

Figure 4. Select linear calibration curves for pentachloronitrobenzene (quintozene), chlordane-*trans*, chlordane-*cis*, and captan.

Thirty GC-amenable pesticides met LOQs currently established in Canada

Required LOQs for pesticides in cannabis established in Canada[2] are generally lower than those established in the U.S. Table 3 shows that 30 pesticides met the established and proposed LOQs in Canada. For 24 of those pesticides, LOQs mandated in Canada were lower than LOQs required in California. For example, the LOQ established by Health Canada for metalaxyl is 100-fold lower, and is 20 ppb compared to 2 ppm. The mandated LOQ was successfully met with Agilent's 8890 GC System in combination with Agilent's 7010B Triple Quadrupole GC/MS when using the analytical method described in this work.

Quantitation of kinoprene: a challenging pesticide regulated in Canada

Similar to captan, chlordane, and pentachloronitrobenzene, kinoprene is known to present a challenge for LC/MS analysis. It is also one of the challenging compounds for GC/MS. Currently, kinoprene is regulated only in Canada in fresh cannabis and plants, and in cannabis oil; however, the environmental assessment by the USEPA classifies kinoprene as *highly toxic*[4].

Figure 5 shows that Agilent's 8890 GC System in combination with Agilent's 7010B Triple Quadrupole GC/MS allowed for kinoprene quantitation with an LOQ of 0.8 ppb in-vial (100 ppb in dried cannabis plant material). MRM transitions for kinoprene were optimized in cannabis matrix with the the MassHunter Optimizer for GC/TQ.

Kinoprene
LOQ 0.8 ppb in vial (100 ppb in cannabis)
RSD 6.9 %
+EI EIC MRM (149.1, 149.0 → 77.0)

Kinoprene
y = 21325.318412*x − 10026.239695
R^2 = 0.9998
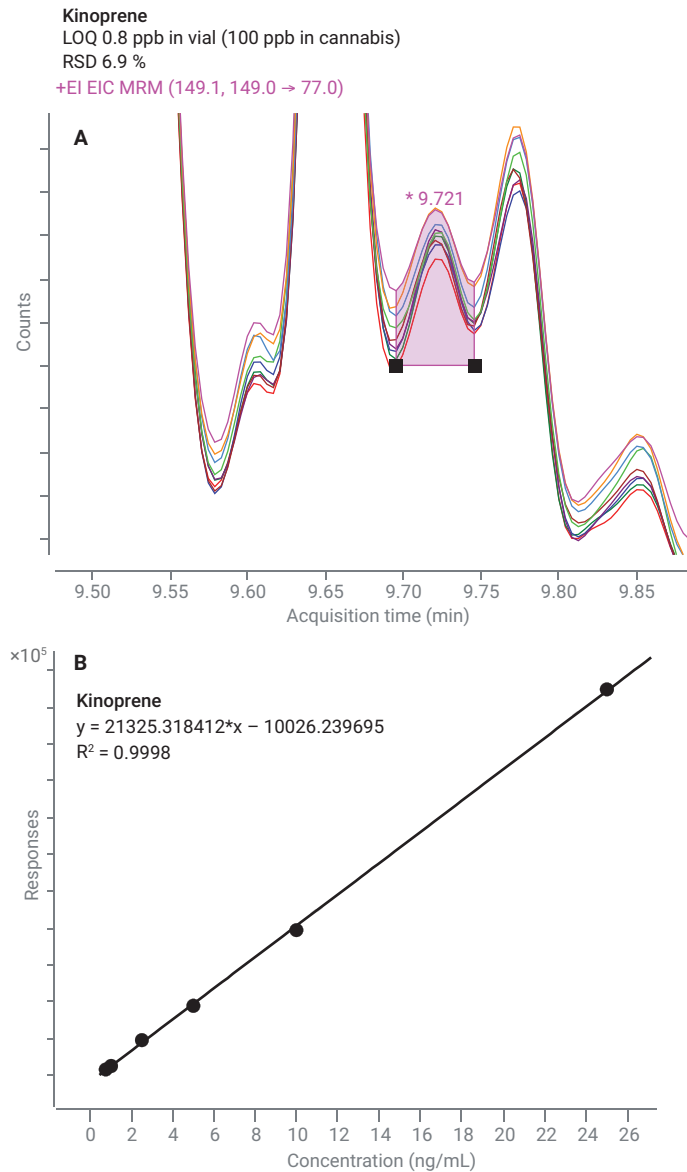
Figure 5. Ten overlaid sequential kinoprene MRM chromatograms at LOQ, and a linear calibration curve.

## Optimized sample preparation that enables the lowest LOQs

Compared to other plants and vegetables, cannabis has higher amounts of potential interferences and notably high concentrations of terpenes, cannabinoids, flavonoids, phenols, and fatty acids. The complexity of the cannabis matrix makes detection and accurate quantification of trace levels of pesticides more challenging. The Agilent recommended sample preparation approach was developed for simplicity, quick turnaround time, and to provide enough cleanup for improved sensitivity and system uptime at the same time.

Pesticides were extracted from the cannabis plant material with acetonitrile and cleaned up on an Agilent SampliQ C18 EC SPE cartridge (p/n 5982-1365) as shown in Figure 2. This is the Agilent standardized sample preparation procedure aligned with Agilent's multiplatform approach to cannabis analysis by GC/MS/MS and LC/MS/MS[3]. The resulting cannabis extracts were then further diluted prior to GC/MS/MS analysis. Analysis of cannabis plant material extract diluted less than 125-fold is not recommended as it may lead to MS source and EM detector overload (Figure 6).

A higher dilution of cannabis is suggested if required LOQs are ≥100 ppb in cannabis. LOQs of 100 ppb in 500-times diluted cannabis plant material were achieved for 85 % of 70 target pesticides with an Agilent 8890 GC System coupled to an Agilent 7010B Triple Quadrupole GC/MS equipped with an HES. The details are presented in full elsewhere[5,6]. A higher dilution factor allows for extended maintenance-free operation because of the decreased matrix load on the system (Figure 6).
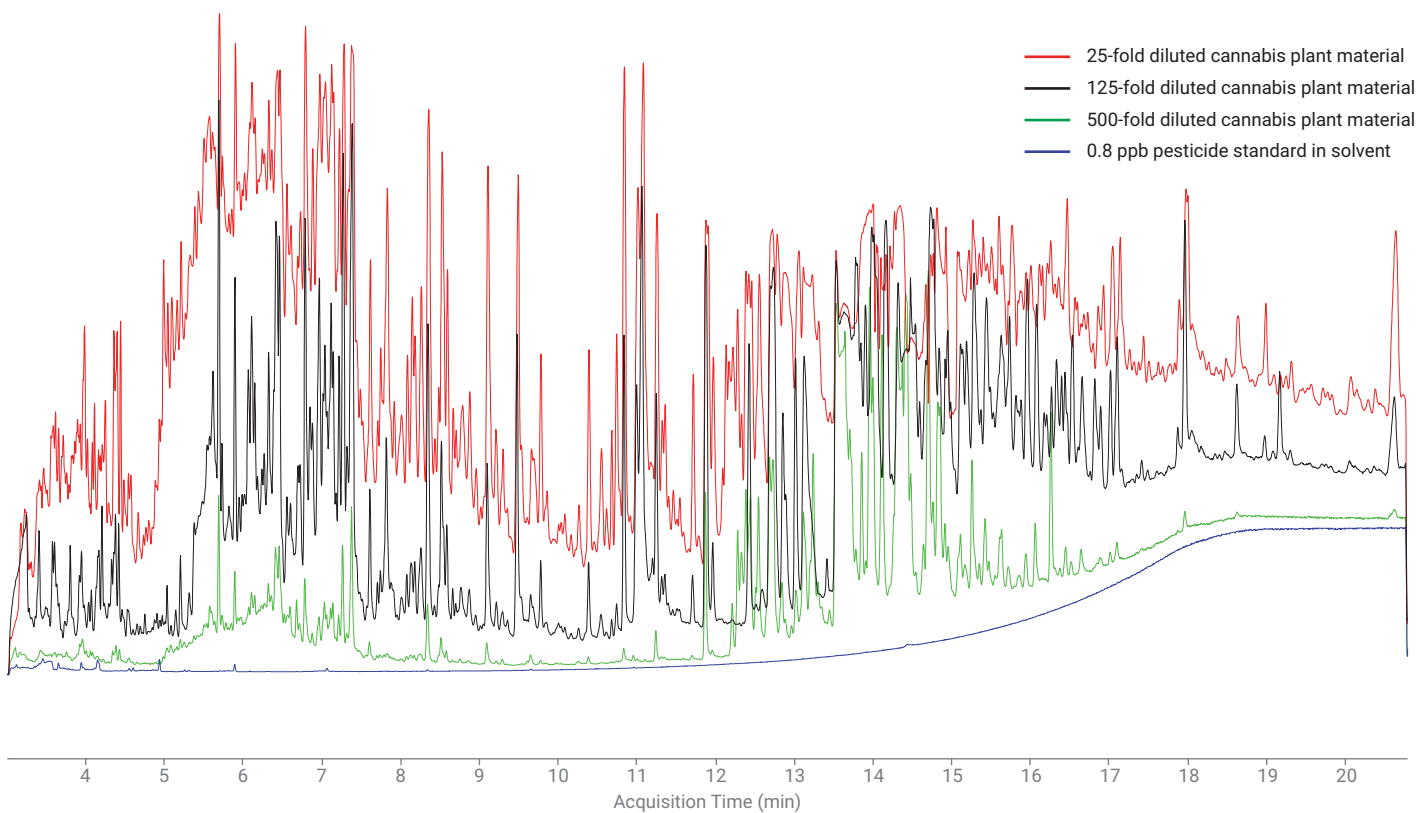
Figure 6. Scan TIC of a 25-, 125-, and 500-fold diluted cannabis extract and a 0.8 ppb (in-vial) pesticide standard in solvent, drawn at the same scale.

Conclusion

MRM transitions for the majority of pesticides from the California target list are included in the MassHunter Pesticide and Environmental Pollutant MRM Database that greatly simplified building acquisition methods. The MassHunter Optimizer for GC/TQ enabled MRM development for new compounds of interest. Backflushing reduced the need for system maintenance, which is a welcome productivity improvement for the lab. The PSD of Agilent's 8890 GC System simplifies pulsed splitless injection mode for use with column backflushing, and significantly reduces the flow of helium, conserving gas and lowering operating cost. Agilent's 7010B Triple Quadrupole GC/MS provides excellent sensitivity and selectivity in the analysis of pesticides in a complex cannabis matrix.

The LOQs established in California were met for 43 GC-amenable pesticides regulated by the Bureau of Cannabis Control. LOQs for 36 pesticides were ≤0.08 ppb in-vial (≤10 ppb in dried cannabis plant material, assuming 100 % pesticide recovery), and 43 pesticides had LOQs ≤0.8 ppb in-vial (≤100 ppb in dried cannabis plant material) when analyzing a 125-fold diluted cannabis extract.

Acknowledgement

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