

Calibration of Pesticides and Mycotoxins on GC/TQ and LC/TQ

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Introduction

Legalization of recreation and medicinal cannabis around the world is the primary driver for regulating pesticides and mycotoxins previously determined to pose a threat to human health. Due to the various classes of pesticides that are regulated, a single analytical instrument is insufficient to meet the regulatory requirements set by governing bodies. Creating streamlined calibrations with standards that can be used across multiple analytical instruments can save time and money. Agilent offers a suite of pesticide and mycotoxin standards in various forms, including neat, single component, multicomponent mixtures, and standard kits that meet specific state and country target lists. These various standards are amenable to both liquid chromatography (LC) and gas chromatography (GC) instruments with their associated triple quadrupole mass spectrometer (TQ) detectors and can be used for cannabis and hemp analysis. This application brief demonstrates achieving calibration in hemp matrix on both chromatographic instruments using the Agilent California Cannabis Pesticide Kit and Agilent Cannabis Mycotoxin Mix.

Experimental

Hemp flower bud was obtained from AccuStandard, Inc. and prepared for analysis using the sample preparation workflow outlined in Figure 1. The sample preparation is suitable for both GC/TQ and LC/TQ analysis.

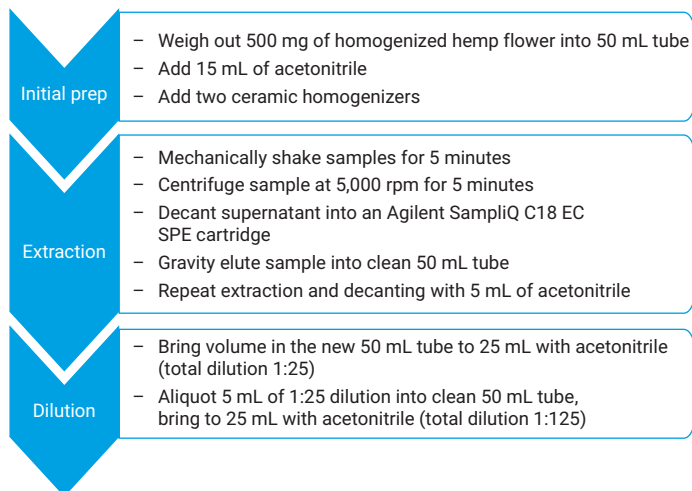


Figure 1. Sample preparation workflow for extraction of hemp flower.

Calibrants were prepared in hemp extract matrix in the range of 100 ppt to 10 ppb. For internal standards (ISTDs), a QuEChERS solution containing alpha-BHC-d6 and parathion-d10 (diethyl-d10) was diluted and added to all calibrants at 1 ppb for analysis. The calibration was carried out on both an Agilent 8890 GC with an Agilent 7010B GC/TQ and an Agilent 1290 Infinity II LC with an Agilent 6470B LC/TQ. Parameters for each instrument are listed in Tables 1 to 4.

Table 1. GC method parameters.

GC Parameters	Agilent 8890 GC
Configuration	Midcolumn backflush
Inlet	Multimode inlet
Inlet Mode	Solvent vent
Inlet Temperature Program	70 °C (hold for 0.5 min), ramp at 600 °C/min to 300 °C (hold 14.8 min), ramp at 600 °C/min to 330 °C
Column 1	Agilent J&W HP-5ms Ultra Inert, 15 m × 0.25 mm × 0.25 µm
Column 2	Agilent J&W HP-5ms Ultra Inert, 15 m × 0.25 mm × 0.25 µm
Flow (Column 1)	He, 1.19 mL/min (postrun backflush 8.6 mL/min)
Flow (Column 2)	He, 1.39 mL/min (postrun backflush 9.02 mL/min)
Oven	60 °C (hold for 1 min), ramp at 40 °C/min to 170 °C, ramp at 10 °C/min to 330 °C (hold for 0.25 min)
Injection	2 µL
Injection Mode	Two-layer sandwich (L1, L2), 0.2 µL air gap
Plunger Speed	Variable

Table 2. GC/TQ method parameters.

GC/TQ Parameters	Agilent 7010B GC/TQ
Ion Source	High-efficiency ion source
Acquisition Mode	Dynamic MRM
Solvent Delay	3 min
Tune File	Atune
Gain	20
MS Source Temperature	300 °C
MS Quadrupole Temperature	150 °C
Transfer Line Temperature	330 °C

Table 3. LC method parameters.

LC Parameters	Agilent 1290 Infinity II LC
Flow Rate	0.5 mL/min
Mobile Phase A	5 mM ammonium formate + 0.1% formic acid in water
Mobile Phase B	0.1% formic acid in acetonitrile
Column Temperature	35 °C
Injection	5 µL
Gradient	Time (min) % B
	0.0 30
	1.0 30
	2.0 75
	8.0 96
	9.0 100
	9.5 100
9.51 30	

Table 4. LC/TQ method parameters.

LC/TQ Parameters	Agilent 6470B LC/TQ
Acquisition	Dynamic MRM
Polarity	Positive
Capillary Voltage	4,500 V
Drying Gas Flow	10 L/min
Drying Gas Temperature	200 °C
Nebulizer Pressure	35 psi
Sheath Gas Temperature	200 °C
Sheath Gas Flow	10 L/min
Nozzle Voltage	3,500 V
Q1 and Q2 Resolution	0.7 amu
Delta Emv Voltage	0

Results and discussion

The two analytical platforms both yielded excellent calibrations in matrix for the California pesticide list, which includes 66 pesticides and five mycotoxins. Extracted MRM chromatograms for each of the analytes are overlaid and shown for GC and LC in Figure 2.

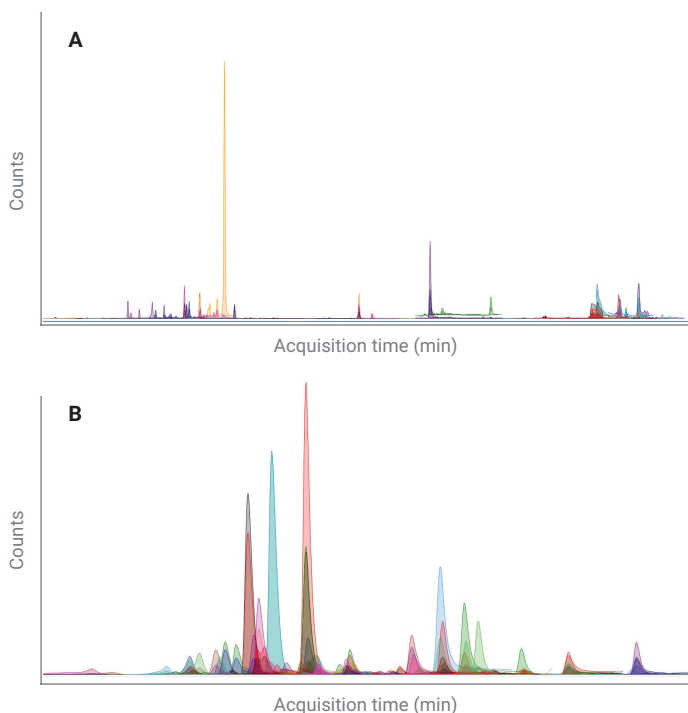


Figure 2. Overlaid chromatograms of pesticides analyzed by GC/TQ (A) and LC/TQ (B). The GC chromatogram contains 27 analytes along with ISTDs parathion-d10 (diethyl-d10) and alpha-BHC-d6. The LC chromatogram contains the five mycotoxins, remaining 39 pesticides, and ISTDs parathion-d10 (diethyl-d10) and alpha-BHC-d6.

The total list of analytes from the California list, which is represented in the chromatograms, is shown in Table 5. For each analyte, the chromatographic method used to obtain the calibration is noted.

Sample calibrations obtained on the LC/TQ (Figure 3) and GC/TQ (Figure 4) are shown. Each calibration contained eight levels in the range of 100 ppt to 10 ppb, which were analyzed in triplicate.

Table 5. California pesticide list of monitored analytes with mycotoxins.

Parameter	Value	Parameter	Value
Aldicarb	LC	Cypermethrin	LC
Carbofuran	GC	Diazinon	GC
Chlordane	GC	Dimethomorph	LC
Chlorfenapyr	GC	Etoazol	LC
Chlorpyrifos	GC	Fenpyroximate	LC
Coumaphos	GC	Bifenazate	LC
Daminozide	LC	Fenhexamid	LC
Dichlorvos	GC	Flonicamid	LC
Dimethoate	GC	Fludioxonil	GC
Ethoprop	GC	Trifloxystrobin	LC
Etofenprox	GC	Aflatoxin G1	LC
Fenoxycarb	GC	Aflatoxin G2	LC
Fipronil	LC	Hexythiazox	LC
Imazalil	LC	Imidacloprid	GC
Methiocarb	LC	Kresoxim-methyl	GC
Methyl parathion	GC	Malathion	GC
Mevinphos	GC	Metalaxyl	GC
Paclobutrazol	GC	Methomyl	GC
Propoxur	GC	Myclobutanil	GC
Spiroxamine	LC	Naled	GC
Thiacloprid	LC	Oxamyl	LC
Thiamethoxam	LC	Pentachloronitrobenzene	GC
Aflatoxin B1	LC	Permethrins	GC
Aflatoxin B2	LC	Phosmet	LC
Abamectin	LC	Piperonyl butoxide	LC
Acephate	LC	Prallethrin	LC
Acequinocyl	LC	Propiconazole	LC
Acetamiprid	LC	Pyrethrins	GC
Azoxystrobin	LC	Pyridaben	LC
Bifenthrin	LC	Spinetoram	LC
Boscalid	GC	Spinosad	LC
Captan	GC	Spiromesifen	LC
Carbaryl	LC	Spirotetramat	LC
Chlorantraniliprole	LC	Tebuconazole	GC
Clofentazine	LC	Ochratoxin	LC
Cyfluthrin	GC		

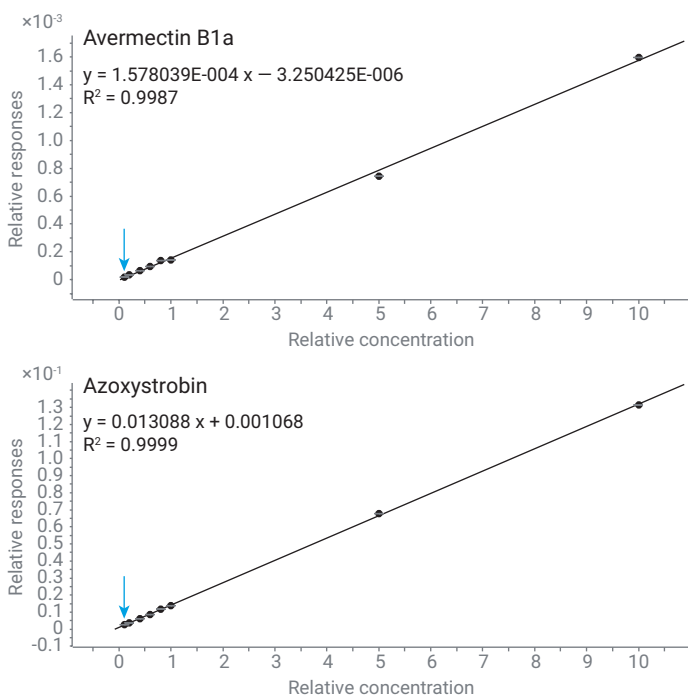


Figure 3. Calibration curves for avermectin B1a (as abamectin) and azoxystrobin analyzed by LC/TQ.

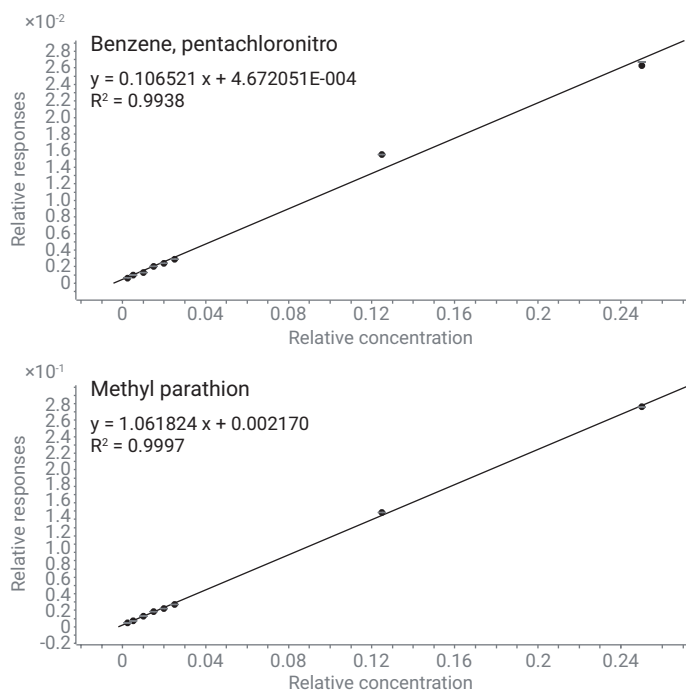


Figure 4. Calibration curves for pentachloronitrobenzene and methyl parathion analyzed by GC/TQ.

Conclusion

Agilent California Cannabis Pesticide Kit and Agilent Cannabis Mycotoxin Mix were successfully used to calibrate on two analytical platforms, GC/TQ and LC/TQ, using a single sample preparation method.

Appendix

Table 6. Chemical standards referenced in this application.

Part Number	Agilent Cannabis Pesticide and Mycotoxin Standards
PST-CBS-CA	California Cannabis Pesticide Kit (2020)
PST-CBS-CAN	Canada Cannabis Pesticide Kit (2020)
PST-CBS-OR	Oregon Cannabis Pesticide Kit (2020)
PST-CBS-NV	Nevada Cannabis Pesticide Mix (2020)
PST-CBS-CO	Colorado Cannabis Pesticide Mix (2020)
TOX-CBS-Mix1	Cannabis Mycotoxin Mix

Part Number	Agilent Cannabis Pesticide and Mycotoxin Standards
TOX-UNI-AflaM1	Aflatoxin M1 (1 ppm)
TOX-UNI-AflaM2	Aflatoxin M2 (1 ppm)
TOX-UNI-DON	Deoxynivalenol (100 ppm)
TOX-UNI-ZON	Zearalenone (100 ppm)
PPS-500X	QuEChERS triphenyl phosphate
TOX-UNI-FumoB1	Fumonisin B1 (50 ppm)
TOX-UNI-FumoB2	Fumonisin B2 (50 ppm)
TOX-UNI-HT2	HT-2 Toxin (100 ppm)
TOX-UNI-T2	T-2 Toxin (100 ppm)
TOX-UNI-AflaB1	Aflatoxin B1 (10 ppm)
TOX-UNI-AflaB2	Aflatoxin B2 (10 ppm)
TOX-UNI-AflaG1	Aflatoxin G1 (10 ppm)
TOX-UNI-AflaG2	Aflatoxin G2 (10 ppm)
TOX-UNI-OchrA	Ochratoxin A (100 ppm)
PPS-610-1	QuEChERS IS standard no. 6

To see the complete lists of lab supplies for cannabis and hemp workflows, please visit www.agilent.com/chem/cannabis-workflow-ordering-guide.

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