Unlock Ultra-Low Analysis of Pesticides in Cannabis by GC-MS/MS

Keep Your Peaks in Good Shape with a Highly Inert Sample Flow Path

By Erica Pack, PhD

- Maximize data quality—TriMax and Topaz deactivations improve peak shapes and responses at trace levels.
- Achieve picogram-level sensitivity across a wide range of challenging GC-amenable pesticides.
- Reliably identify pesticides, even in complex cannabis flower sample matrix.

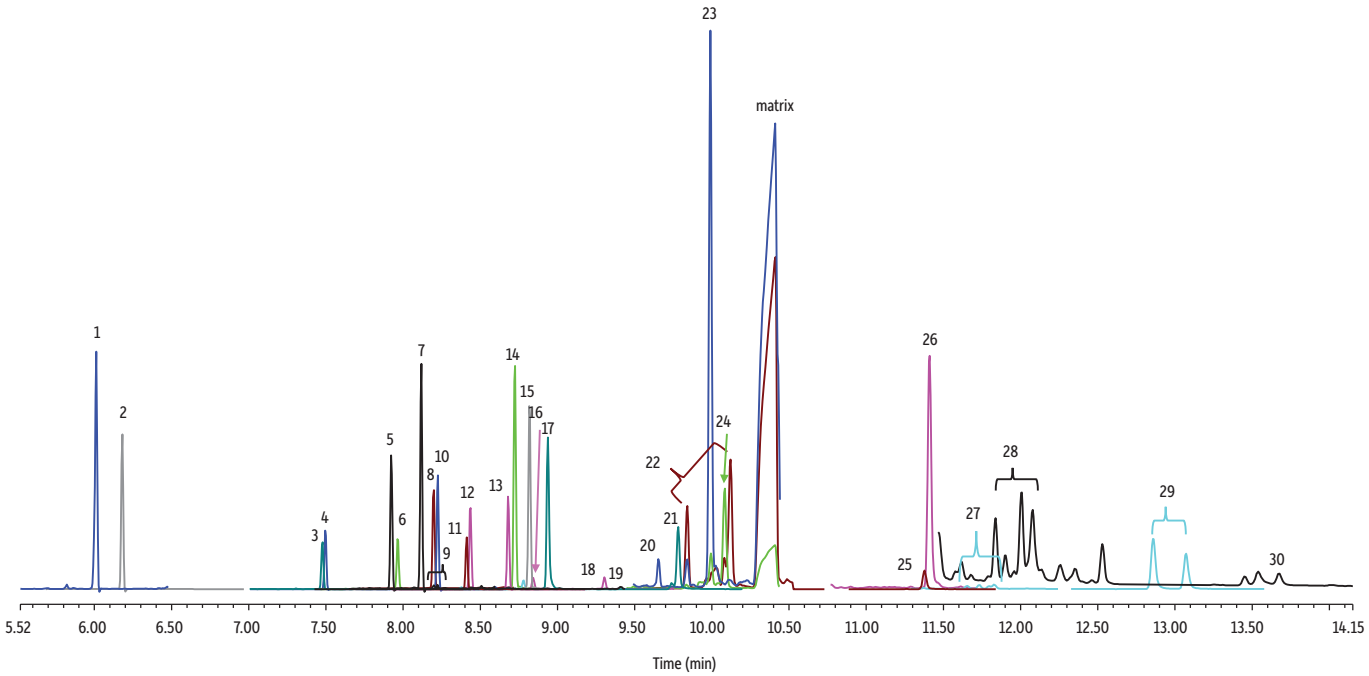
Pesticide analysis is a continuous challenge for cannabis testing labs due to the changing regulatory landscape and the complexity of analytes and matrices. Regulations continue to fluctuate state-to-state, monitoring different analytes at different levels. In addition, pesticide chemistries vary considerably, and labs face a prevalence of harsh matrices ranging from plant material to edibles to concentrates and more. Diluting samples can be an effective strategy for extending instrument uptime because less matrix is introduced, but it means the target analytes are also present at lower levels, making them more challenging to detect.

For accurate, low-level analysis of pesticides in cannabis, a highly inert sample flow path is needed, so column quality becomes critical. If the column is not sufficiently inert, interactions between the analytes and imperfections (active sites) on the column surface can occur, which negatively affects peak shape and response. These problems are traditionally exacerbated at low levels, but they can be overcome by using an RMX-5Sil MS column. This column is an exceptionally inert, long-lasting, high-efficiency column that performs well for sensitive GC-MS/MS analyses. It is powered by the novel TriMax deactivation, which provides a robust interface between the polymer and capillary tubing. This results in a highly inert surface that resists degradation from thermal and matrix-related stressors, keeping the instrument running at maximum performance longer.

To demonstrate its performance, we subjected the RMX-5Sil MS column to low-level analysis of pesticides in cannabis flower—a notoriously difficult matrix. We paired the column with a Topaz inlet liner for maximum inertness throughout the sample flow path. To assess analytical results independent of sample prep efficiency (since techniques vary greatly lab to lab), cannabis flower extract (see chromatogram) was spiked post-extraction at 1.7 ng/mL with 30 pesticides covering a range of chemical functionalities. This level is comparable to 0.1 µg/g pesticide in cannabis flower and ultimately presented as 0.17 pg on-column.

Highly symmetrical peak shapes were obtained on the RMX-5Sil MS column, facilitating easy, accurate integration. Across all pesticides over triplicate injections, the average peak symmetry was 1.00 ± 0.14, showing that even at extremely low levels in a difficult matrix, the sample flow path is inert, and pesticide peaks are in the preferred 0.8-1.2 symmetry window. Sensitive systems, such as GC-MS/MS, require inert consumables to maintain maximum performance, and, as shown here, the RMX-5Sil MS column and Topaz liner are ideal for the analysis of pesticides in cannabis flower.

Figure 1: Achieve superior peak shapes for pesticides in cannabis at trace levels on-column with highly inert RMX-5Sil MS columns and Topaz liners.



Peaks	t_R (min)	Quant SRM	Qual SRM
1. Mevinphos	6.011	127.0 / 109.1	192.0 / 127.1
2. Etridiazole	6.178	211.0 / 183.0	182.9 / 140.0
3. Pentachloronitrobenzene	7.478	213.9 / 179.0	236.9 / 143.0
4. Diazinone	7.498	152.1 / 137.1	137.0 / 84.1
5. Parathion methyl	7.922	263.0 / 109.1	109.0 / 79.0
6. Metalaxyl-m	7.963	132.1 / 117.1	206.1 / 132.1
7. Malathione	8.118	127.1 / 99.0	173.1 / 117.1
8. Chlorpyrifos	8.194	197.0 / 169.0	314.0 / 257.9
9. Allethrin	8.219	123.1 / 122.1	79.1 / 77.1
10. Fenthion	8.224	278.0 / 109.0	278.0 / 245.1
11. Fipronil	8.409	213.0 / 143.0	- / 212.9
12. Cyprodinil	8.434	224.1 / 222.2	210.1 / 208.1
13. Tetrachlorvinphos	8.68	328.9 / 109.1	109.0 / 79.0
14. Paclobutrazol	8.72	236.1 / 125.1	125.0 / 89.1
15. Fludioxonil	8.816	248.0 / 127.1	127.1 / 100.1

Peaks	t_R (min)	Quant SRM	Qual SRM
16. Endosulfan I	8.84	240.9 / 121.1	240.9 / 199.1
17. Myclobutanil	8.936	179.1 / 125.1	152.0 / 125.0
18. Iprodione	9.207	244.0 / 160.0	244.0 / 187.0
19. Endosulfan II	9.302	195.0 / 159.0	207.0 / 172.0
20. Piperonyl butoxide	9.729	176.1 / 91.1	176.1 / 146.1
21. Tebuconazole	9.739	250.1 / 125.1	250.1 / 153.1
22. Tetramethrin	9.941	164.1 / 107.1	123.1 / 81.1
23. Bifenthrin	9.991	181.1 / 165.1	166.1 / 165.1
24. Phosmet	10.072	160.0 / 77.1	133.0 / 77.1
25. Pyridaben	11.26	147.1 / 117.1	117.1 / 115.1
26. Coumaphos	11.369	226.0 / 163.1	109.0 / 81.0
27. Cyfluthrin	11.651	206.1 / 151.1	163.0 / 91.1
28. Cypermethrin	11.954	181.1 / 152.1	163.0 / 91.1
29. Fenvalerate	12.865	125.0 / 89.1	167.1 / 125.1
30. Deltamethrin	13.681	181.1 / 152.1	252.9 / 174.0

Column	RMX-5Sil MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 17323)
Standard/Sample	Canadian pesticide standards kit (cat.# 32592)
Diluent:	1:1 hexane:acetone (1% acetic acid)
Conc.:	1.7 ng/mL (0.17 pg on-column)
Injection	
Inj. Vol.:	1 µL split (split ratio 10:1)
Liner:	Topaz 5.0 mm ID Precision inlet liner w/wool (cat.# 23327)
Inj. Temp.:	280 °C
Oven	
Oven Temp.:	50 °C (hold 1 min) to 270 °C at 30 °C/min to 300 °C at 5 °C/min (hold 0.5 min)
Carrier Gas	He, constant flow
Flow Rate:	1.4 mL/min @ 50 °C
Detector	TSQ 9000 MS/MS
Transfer Line Temp.:	300 °C
Aperture Size:	6 mm ID
Source Temp.:	300 °C
Tune Type:	PFTBA
Ionization Mode:	EI
Instrument	Thermo Trace GC
Sample Preparation	Cannabis flower extract was spiked at 1.7 ng/mL with select pesticides featured in the Canadian pesticides standards kit (cat.# 32592). Extracts represent a 1:5 ratio of cannabis flower in acetonitrile (1% acetic acid) further diluted in hexane and acetone. Conc. reflects approximately 0.1 µg/g pesticide in flower.

Featured Products

RMX-5Sil MS GC Capillary Column

Catalog No.	Product Name	Units
17323	RMX-5Sil MS GC Capillary Column, 30 m, 0.25 mm ID, 0.25 µm	ea.

Topaz Split Precision Inlet Liner

Catalog No.	Product Name	Units
23327	Topaz, Split Precision Inlet Liner, 5 mm x 8.0 x 105, for Thermo GCs, w/Quartz Wool, Premium Deactivation	5-pk.

Restek Electronic Leak Detector

Catalog No.	Product Name	Units
28500	Restek Electronic Leak Detector (includes carrying case; universal AC power adaptor [U.S., UK, Europe, Australia, Japan]; 6-ft USB charging cable)	ea.

Restek ProFLOW 6000 Electronic Flowmeter

Catalog No.	Product Name	Units
22656	Restek ProFLOW 6000 Electronic Flowmeter	ea.

Canadian Pesticide Kit

Cat.# 32593: Canadian Pesticide Standard #1 (15 components)
Aldicarb (116-06-3)
Bifenazate (149877-41-8)
Carbaryl (Sevin) (63-25-2)
Carbofuran (1563-66-2)
Fenoxycarb (72490-01-8)
Iprodione (36734-19-7)
Methiocarb (2032-65-7)
Methomyl (16752-77-5)
Novaluron (116714-46-6)
Oxamyl (23135-22-0)
Pirimicarb (23103-98-2)
Propoxur (Baygon) (114-26-1)
Pyraclostrobin (175013-18-0)
Teflubenzuron (83121-18-0)
Thiophanate-methyl (23564-05-8)

Cat.# 32594: Canadian Pesticide Standard #2 (14 components)
Acephate (30560-19-1)
Chlorpyrifos (2921-88-2)
Coumaphos (56-72-4)
Diazinon (333-41-5)
Dimethoate (60-51-5)
Ethoprophos (13194-48-4)
Fensulfothion (115-90-2)
Fenthion (55-38-9)
Malathion (121-75-5)
Methyl parathion (298-00-0)
Mevinphos (7786-34-7)
Naled (300-76-5)
Phosmet (732-11-6)
Tetrachlorvinphos (22248-79-9)

Cat.# 32595: Canadian Pesticide Standard #3 (18 components)
Abamectin (71751-41-2)
Allethrin (584-79-2)
Bifenthrin (82657-04-3)
Cyfluthrin (68359-37-5)
Cypermethrin (52315-07-8)
Deltamethrin (52918-63-5)
Etofenprox (80844-07-1)
Fenvalerate (51630-58-1)
Permethrin (cis & trans) (52645-53-1)
Phenothrin (cis & trans) (26002-80-2)
Prallethrin (23031-36-9)
Pyrethrins (8003-34-7)
Resmethrin (10453-86-8)
Spinetoram (J&L) (935545-74-7)*
Spinosad (A&D) (168316-95-8)†
Spirodiclofen (148477-71-8)
Spiromesifen (283594-90-1)
Tetramethrin (7696-12-0)

Cat.# 32596: Canadian Pesticide Standard #4 (16 components)
Azoxystrobin (131860-33-8)
Chlorfenapyr (122453-73-0)
Clofentezine (74115-24-5)
Cyprodinil (121552-61-2)
Daminozide (1596-84-5)
Etoxazole (153233-91-1)
Etridiazole (2593-15-9)
(E)-Fenpyroximate (134098-61-6)
Fipronil (120068-37-3)
Fludioxonil (131341-86-1)
Imazalil (35554-44-0)
Myclobutanil (88671-89-0)
Paclobutrazol (76738-62-0)
Propiconazole (Tilt) (60207-90-1)
Pyridaben (96489-71-3)
Tebuconazole (107534-96-3)

Cat.# 32597: Canadian Pesticide Standard #5 (17 components)
Benzovindiflupyr (1072957-71-1)
Boscalid (188425-85-6)
Buprofezin (69327-76-0)
Chlorantraniliprole (500008-45-7)
Clothianidin (210880-92-5)
Cyantraniliprole (736994-63-1)
Dimethomorph (110488-70-5)
Dinotefuran (165252-70-0)
Flonicamid (158062-67-0)
Fluopyram (658066-35-4)
Hexythiazox (78587-05-0)
Imidacloprid (138261-41-3)
Metalaxyl (57837-19-1)
Pentachloronitrobenzene (82-68-8)
Spirotetramat (203313-25-1)
Tebufenozide (112410-23-8)
Thiamethoxam (153719-23-4)

Cat.# 32598: Canadian Pesticide Standard #6 (14 components)
Acequinocyl (57960-19-7)
Acetamiprid (135410-20-7)
Dodemorph (1593-77-7)
Endosulfan I (959-98-8)
Endosulfan II (33213-65-9)
Endosulfan sulfate (1031-07-8)
Kinoprene (42588-37-4)
Kresoxim methyl (143390-89-0)
Methoprene (40596-69-8)
MGK-264 (113-48-4)
Piperonyl butoxide (51-03-6)
Spiroxamine (118134-30-8)
Thiacloprid (111988-49-9)
Trifloxystrobin (141517-21-7)

Cat.# 32599: Azadirachtin Standard
Azadirachtin (11141-17-6)

Cat.# 32585: Dichlorvos Standard
Dichlorvos (DDVP) (62-73-7)

Conc. in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	qty.	cat.#
Canadian Pesticide Kit							
1 mL/ampul, 8 ampuls/kit	Yes	6 months	14 months	Ambient	–20 °C or colder	kit	32592
Note: The kit contains one ampul of each of the following. Each ampul may also be purchased individually.							
Canadian Pesticide Standard #1							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	24 months	Ambient	–20 °C or colder	ea.	32593
Canadian Pesticide Standard #2							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	36 months	Ambient	0 °C or colder	ea.	32594
Canadian Pesticide Standard #3							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	24 months	Ambient	–20 °C or colder	ea.	32595
Canadian Pesticide Standard #4							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	36 months	Ambient	–20 °C or colder	ea.	32596
Canadian Pesticide Standard #5							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	36 months	Ambient	0 °C or colder	ea.	32597
Canadian Pesticide Standard #6							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	36 months	Ambient	–20 °C or colder	ea.	32598
Azadirachtin Standard (CAS # 11141-17-6)							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	36 months	Ambient	0 °C or colder	ea.	32599
Dichlorvos Standard (CAS # 62-73-7)							
100 µg/mL, Acetonitrile, 1 mL/ampul	Yes	6 months	14 months	Ambient	0 °C or colder	ea.	32585

* This reference material contains both the Spinetoram J and L isomers (187166-40-1 and 187166-15-0); however, CAS# 935545-74-7 is displayed on the certificate as this is the neat material dissolved in the solution. CAS# 935545-74-7 is a blend of Spinetoram J and L, and the ratio of each material isomer are displayed on your certificate of analysis.

† This reference material contains Spinosad A and D, isomers (131929-60-7 and 131929-63-0); however, CAS# 168316-95-8 is displayed on the certificate as this is the neat material dissolved in the solution. CAS# 168316-95-8 is a blend of Spinosad A and D, and the ratio of each material isomer are displayed your certificate of analysis.