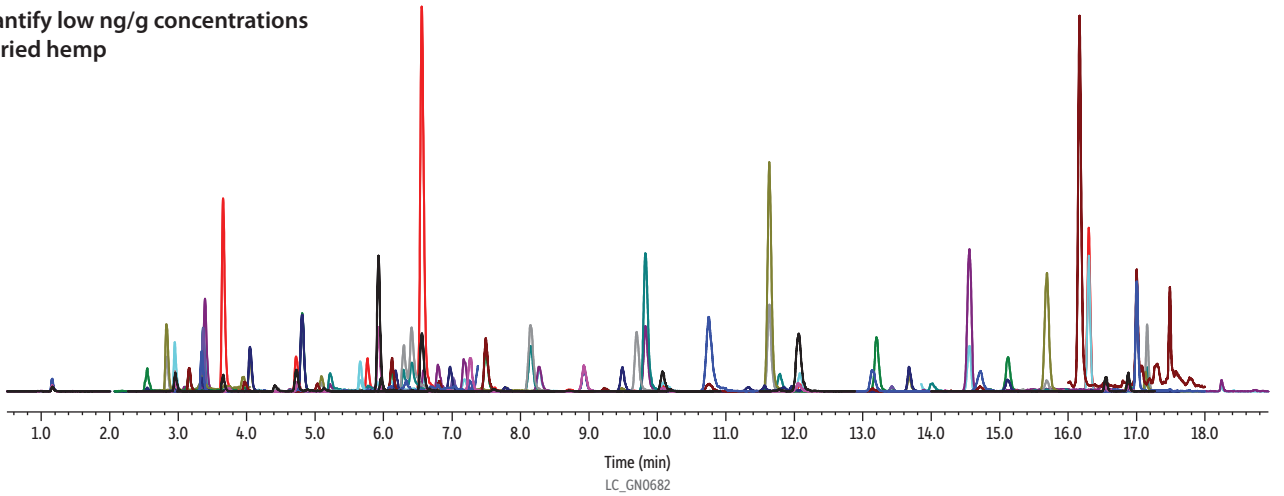
California Cannabis Pesticides and Mycotoxins in Dried Hemp on Raptor ARC-18

- LC amenable pesticides and mycotoxins regulated by California (2022)
- Quantify low ng/g concentrations in dried hemp



	Peaks	tR (min)	Precursor Ion	Product Ion 1	Product Ion 2	Polarity
1.	Daminozide	1.2	161.1	143.2	44.1	+
2.	Daminozide-d6	1.2	167.0	149.3		+
3.	Acephate	2.5	184.0	143.1	95.1	+
4.	Oxamyl	2.8	237.1	72.1	90.1	+
5.	Flonicamid	2.9	230.1	203.1	174.1	+
6.	Methomyl	2.9	163.1	88.1	106.1	+
7.	Thiamethoxam	2.9	292.0	211.1	181.1	+
8.	Imidacloprid	3.2	256.1	209.1	175.1	+
9.	Clothianidin	3.2	250.1	169.2	132.3	+
10.	Dimethoate-d6	3.3	236.1	205.1		+
11.	Mevinphos I	3.3	225.1	127.1	193.2	+
12.	Dimethoate	3.4	230.0	199.1	125.1	+
13.	Acetamiprid	3.4	223.0	126.1	56.1	+
14.	Thiacloprid	3.7	253.0	126.0	90.1	+
15.	Mevinphos II	3.7	225.1	127.1	193.2	+
16.	Aflatoxin G2	3.7	331.2	189.3	115.2	+
17.	Aflatoxin G1	4.0	329.2	243.2	215.3	+
18.	Aldicarb	4.0	208.4	116.0	89.0	+
19.	Aflatoxin B2	4.4	315.3	287.2	243.3	+
20.	Dichlorvos (DDVP)	4.6	220.9	108.8	79.2	+
21.	Propoxur (Baygon)	4.7	210.1	111.1	93.1	+
22.	Aflatoxin B1	4.8	313.2	241.2	128.2	+
23.	Carbofuran	4.8	222.1	123.1	165.2	+
24.	Carbary-d7	5.0	209.2	125.2		+
25.	Carbaryl (Sevin)	5.1	202.1	145.1	127.1	+
26.	Imazalil	5.2	297.0	159.0	201.0	+
27.	Atrazine-d5	5.7	221.2	179.1		+
28.	Diuron-d6	5.8	239.1	78.2		+
29.	Metalaxyl	5.9	280.2	220.2	192.2	+
30.	Naled	6.0	397.8	127.1	109.1	+
31.	Chlorantraniliprole	6.1	483.9	452.9	285.9	+
32.	Phosmet	6.2	318.0	160.1	77.2	+
33.	Spiroxamine	6.3	298.3	144.2	100.2	+
34.	Linuron-d6	6.3	255.1	160.1		+
35.	Azoxystrobin	6.6	404.0	372.1	344.1	+
36.	Methiocarb	6.6	226.1	169.1	121.1	+
37.	Fludioxonil	6.6	274.0	180.0	126.0	-
38.	Dimethomorph I	6.8	388.2	301.2	165.3	+
39.	Boscalid	6.8	342.9	307.5	140.1	+
40.	Paclobutrazol	7.0	294.3	70.1	125.1	+
41.	Malathion	7.0	331.0	127.2	285.2	+
42.	Dimethomorph II	7.2	388.2	301.2	165.3	+
43.	Myclobutanil	7.3	289.1	70.1	125.1	+
44.	Bifenazate	7.5	301.0	198.1	170.2	+
45.	Ochratoxin A	7.6	404.2	239.1	358.3	+
46.	Fenhexamid	7.8	302.1	97.1	55.2	+
47.	Spirotetramat	8.2	374.2	216.1	302.1	+
48.	Ethoprophos	8.3	243.1	131.1	97.1	+
49.	Fipronil	8.7	436.8	331.8	251.9	-
50.	Fenoxycarb	8.9	302.1	88.1	116.1	+
51.	Kresoxim methyl	9.3	314.2	267.2	222.2	+
52.	Tebuconazole	9.5	308.1	70.1	125.1	+
53.	Diazinon-d10	9.7	315.2	170.2		+
54.	Diazinon	9.8	305.1	169.2	153.2	+
55.	Coumaphos	10.1	363.1	227.1	307.1	+
56.	Propiconazole (Tilt)	10.1	342.0	159.0	69.2	+
57.	Clofentezine	10.2	303.0	138.1	102.1	+
58.	Spinosad (Spinosyn A)	10.8	732.4	142.2	98.1	+
59.	MGK-264 I	11.1	276.2	210.1	121.1	+
60.	Prallethrin	11.6	301.2	123.2	104.9	+
61.	Trifloxystrobin	11.6	409.2	186.1	145.1	+
62.	Spinosad (Spinosyn D)	11.8	746.5	142.3	98.4	+
63.	Spinetoram (Spinosyn J)	12.1	748.5	142.5	98.3	+
64.	Pyrethrins (Pyrethrin II)	12.1	373.1	161.1	133.2	+
65.	Spinetoram (Spinosyn L)	13.2	760.5	142.2	98.1	+
66.	Piperonyl butoxide-d9	13.2	365.4	177.4		+
67.	Piperonyl butoxide	13.3	356.3	177.2	119.2	+
68.	Chlorpyrifos	13.4	349.9	198.0	97.1	+
69.	Hexythiazox	13.7	353.1	228.1	168.1	+
70.	Etoxazole	14.6	360.2	141.1	304.2	+
71.	Spiromesifen	14.7	388.4	273.3	255.3	+
72.	Pyrethrins (Pyrethrin I)	15.1	329.2	161.2	133.2	+
73.	Cyfluthrin	15.3	451.1	191.2		+
74.	Cyfluthrin (qualifier)	15.3	453.1	193.2		+
75.	Cypermethrin	15.6	433.1	191.0	416.0	+
76.	(E)-Fenpyroximate	15.7	422.2	366.1	138.1	+
77.	Pyridaben	16.3	365.1	147.2	309.2	+
78.	Permethrin (trans)	16.6	408.3	183.2	355.1	+
79.	Permethrin (cis)	16.9	408.3	183.2	355.1	+
80.	Abamectin	16.9	890.5	305.4	567.4	+
81.	Etofenprox	17.0	394.3	177.2	359.3	+
82.	Bifenthrin-d6	17.1	446.3	181.3		+
83.	Bifenthrin	17.2	440.0	181.2	166.2	+
84.	Acequinocyl	18.2	402.3	343.2	189.0	+

Column	Raptor ARC-18 (cat.# 9314A62)
Dimensions:	150 mm x 2.1 mm ID
Particle Size:	2.7 µm
Pore Size:	90 Å
Guard Column:	Raptor ARC-18 EXP guard column cartridge 5 mm, 2.1 mm ID, 5 µm (cat.# 9314A0252)
Temp.:	40 °C
Standard/Sample	California Pesticide Standard #1 (cat.# 34124) California Pesticide Standard #2 (cat.# 34125) California Pesticide Standard #3 (cat.# 34126) California Pesticide Standard #4 (cat.# 34127) California Pesticide Standard #5 (cat.# 34128) California Pesticide Standard #6 (cat.# 34129) Aflatoxins (B1, B2, G1, G2) Standard (cat.# 34121) Ochratoxin A (cat.# 34122) Dimethoate-d6 (cat.# 31988) Carbaryl-d7 (cat.# 31985) Diazinon-d10 (diethyl-d10) (cat.# 31986) Atrazine-d5 (cat.# 31984) Diuron-d6 (cat.# 31989) Linuron-d6 (cat.# 31990) Compounds not present in these mixes were obtained separately.
Diluent:	67.4:21.2:11.4 Acetonitrile:methanol:water
Conc.:	6-12 ng/mL (Expected concentration range in extract of hemp initially spiked at 100 ng/g.)
Inj. Vol.:	1.5 µL
Mobile Phase	
A:	Water, 2 mM ammonium formate, 0.1% formic acid
B:	Methanol, 2 mM ammonium formate, 0.1% formic acid

Time (min)	Flow (mL/min)	%A	%B
0.00	0.4	95	5
1.0	0.4	50	50
2.5	0.4	50	50
4.0	0.4	35	65
7.0	0.4	35	65
7.5	0.4	30	70
9.0	0.4	30	70
9.5	0.4	25	75
11.0	0.4	25	75
11.5	0.4	20	80
13.5	0.4	20	80
15.5	0.4	5	95
16.5	0.4	0	100
19.5	0.4	0	100
19.6	0.4	95	5

Detector	MS/MS (Shimadzu LCMS-8045)
Ion Source:	Electrospray
Ion Mode:	ESI+/ESI-
Mode:	MRM
Instrument	UHPLC (Shimadzu Nexera X2)
Sample Preparation	1 g of pulverized hemp was weighed in a 15 mL polypropylene tube and fortified with pesticides and mycotoxins at 100 ng/g. A mix of internal standards was added at 100 ng/g. 5 mL of acetonitrile acidified with 1% acetic acid was added to the sample followed by a 5 min vortex extraction at 2500 rpm. 200 µL of water was added to a 6 mL hydrophilic lipophilic balanced (HLB) cartridge (200 mg) (Restek cat.# 28451). Then, 3 mL of hemp extract was transferred to the cartridge. Vacuum was applied to collect the cleaned extract. After collecting all the sample, the vacuum was stopped, and 300 µL of methanol was added to help elute all the target analytes (vacuum was reapplied and the rinsing solvent was collected with the rest of the extract). 600 µL of extract was mixed with 400 µL of 2:2:1 methanol:acetonitrile(1% acetic acid):water. 1.5 µL of final extract was injected into the LC-MS/MS system.
Notes	Autosampler temperature: 10 °C Want even better performance when analyzing metal-sensitive compounds? Check out Inert LC columns at www.restek.com/inert.