HALO®

ENVIRONMENTAL

LC-MS Analysis of the Oregon Pesticide Mix on HALO® AQ-C18

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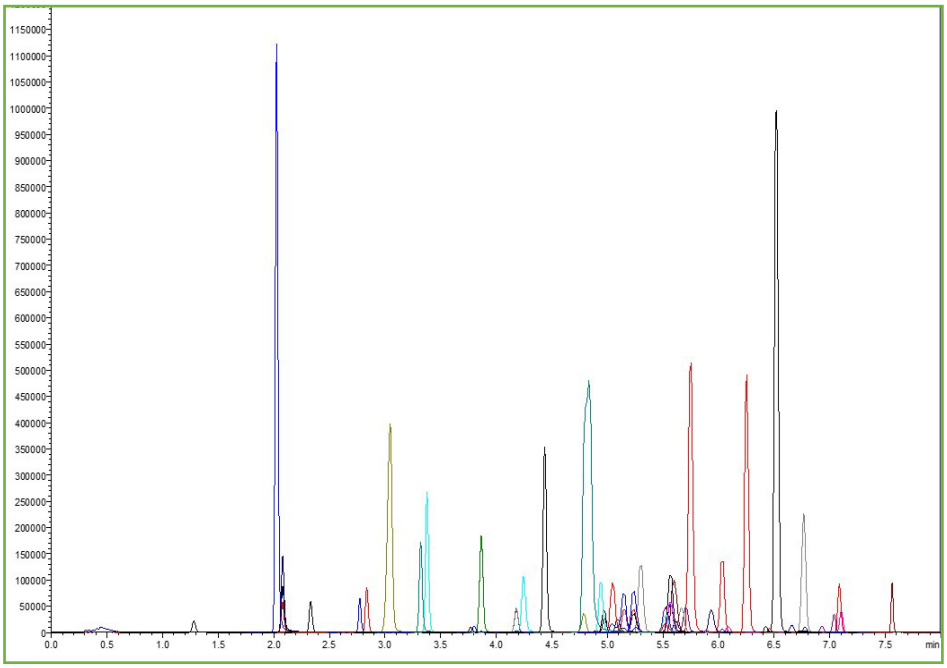

In the U.S., each state maintains its own list of pesticides that must be screened during cannabis testing. The Oregon pesticide list consists of 59 compounds and 2 of these are isomers (spinosad A and D and pyrethrin I and II). All of the compounds were run using positive ESI except fludioxinil and fipronil, which were run in negative mode since it is easier for these compounds to be oxidized. Since the list of pesticides has a broad range of polarities, it is useful to run the gradient starting at 0% organic. This means that a 100% aqueous compatible phase is needed and is why HALO® AQ-C18 was used for the analysis. The entire analysis including re-equilibration is 12 minutes, which highlights the speed of Fused-Core® analysis.

TEST CONDITIONS:

Column: HALO 90 Å AQ-C18 , 2.7 µm, 2.1 x 50 mm
Part Number: 92812-422
Mobile Phase A: Water, 5 mM Ammonium Formate & 0.1% Formic Acid
Mobile Phase B: Methanol, 5 mM Ammonium Formate & 0.1% Formic Acid
Gradient:

Time	%B
0.0	0
7.0	100
8.0	100
8.5	0
12.0	0

Flow Rate: 0.5 mL/min
Pressure: 200 bar
Temperature: 35 °C
Injection Volume: 1 µL
Sample: Oregon Pesticide Mixture 10x Action Limit 2-20 µg/mL in ACN from LGC (DRE-GA09000244AL) diluted 30 µL in 120 µL of Mobile Phase A
Sample Solvent: 80/20 Mobile Phase A/ACN
LC System: Shimadzu Nexera X2

MS CONDITIONS:

System: Shimadzu 8040
ESI + and -
Spray Voltage: 4.5 kV
Nebulizer Gas Flow: 2 L/min
DL Temperature: 250 °C
Heat Block Temperature: 400 °C
Drying Gas Flow: 15 L/min

Tubing Optimization:

Column outlet to Ground:
AMT MarvelXACT™ PEEKsil™ 75 µm ID x 600 mm
Part Number: PS7075600
Ground to Source:
AMT MarvelXACT™ PEEKsil™ 75 µm ID x 150 mm
Part Number: PS7075150

LGC Standards

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Peak #	Compound	m/z transition	Retention Time (min)
1	Daminozide	161.1000>61.1000	0.47
2	Acephate	184.1000>143.1000	1.28
3	Oxamyl	237.1000>72.1000	2.03
4	Flonicamid	230.1000>203.1000	2.08
5	Methomyl	163.1000>88.1000	2.12
6	Thiamethoxam	292.1000>132.1000	2.33
7	Imidacloprid	256.1000>175.1000	2.77
8	Dimethoate	230.1000>125.1000	2.84
9	Acetamiprid	223.1000>126.1000	3.05
10	Aldicarb	208.1000>89.1000	3.32
11	Thiacloprid	253.1000>126.1000	3.38
12	Dichlorvos	221.1000>109.1000	3.77
13	Propoxur	210.1000>111.1000	3.80
14	Carbofuran	222.1000>165.1000	3.87
15	Carbaryl	202.1000>145.1000	4.18
16	Imazalil	297.1000>159.1000	4.25
17	Metalaxyl	280.2000>220.1000	4.44
18	Naled	381.1000>127.1000	4.61
19	Parathion methyl	264.2000>232.1000	4.68
20	Chlorantraniliprole	481.9000>283.9000	4.79
21	Spiroxamine	298.1000>144.1000	4.83
22	Phosmet	318.1000>160.1000	4.94
23	Methiocarb	226.1000>121.1000	4.97
24	Azoxystrobin	404.1000>344.1000	4.97
25	Paclobutrazol	294.1000>70.2000	5.05
26	Boscalid	343.1000>307.1000	5.09
27	Fludioxinil	246.6000>125.9000	5.14
28	Malathion	331.1000>99.1000	5.15
29	Myclobutanil	289.1000>69.9000	5.15
30	Spirotetramat	374.1000>330.1000	5.24

Peak #	Compound	m/z transition	Retention Time (min)
31	Ethoprophos	243.1000>130.9000	5.26
32	Bifenazate	301.1000>170.2000	5.30
33	Pyrethrin I	329.1000>161.1000	5.47
34	Fipronil	434.7000>330.3000	5.52
35	Fenoxycarb	302.1000>116.1000	5.57
36	Tebuconazole	308.2000>70.1000	5.60
37	Kresoxim-methyl	314.1000>116.1000	5.62
38	Propiconazole	342.1000>69.1000	5.67
39	Diazinon	305.1000>169.1000	5.71
40	Spinosad A	732.6000>142.1000	5.75
41	MGK 264	276.1000>210.1000	5.89
42	Prallethrin	301.2000>133.1000	5.91
43	Spinosad D	746.5000>142.1000	5.93
44	Pyrethrin II	373.2000>161.1000	6.02
45	Chlorfenapyr	409.2000>59.0000	6.03
46	Trifloxystrobin	409.1000>186.1000	6.03
47	Clofentezine	303.1000>138.1000	6.09
48	Piperonyl butoxide	356.2000>177.1000	6.25
49	Hexythiazox	353.1000>228.1000	6.43
50	Chlorpyrifos	351.9000>124.9000	6.45
51	Spiromesifen	388.2000>273.1000	6.46
52	Etoxazole	360.2000>141.1000	6.52
53	Fenpyroximate	422.2000>366.1000	6.66
54	Cyfluthrin	451.1000>191.1000	6.71
55	Cypermethrin	433.1000>191.0000	6.74
56	Pyridaben	365.2000>147.1000	6.77
57	Abamectin	890.7000>305.3000	6.78
58	Permethrin	408.1000>183.1000	7.04
59	Etofenprox	394.3000>177.1000	7.09
60	Bifenthrin	440.1000>166.2000	7.10
61	Acequinocyl	343.2000>189.1000	7.56