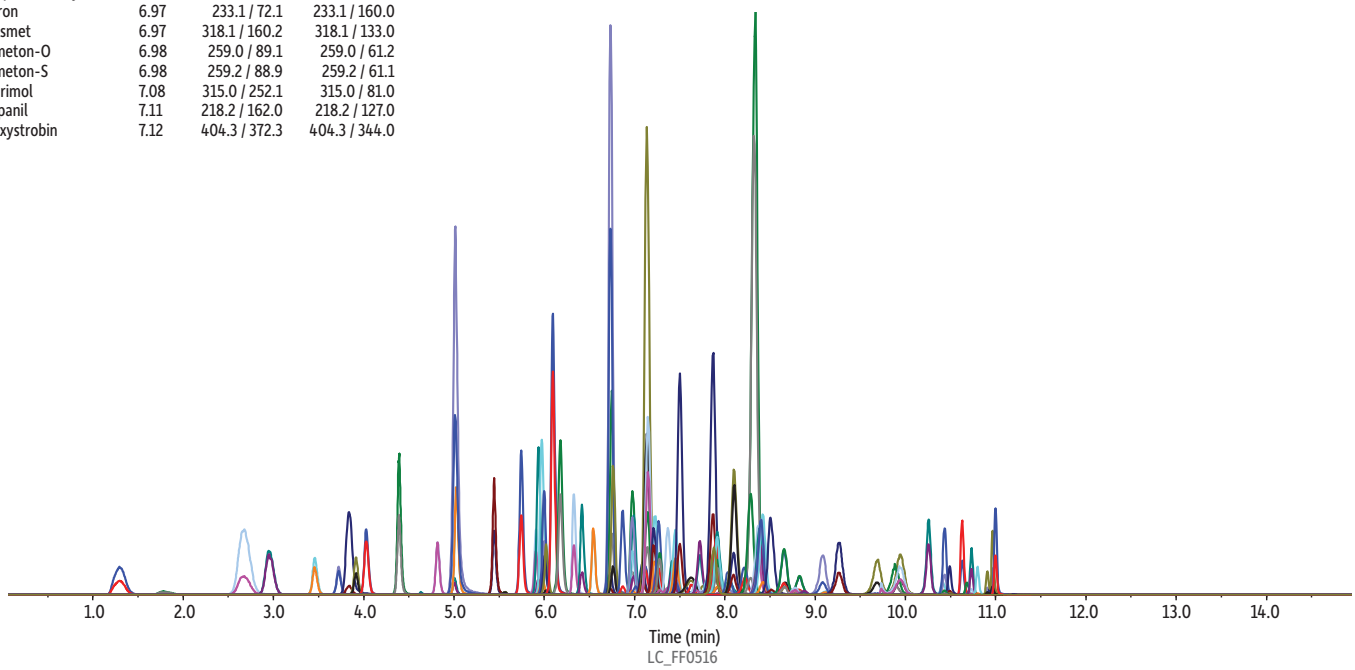
Pesticide Residue Analysis in Kale by LC-MS/MS on Ultra Aqueous C18

Peaks	t_R (min)	MRM 1	MRM 2
1. Methamidophos	1.27	142.1 / 94.1	142.1 / 112.2
2. Acephate	1.78	184.1 / 125.1	184.1 / 95.1
3. Propamocarb	2.65	189.2 / 102.0	189.2 / 144.2
4. Omethoate	2.94	214.1 / 125.2	214.1 / 155.1
5. Aldicarb sulfone	3.45	223.2 / 148.0	223.2 / 76.2
6. Aldicarb sulfoxide	3.71	207.2 / 132.1	207.2 / 89.1
7. Pymetrozine	3.82	218.1 / 105.0	218.1 / 78.2
8. Oxamyl	3.90	237.1 / 71.9	237.1 / 90.1
9. Methomyl	4.02	163.1 / 88.1	163.1 / 106.2
10. Monocrotophos	4.38	224.1 / 127.1	224.1 / 98.1
11. Dimethoate	4.81	230.1 / 125.2	230.1 / 171.2
12. Mevinphos E	4.99	225.2 / 193.3	225.2 / 127.2
13. Thiabendazole	4.99	202.2 / 175.0	202.2 / 131.2
14. Imidacloprid	5.02	256.3 / 209.1	256.3 / 175.2
15. Mevinphos Z	5.43	225.1 / 193.2	225.1 / 127.1
16. Aldicarb	5.55	208.2 / 116.2	208.2 / 89.1
17. Carbetamide	5.73	237.1 / 192.0	237.1 / 118.1
18. Imazethapyr	5.90	290.1 / 245.2	290.1 / 177.3
19. Thidiazuron	5.93	221.2 / 102.0	221.2 / 128.1
20. Thiophanate methyl	5.96	343.2 / 151.1	343.2 / 93.1
21. Propoxur	5.99	210.2 / 168.1	210.2 / 111.2
22. Bendiocarb	6.00	224.1 / 109.2	224.1 / 167.2
23. Dichlorvos	6.01	220.9 / 109.2	220.9 / 95.0
24. Carbofuran	6.09	222.3 / 165.2	222.3 / 123.1
25. Pirimicarb	6.15	239.2 / 72.2	239.2 / 182.2
26. Carbaryl	6.32	202.3 / 127.1	202.3 / 117.2
27. Imazalil	6.40	297.1 / 159.0	297.1 / 173.1
28. Isoprocarb	6.53	194.3 / 95.2	194.3 / 137.3
29. Metalaxyl	6.72	280.4 / 192.3	280.4 / 160.2
30. Metalaxyl-m	6.72	280.4 / 220.3	280.4 / 192.1
31. Atrazine	6.75	216.2 / 174.3	216.2 / 132.1
32. Atrazine-d5 (IS)	6.77	221.1 / 179.0	221.1 / 101.2
33. Isoproturon	6.86	207.2 / 72.3	207.2 / 134.3
34. Azinphos-methyl	6.96	318.2 / 160.0	318.2 / 132.1
35. Diuron	6.97	233.1 / 72.1	233.1 / 160.0
36. Phosmet	6.97	318.1 / 160.2	318.1 / 133.0
37. Demeton-O	6.98	259.0 / 89.1	259.0 / 61.2
38. Demeton-S	6.98	259.2 / 88.9	259.2 / 61.1
39. Nuarimol	7.08	315.0 / 252.1	315.0 / 81.0
40. Propanil	7.11	218.2 / 162.0	218.2 / 127.0
41. Azoxystrobin	7.12	404.3 / 372.3	404.3 / 344.0
42. Malathion	7.13	331.1 / 127.1	331.1 / 99.1
43. Methiocarb	7.14	226.1 / 169.1	226.1 / 121.1
44. Chlorpropham	7.20	214.1 / 154.0	214.1 / 126.1
45. Linuron	7.20	249.2 / 160.1	249.2 / 182.1
46. Crotoxyphos	7.21	332.2 / 211.2	332.2 / 167.2
47. Promecarb	7.21	208.8 / 109.2	208.8 / 151.3
48. Propetamphos	7.22	282.1 / 138.0	282.1 / 110.2
49. Boscalid	7.25	343.2 / 307.2	343.2 / 140.0
50. Triadimefon	7.26	294.3 / 197.1	294.3 / 69.2
51. Triadimenol	7.35	296.3 / 70.2	296.3 / 227.1
52. Fenhexamid	7.40	302.1 / 97.1	302.1 / 55.0
53. Myclobutanil	7.43	289.3 / 70.2	289.3 / 124.9
54. Dichlofluanid	7.44	332.9 / 224.0	332.9 / 123.1
55. Triazophos	7.48	314.1 / 162.0	314.1 / 119.2
56. Alachlor	7.60	270.2 / 238.1	270.2 / 162.2
57. Fenarimol	7.61	331.0 / 268.0	331.0 / 81.0
58. Iprodione	7.66	330.3 / 245.2	332.3 / 247.0
59. Ethoprop	7.70	243.1 / 131.0	243.1 / 173.0
60. Parathion	7.77	292.1 / 236.0	292.1 / 140.1
61. Fenamiphos	7.83	304.4 / 217.2	304.4 / 202.0
62. Diflubenzuron	7.85	311.1 / 158.2	311.1 / 141.1
63. Fenoxycarb	7.87	302.1 / 88.0	302.1 / 116.1
64. Etaconazole isomer 1	7.89	328.2 / 159.1	328.2 / 123.0
65. Fenbuconazole	7.89	337.3 / 125.3	337.3 / 70.3
66. Kresoxim-methyl	7.90	314.2 / 115.9	314.2 / 131.0
67. Tolyfluanid	8.00	364.0 / 238.0	364.0 / 137.1
68. Etaconazole isomer 2	8.01	328.3 / 159.2	328.3 / 123.1
69. Fenthion	8.06	279.1 / 169.1	279.1 / 105.1
70. Quinalphos	8.07	299.3 / 243.1	299.3 / 163.2
71. Cyprodinil	8.16	226.1 / 93.3	226.1 / 77.1
72. Tebuconazole	8.24	308.3 / 70.1	308.3 / 125.1
73. Chlorfenvinphos	8.32	359.2 / 155.1	359.2 / 99.2
74. Diazinon	8.34	305.2 / 169.3	305.2 / 153.1
75. Pirimiphos methyl	8.35	306.1 / 164.3	306.1 / 108.1
76. Phosalone	8.38	368.1 / 182.1	368.1 / 138.0
77. Diazinon-d10 (IS)	8.39	315.3 / 170.1	315.3 / 154.1
78. Coumaphos	8.46	363.1 / 227.2	363.1 / 211.1
79. Propiconazole isomer 1	8.60	342.3 / 159.0	342.3 / 69.3
80. Pyraclostrobin	8.61	388.0 / 164.2	388.0 / 194.3
81. Chlorpyrifos methyl	8.72	323.9 / 125.0	323.9 / 291.8
82. Propiconazole isomer 2	8.77	342.4 / 159.1	342.4 / 69.4
83. Dialifos	8.81	394.3 / 208.0	394.3 / 187.0
84. Prochloraz	9.01	376.1 / 308.1	376.1 / 266.0
85. Indoxacarb	9.04	528.6 / 218.0	528.6 / 150.2
86. Trifloxystrobin	9.18	409.4 / 186.0	409.4 / 145.1
87. Spinosyn A	9.58	733.1 / 142.4	733.1 / 98.4
88. Difenoconazole isomer 1	9.60	406.3 / 251.1	408.2 / 253.1
89. Triflumizole	9.79	346.2 / 278.2	346.2 / 73.1
90. Difenoconazole isomer 2	9.85	406.4 / 251.2	408.3 / 253.2
91. Ethion	10.20	385.3 / 199.0	385.3 / 171.0
92. Spinosyn D	10.38	746.8 / 142.4	746.8 / 98.3
93. Chlorpyrifos	10.40	350.0 / 198.0	350.0 / 97.0
94. Pendimethalin	10.47	282.3 / 212.2	282.3 / 194.3
95. Emamectin B1a benzoate	10.48	887.2 / 158.3	887.2 / 126.3
96. Propargite	10.61	368.4 / 175.1	368.4 / 231.2
97. Fenpropathrin	10.67	350.3 / 125.0	350.3 / 97.4
98. Flufenoxuron	10.72	489.5 / 158.2	489.5 / 141.1
99. Lambda cyhalothrin	10.72	467.4 / 225.1	467.4 / 181.0
100. Deltamethrin	10.78	523.3 / 280.9	523.3 / 181.0
101. *trans*-Permethrin	10.90	408.4 / 183.3	408.4 / 153.2
102. Leptophos	10.92	413.2 / 171.0	413.2 / 77.1
103. *cis*-Permethrin	10.95	408.5 / 183.1	408.5 / 153.2
104. Bifenthrin	10.98	440.3 / 181.2	440.3 / 166.2

*Isomers were designated 1 or 2 by elution order



Column	Ultra Aqueous C18 (cat.# 9178312)
Dimensions:	100 mm x 2.1 mm ID
Particle Size:	3 µm
Pore Size:	100 Å
Temp.:	50 °C
Sample	Kale extract diluted 10x in mobile phase A
Diluent:	Mobile phase A
Conc.:	10 ng/mL
Inj. Vol.:	20 µL

Mobile Phase	
A:	Water + 0.1% formic acid + 4 mM ammonium formate
B:	Methanol + 0.1% formic acid + 4 mM ammonium formate

Time (min)	Flow (mL/min)	%A	%B
0.00	0.5	90	10
1.50	0.5	90	10
6.00	0.5	30	70
9.00	0.5	30	70
10.00	0.5	0	100
12.00	0.5	0	100
12.01	0.5	90	10
15.00	0.5	90	10

Detector	ABSCIEX MS/MS
Model #:	API 4000
Ion Source:	TurboIonSpray®
Ion Mode:	ESI+
Ion Spray Voltage:	5.5 kV
Curtain Gas:	30 psi (206.8 kPa)
Gas 1:	40 psi (275.8 kPa)
Gas 2:	45 psi (310.3 kPa)
CAD:	10 psi (68.9 kPa)
Source Temp.:	450 °C
Mode:	Scheduled MRM
MRM Detection Window:	45 sec
Target Scan Time:	0.33 sec
Instrument	API LC-MS/MS

Notes The AOAC QuEChERS method was used. 15 mL of acetonitrile with 1% acetic acid (v/v) was added to 15 g of fortified homogenized kale. Q-sep™ AOAC buffering extraction salts (cat.# 26237) containing 6 g $MgSO_4$ and 1.5 g sodium acetate were added. Following 1 minute of manual shaking, samples were centrifuged for 5 minutes at 3,000 U/min with a Q-sep™ 3000 centrifuge (cat.# 26230). The top acetonitrile layer was removed to a clean vial. A Restek Q-sep™ dSPE tube, cat.# 26126, containing 300 mg PSA, 150 mg GCB, and 900 mg $MgSO_4$ was used to process 6 mL of kale extract. The tube was shaken for 2 minutes and centrifuged for 5 minutes at 3,000 U/min. The sample was diluted 10-fold in mobile phase A before injection.

Restek Corporation 110 Benner Circle Bellefonte, PA 16823
1-814-353-1300 • 1-800-356-1688 • Fax: 1-814-353-1309 • www.restek.com