Multi-Residue Pesticide Screening Method using GC/MS

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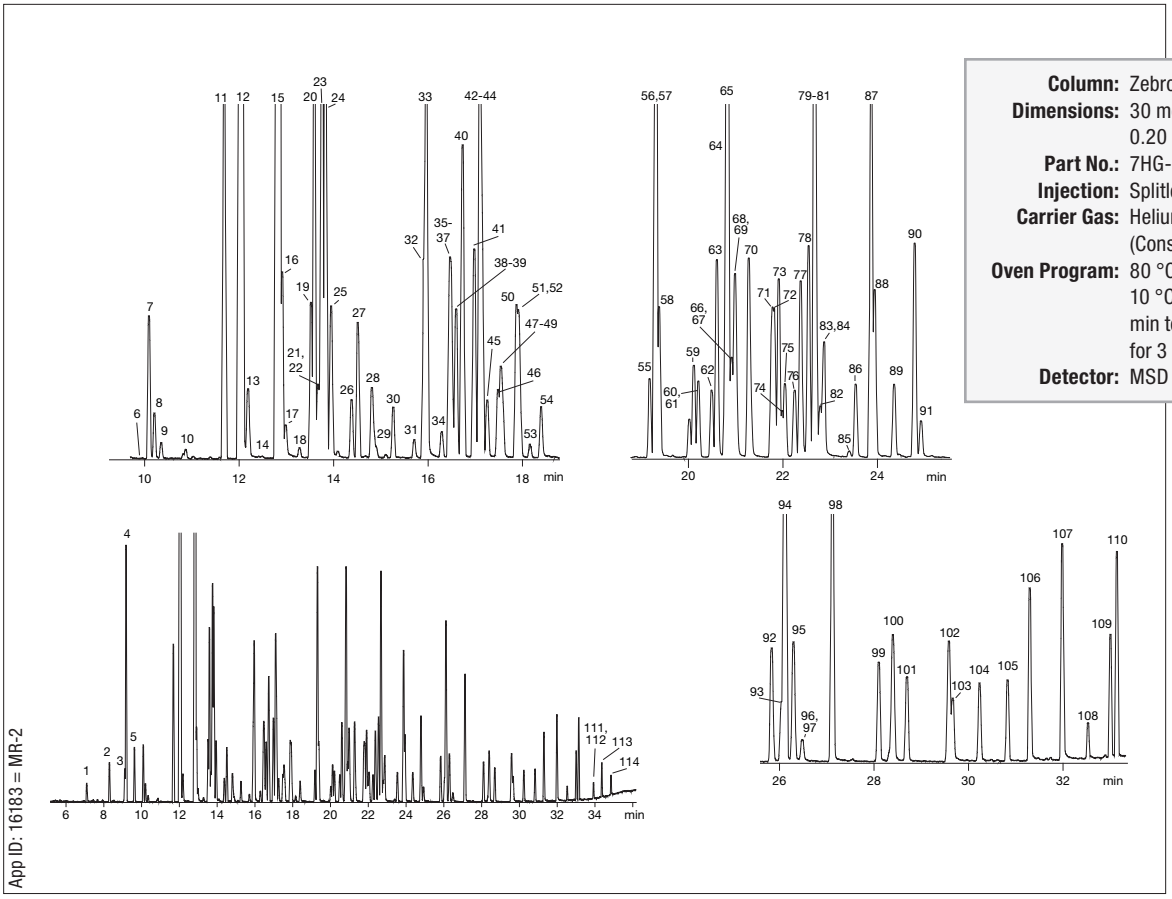
Pesticides are widely used by farmers to control pests, weeds and molds that would otherwise decrease crop production. While this has significantly increased worldwide food productions, these same pesticides pose significant health and environmental risks.

The restrictions for specific pesticides differ from one country to the next. As world trade increases, the potential threat to other countries' populations increases. This is especially true in the European Union, where produce can be transported from one country to another quite easily.

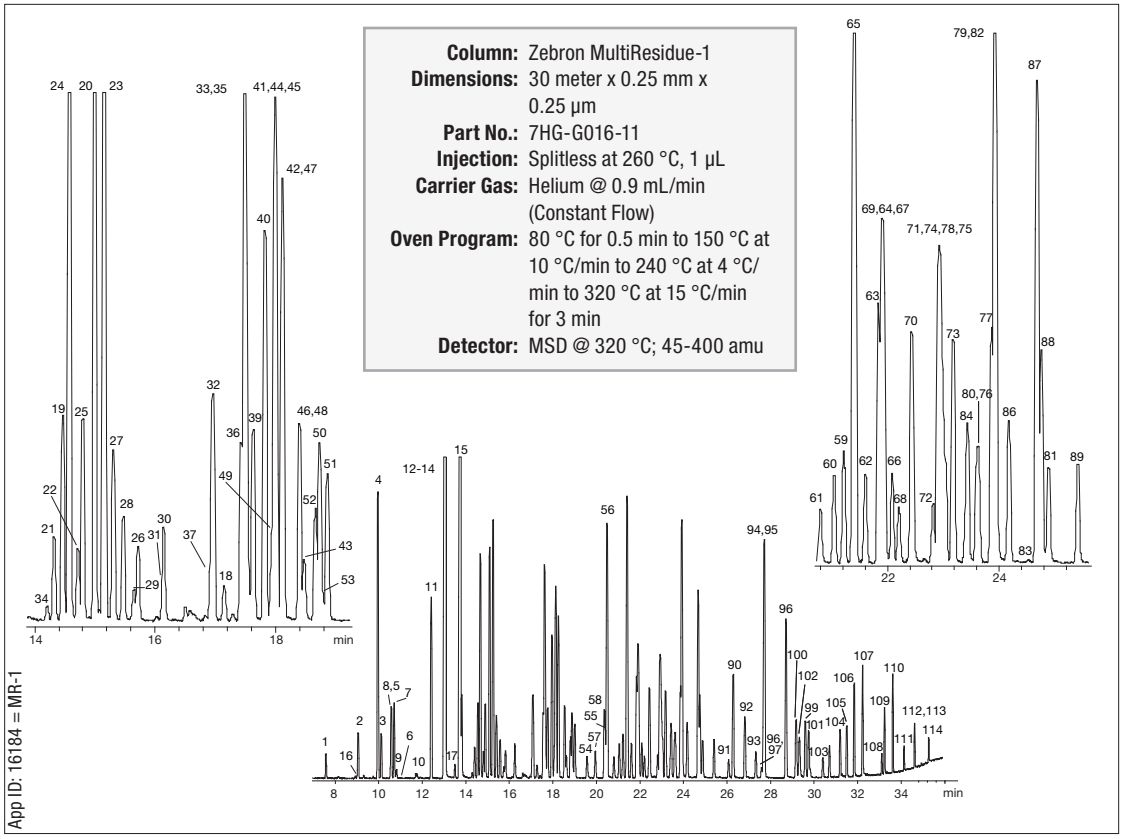
For this reason, pesticides are the subjects of increasing regulation. Since many different types of pesticides can be used on the same food product, Multi-Residue screening approaches are used to look for more than 300 compounds at a time. Gas Chromatography (GC) is still the most commonly used method for the majority of the pesticide classes. While ECD or NPD may be used for screening, Mass Spectrometer (MS) detection must be employed to provide positive confirmation.

Zebron MultiResidue™ columns represent a solution for all classes of pesticides analysis. The columns were developed using two new stationary phases that are unlike any commercially available today. Each phase was optimized to resolve a different set of analytes. However, both are good for a wide variety of pesticides.

Zebron MultiResidue columns are well suited for use on all types of detectors. Both columns are MS certified, so they can also be used with GC/MS for multi-residue pesticide methods. The unique selectivity offered by each phase allows for increased resolution of critical compounds vs. standard 5 ms type phases. Retention time data is available for over 300 different pesticides on GC/MS, further simplifying new method development.



Column:	Zebron MultiResidue-2
Dimensions:	30 meter x 0.25 mm x 0.20 µm
Part No.:	7HG-G017-10
Injection:	Splitless at 260 °C, 1 µL
Carrier Gas:	Helium @ 0.9 mL/min (Constant Flow)
Oven Program:	80 °C for 0.5 min to 150 °C at 10 °C/min to 240 °C at 4 °C/min to 320 °C at 15 °C/min for 3 min
Detector:	MSD @ 320 °C; 45-400 amu



Peak No.	Sample Analyte
1	Dichlorvos
2	EPTC
3	Butylate
4	3,5-Dichlorobenzoic acid (methyl ester)
5	Vernolate
6	Pebulate
7	Mevinphos
8	4-Nitrophenol (methyl ester)
9	Mevinphos Isomer
10	Trichlorfon
11	Dicamba (methyl ester)
12	MCPP (methyl ester)
13	Molinate
14	Tebuthiuron
15	MCPA (methyl ester)
16	DEET
17	Tetraethyl pyrophosphate (methyl ester)
18	Demeton
19	Dichloroprop (methyl ester)
20	Trifluralin
21	Thionazin
22	Cycloate
23	Benefin
24	Propachlor
25	Ethoprop
26	Chlorpropham
27	2,4-D (methyl ester)
28	Sulfotep
29	Naled
30	Phorate

Peak No.	Sample Analyte
31	Dicrotophos
32	Pentachlorophenol (methyl ester)
33	Profluralin
34	Demeton Isomer
35	Prometon
36	Atraton
37	Monocrotophos
38	Atraton Isomer
39	Silvex (methyl ester)
40	Terbufos
41	Propazine
42	Diazinon
43	Pronamide
44	Atrazine
45	Simazine
46	Terbuthylazine
47	Dioxathion
48	Fonofos
49	Dimethoate
50	2,4,5-T Methyl ester
51	Disulfoton
52	Chloramben (methyl ester)
53	Phosphamidon Isomer
54	Secbumeton
55	Dichlofenthion
56	2,4-DB (methyl ester)
57	Terbacil
58	Dinoseb (methyl ester)
59	Alachlor
60	Chlorpyrifos methyl

Peak No.	Sample Analyte
61	Phosphamidon
62	Ronnel
63	Prometryn
64	Ametryn
65	Bentazon (methyl ester)
66	Aspon
67	Simetryn
68	Metribuzin
69	Methyl parathion
70	Terbutryn
71	Metolachlor
72	Malathion
73	DCPA
74	Fenitrothion
75	Chlorpyrifos
76	Trichloronate
77	Triadimeton
78	Pichloram (methyl
79	Isopropalin
80	Fenthion
81	MGK-264 Isomer
82	Parathion
83	Merphos
84	Bromacil
85	Clofenvinfos Isomer
86	MGK-624
87	Pendimethalin
88	Diphenamid
89	Clofenvinfos
90	Butachlor

Peak No.	Sample Analyte
91	Crotoxyphos
92	Stirofos
93	Tokuthion
94	Oxadiazon
95	Merphos Oxide
96	Napropamide
97	Fenamiphos
98	Oxyflurofen
99	Acifluorfen
100	Carboxin
101	Ethion
102	Tricyclazole
103	Fensulfothion
104	Carbofenotion
105	Famfur
106	Norflurazon
107	Hexazinone
108	EPN
109	Phosmet
110	Leptophos
111	Azinphos-Methyl
112	Fenarimol
113	Azinphos-ethyl
114	Coumaphos

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Retention times for additional pesticides using Zebron MultiResidue-1

Column: Zebron MultiResidue-1
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HG-G016-11
Injection: Splitless at 280 °C, 1 µL
Carrier Gas: Constant Flow Helium 1.2 mL/min
Oven Program: 60 °C to 130 °C at 10 °C/min to 230 °C at 4 °C/min to 300 °C at 10 °C/min
Detector: MSD; 300 °C

Retention Time	Compound
6.34	Metaldehyde
7.14	Phenmedipham (frag-1)
7.81	Monolinuron IP (frag)
8.28	Propoxur - C2H3NO
8.61	Ethiolate
9.07	Isoproturon (frag)
10.21	Bendiocarb - C2H3NO
10.70	Carbofuran - C2H3NO
10.85	Linuron IP (frag)
11.43	EPTC/Eptam
11.85	Dichlobenil
12.52	Biphenyl
12.78	Butylate
13.36	Vernolate
13.49	Dioxacarb - C2H3NO
13.73	Pebulate
13.88	Etridiazole
14.00	3,4-Dichloroaniline
14.36	Propham
14.68	Metolcarb
15.30	Folpet IP (frag)
15.44	Chloroneb
15.71	Pentachlorobenzene
15.91	o-Phenylphenol
15.92	Captan IP frag (THPI)
15.94	THPI
15.95	Carbaryl - C2H3NO
16.14	Molinate
16.24	Tebuthiuron
16.38	Isoprocarb
17.80	Tecnazene
18.08	Fenobucarb
18.14	Propachlor
18.31	Cycloate/Ro-Neet
18.40	Propoxur - C2H3NO
18.50	Ethafluralin
18.53	2,3,5,6-Tetrachloroaniline
18.82	Trifluralin
18.86	Diphenylamine
18.99	Benfluralin
19.27	Chlordimeform
19.50	CIPC
19.57	2,3,5-Trimethacarb
19.63	Tebutam
19.67	Diallate-1
19.83	Penmediapham (frag-2)
20.11	Bendiocarb - C2H3NO
20.16	MBTZ
20.19	Diallate-2
20.31	Promecarb
20.36	Alpha-BHC
20.55	2,6-DIPN
20.66	Hexachlorobenzene
21.30	Ethoxyquin
21.56	Prometon
21.58	Profluralin
21.65	3,4,5-Trimethacarb
21.68	Bufencarb-1
21.77	Desmedipham
21.79	Dichloran
21.86	Command/Clomazone
21.97	Carbofuran
21.98	Simazine
22.01	Monolinuron
22.02	Atrazine
22.02	Terbumeton
22.23	Quintozene
22.38	gamma-BHC (Lindane)
22.45	Cycluron
22.46	Terbuthylazine
22.47	Aminocarb
22.63	Fluchloralin
22.74	Pronamide
22.76	Tefluthrin
22.84	Isocarbamid
22.98	Triallate
23.11	Dinitramine
23.19	Pyrimethanil
23.19	Pentachlorobenzonitrile
23.25	Bufencarb-2
23.58	Secbumeton
23.59	Mexacarbate
23.93	Pirimacarb
23.99	beta-BHC
23.99	Fenfuram
24.19	Octhilinone
24.22	beta-Spiroxamine
24.48	Pentachloroaniline
24.83	Ethiofencarb
24.84	Dimethachlor
24.98	delta-BHC
25.08	Desmetryn
25.23	Heptachlor
25.29	Vinclozolin
25.51	Dioxacarb - C2H3NO
25.55	Chlorothalonil
25.59	α-Spiroxamine
25.66	Propanil
25.73	Cymiazole
25.91	Metalaxyl
25.94	Prometryn
25.98	Metribuzen
25.98	Ametryn
25.98	Simetryn
26.20	Kresoxim-Methyl
26.35	Carbaryl - C2H3NO
26.37	3-OH Carbofuran
26.55	Terbutryn
26.57	Fuberidazole
26.70	Pentachlorothioanisole
26.84	Aldrin
26.86	Ethofumesate
26.95	Fenpropimorph
26.95	Dichlofluanid
27.04	Metolachlor
27.05	Methiocarb
27.14	Dursban
27.15	Norea
27.25	Dacthal/DCPA
27.49	Naphthalene Acetamide
27.66	Butralin
27.74	Diethofencarb
27.97	Triadimefon
28.09	Isopropalin
28.10	Nitrothal isopropyl
28.28	MGK-264 (peak-1)
28.39	Methfuroxan
28.44	Tetraconazole
28.50	p,p'-Dicofol (frag)
28.51	Cyanazine
28.81	Pendimethalin

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Retention Time	Compound
28.86	Diphenamid
28.89	Desmethyl diphenamid
28.91	Oxychlordane
29.01	MGK-264 (peak-2)
29.20	Heptachlor Epoxide
29.24	Cyprodinil
29.25	Pyracarbolid
29.34 / 29.4	Allethrin
29.48	Tolyfluanid
29.60	Pyrifenox-1
29.64	Penaconazole
29.85	Fipronil
30.00	Furalaxyl
30.06	Triflumizole
30.18	Procymidone
30.33	Triadimenol (2 Peaks)
30.51	trans-Chlordane
30.55	Aniten/Flurecol Butyl E.
30.60	Captan IP frag (THPI)
30.81	Pyrifenox-2
30.82	Folpet
30.86	cis-Chlordane
30.88	Thiabendazole
30.97	Endosulfan-I
30.98	trans-Nonachlor
31.62	Napropamide
31.94	Oxydiazon
32.02	Hexaconazole
32.04	Isoprothiolane
32.10	Flutolanil
32.18	Fludioxinil
32.24	p,p'-DDE
32.25	Imazalil
32.36	Pyrethrin-1
32.39	Dieldrin
32.42	Buprofezin
32.49	Bupirimate
33.13	Methoprotryne
33.20	Flusilazole
33.30	Carboxin
33.36	Myclobutanil
33.36	Tricyclazole
33.39	Endrin
33.85	Cyproconazole
33.88	Chlorobenzilate
34.01	Nitrofen
34.06	o,p'-DDT
34.10	cis-Nonachlor
34.20	Diniconazole
34.32	Etaconazole-1
34.49	Etaconazole-2

Retention Time	Compound
34.51	Endosulfan-II
34.62	Pyrethrin-2
34.81	Oxadixyl
35.05	Benalaxyl
35.11	Mepronil
35.14	Trifloxystrobin
35.53	Propiconizole/Tilt-1
35.55	Quinoxyfen
35.68	p,p'-DDT
35.73	o,p'-Methoxychlor
35.86	Propiconizole/Tilt-2
35.92 / 35.97	Propargite
35.95	Lenacil
36.00	Endosulfan Sulfate
36.04	Piperonyl Butoxide
36.09	Resmethrin
36.17	Nuarimol
36.27	Hexazinone
36.27	p,p'-Methoxychlor
36.30	Carbosulfan
36.34	Tebuconazole
36.44	Nitralin
36.45	Sethoxydim
36.65	Bifenthrin
36.70	Epoxiconazole
36.91	Tetramethrin-1
37.05	Bromopropylate
37.13	Fenpropathrin
37.16	Tetramethrin-2
37.39	Iprodione
37.40	Tebufenpyrad
37.45	Fenoxycarb
37.47	Bifenazate
37.48	Fenhexamid
37.55	Fenazaquin
37.80	d-Phenothrin
37.93	Acetamiprid
38.01	Acrinathrin
38.08	Tetradifon
38.22	Pyriproxifen
38.24	lambda-Cyhalothrin
38.34	Triticonazole
38.37	Mirex
38.42	Amitraz
38.66	Tralkoxydim
38.93	Fenarimol
38.99	Naproanalid
39.32	cin-Permethrin
39.54	trans-Permethrin
39.56	Biteranol-1
39.72	Biteranol-2

Retention Time	Compound
39.79	Pyridaben
39.97	Prochloraz
40.06	Fluquinconazole
40.68	Cpermethrins (4 peaks)
40.88	Etofenprox
40.91	Fenbuconazole
41.07	Flucythrinate (2 peaks)
41.41	Boscalid
42.47	Fluvalinate (2 peaks)
42.50	Fenvalerate-1
42.94	Fenvalerate-2
43.30	Pyraclostrobin
43.79	Decamethrin (2 peaks)
43.87	Azoxystrobin
44.19	Difenoconazole (2 peaks)
44.63	Famoxadone
45.84	Dimethomorph (2 peaks)

We would like to thank **Greg Mercer** at the **US Food and Drug Administration** for supplying the retention time data for the pesticides.

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