High Productivity Analysis for Pesticide Residues in Vegetables

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Introduction

Modern day pesticide residue GC/MS analysis is faced with a number of challenges, including low and decreasing regulatory levels, a wide range of compounds in complex food matrices and increasing pressure to deliver results quickly. One option to address several of these challenges is the use of 0.15 mm small diameter capillary GC columns. Their main benefit is a significant reduction in analysis time, increasing instrument productivity by as much as 50 % in some cases. Fast compound elution on these columns also lowers detection limits and improves signal to noise ratios.

The pesticide residue GC/MS method described here allows the fast, successful separation and quantification of 200 pesticides using a short 20 m x 0.15 mm ID FactorFour™ VF-5ms column, see Figure 1.

Conditions

GC: Varian CP-3800/450-GC
Column: VF-5ms, 20 m x 0.15 mm, df = 0.15 µm (pn: CP9036)
Sample: 1 µL hexane standard, 0.5-1 mg/kg per analyte
Oven: 50 °C (2 min), 45 °C/min to 200 °C, (5min), 20 °C/min to 260 °C (2 min), 15 °C/min to 280 °C (5 min), 10 °C/min to 300 °C
Carrier Gas: Helium, constant flow 1.0 mL/min
Injector Type: PTV, Model 1079, 2 mm splitless liner
Injector Temperature: 90 °C (0.20 min), 200 °C/min to 280 °C/min
Injection Speed: 10 µL/sec
Detection: Varian 4000/240-MS Ion Trap

Time	State	Split Ratio
Initial	On	5
0.00	Off	Off
2.00	On	50

Peak	Compound	RT	Quant. Ion
1	DPA	6.05	169.0
2	Pencycuron	6.32	125.1
3	Dimethoate	6.54	125.0
4	Quintozene	6.77	237.0
5	Pirimicarb	7.18	166.1
6	Vinchlozoline	7.67	212.2
7	Pyrimiphos methyl	8.09	290.1
8	Malathion	8.34	173.0
9	Fenthion	8.68	278.1
10	Pyrimiphos ethyl	9.06	318.2
11	Bromophos methyl	9.17	331.0
12	Penconazole	9.72	248.2
13	Chlorfenvinphos	9.78	267.2
14	Methidathion	10.54	145.0
15	Endosulfan A	10.91	195.2 + 241.0
16	Imazalil	11.27	173.0 + 215.0
17	Myclobutanil	11.57	179.1
18	Kresoxym methyl	11.64	116.1
19	Endosulfan B	12.18	195.1
20	Diethion	12.29	231.1
21	Endosulfan S	12.82	195.1
22	Trifloxystrobine	12.73	116.0
23	Dicofol	13.81	139.2
24	Propargite	13.11	135
25	Iprodione	13.49	314.1
26	Bifenthrine	13.56	181.1
27	Fenpropathrin	13.74	181
28	Phosalone	14.19	182.1
29	L-cyhalothrine	14.50	181.1
30	Azinphos methyl	14.92	132.1
31	Cyfluthrine 1 - 4	16.08 - 16.30	163.1
32	Cypermethrine 1 - 4	16.43 - 16.64	181.1
33	Deltamethrine	18.47	181.1

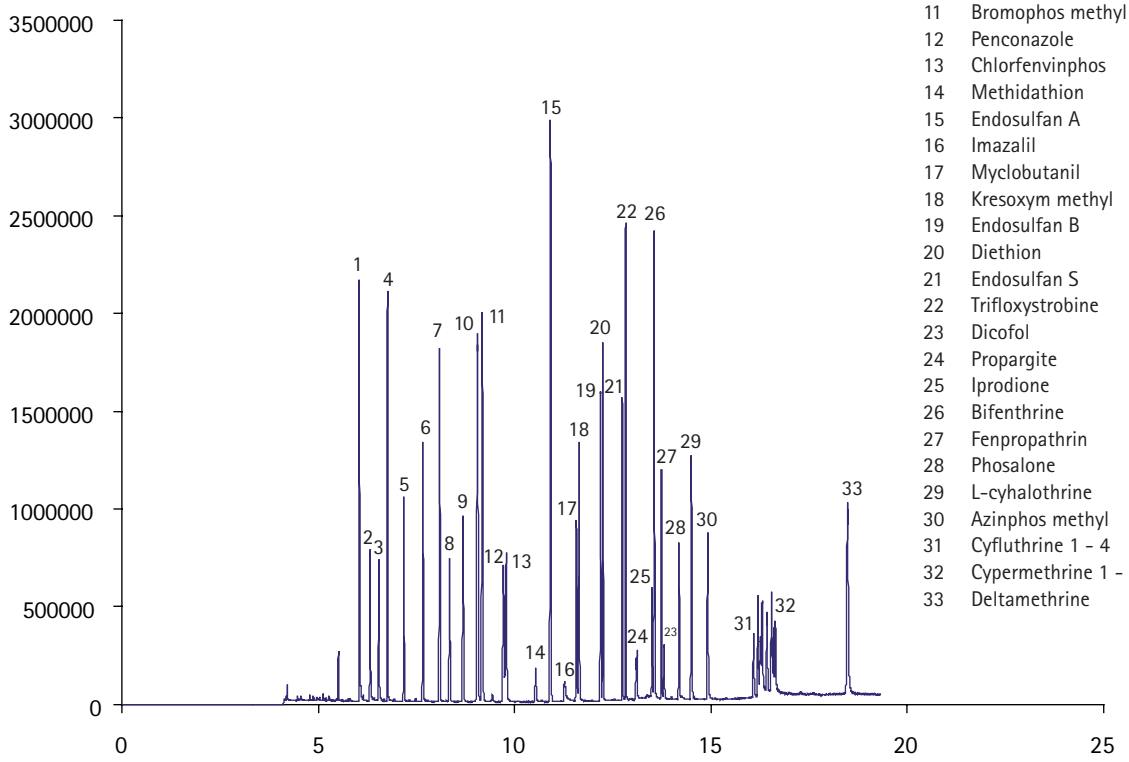

Figure 1. Chromatogram showing fast analysis using a 0.15 mm small diameter capillary GC column.

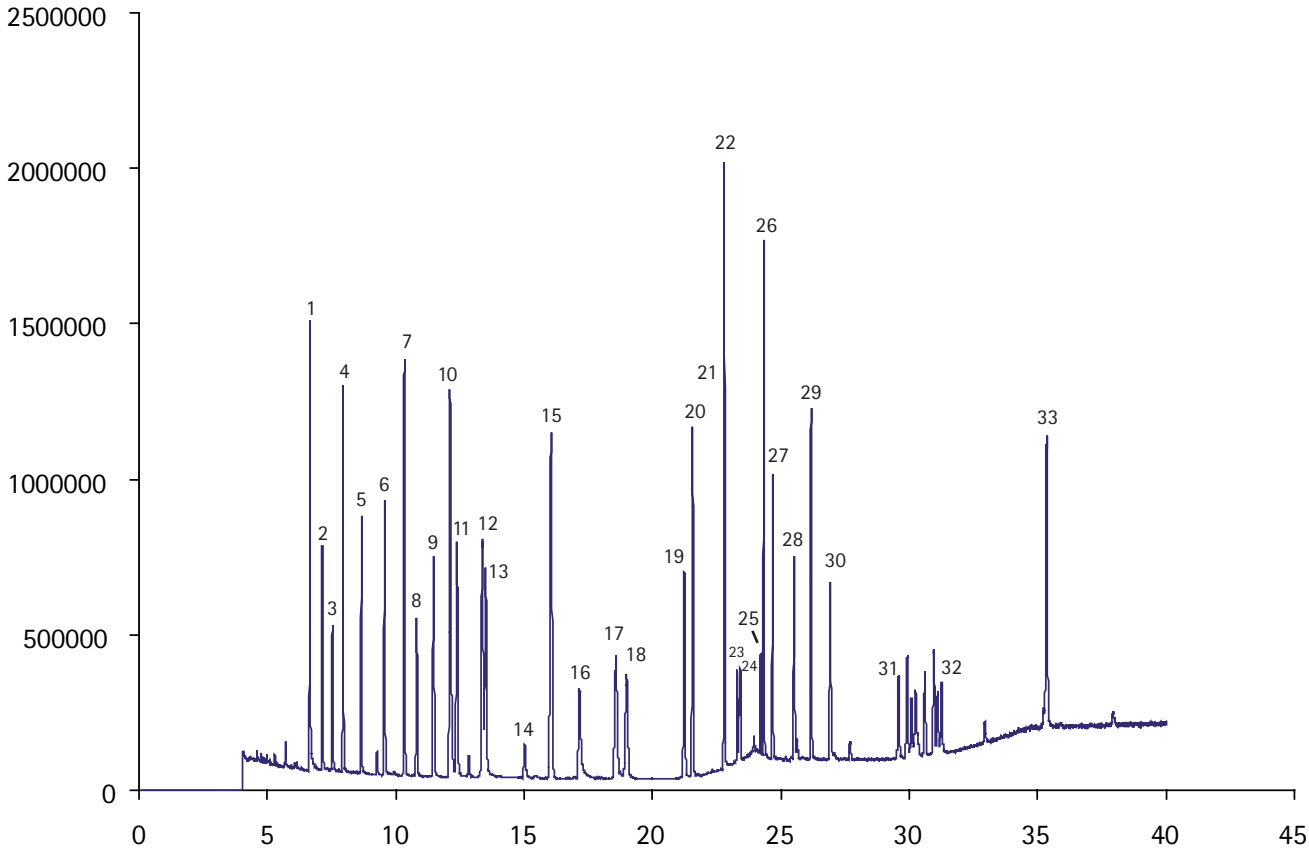

Figure 2. Chromatogram showing the original analysis time using a 0.25 mm ID column, 2 µL injection volume and 1-2 mg/kg per analyte.

The original analysis time of 35 min on the 30 m x 0.25 mm ID column (see Figure 2) is reduced to less than 20 min while resolution and quality of separation is fully maintained. Due to the fast elution on the 0.15 mm ID column, overall sensitivity has increased four fold. This means that smaller injection volumes can be used to reach identical or even better levels of detection. Reduced injection volumes will also result in less injector maintenance.

Many pesticides are thermally labile and tend to degrade in GC hot zones such as the injector, affecting accuracy of quantification and detection levels. A temperature programmable splitless injection can be applied using the Varian 1079 Programmable Temperature Vaporizing Injector (PTV), which significantly reduces this thermal degradation process for compounds such as iprodione or λ-cyhalothrin.

These data represent typical results.
For further information, contact your local Varian Sales Office.

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