Multi-residue Analysis for PAHs, PCBs and OCPs on Agilent J&W FactorFour VF-Xms

Application Note

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Introduction

In multi-residue analysis different groups of compounds are separated in a single operation, saving time and increasing productivity. This application note describes a multi-residue method for the assessment of polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs) and organochlorine pesticides (OCPs), using GC/MS with a VF-Xms column.

PAHs, PCBs and OCPs have different origins and belong to different chemical classes. PAHs are formed during incomplete combustion or pyrolysis of organic matter. They contain two or more aromatic rings. PCBs are a group of chemicals containing two phenyl groups but with varying numbers of chlorine atoms. PCBs are used in many industrial and commercial applications because of their non-flammability, chemical stability, high boiling point and electrical insulating properties. OCPs mainly consist of carbon, hydrogen and chlorine. Most persistent OCPs break down slowly and can remain in the environment long after application, and bio-accumulate in living organisms.

16 PAHs, 17 PCBs and 24 OCPs were analyzed in a single operation at different concentrations. The concentration of the PAHs was ten times higher than the concentration of PCBs and OCPs, typical for environmental samples.

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Materials and Methods

Technique: GC/MS

Column: VF-Xms, 30 m x 0.25 mm, df = 0.25 µm (part number CP8806)

Sample Conc: OCPs and PCBs 0.1 µg/mL, PAHs 1 µg/mL

Injection Volume: 1 µL

Temperature: 70 °C, 40 °C/min, 185 °C, 15 °C/min, 250 °C (1 min), 5 °C/min, 325 °C

Carrier Gas: Helium, constant flow, 1 mL/min

Injection: 70 °C (0.4 min), 600 °C/min, 300 °C (15 min), 50 °C/min, 120 °C, splitless

Detection: Quadrupole MS, EI in SIM, source 250 °C, transfer line 280 °C

Results and Discussion

The VF-Xms column provided a multi-residue analysis of PAHs, PCBs and OCPs in 21 minutes (Figure 1). Analyzing 57 compounds in a single run can be quite challenging because every group of compounds presents its own difficulties in separation.

Among the PAHs, two pairs are normally difficult to resolve; benzo[b]fluoranthene/benzo[k]fluoranthene, which have the same mass and therefore cannot be separated by MS only, and indeno[1,2,3-cd]pyrene/dibenz[a,h]anthracene. These latter compounds have different masses, 276 and 278, respectively, and are again difficult to resolve using only MS.

When analyzing PCBs, PCB 138 and PCB 163 have the same mass spectra and cannot be resolved by MS. OCPs also contain a difficult-to-resolve pair, namely cis-heptachlor epoxide/trans-heptachlor epoxide, which both have m/z 353 in their mass spectra. In SIM mode, a second m/z must be analyzed to confirm the presence of cis- or trans-heptachlor epoxide. Figures 2 to 5 show the peak pairs that can be difficult to resolve.

Table 1. Peak Identification and SIM ions

Peak	Compound	Ions
1	Naphthalene	128
2	Acenaphthene	152
3	Acenaphthylene	154
4	Fluorene	166
5	Hexachlorobenzene	284, 249
6	α-HCH	183, 219
7	β-HCH	181, 219
8	γ-HCH	183, 219
9	PCB 18	186, 256
10	Phenanthrene	178
11	Anthracene	178
12	δ-HCH	181, 219
13	PCB 28	256, 186
14	PCB 31	256, 186
15	PCB 20	256, 186
16	Heptachlor	272, 100
17	PCB 52	292, 220
18	Aldrin	66, 263
19	PCB 44	292, 220
20	trans-Heptachlor epoxide	81, 353
21	cis-Heptachlor epoxide	81, 237
22	PCB 155	360, 290
23	PCB 101	326, 254
24	o,p'-DDE	246, 318
25	Fluoranthene	202
26	trans-Chlordane	373, 237
27	cis-Chlordane	373, 237
28	Endosulfan I	237, 205
29	p,p'-DDE	246, 318
30	Pyrene	202
31	o,p'-DDD	235, 165
32	Dieldrin	79, 261
33	PCB 149	360, 290
34	PCB 118	326, 256
35	Endrin	263, 345
36	o,p'-DDT	235, 165
37	p,p'-DDD	235, 165
38	PCB 153	360, 290
39	Endosulfan II	239, 195
40	PCB 105	326, 256
41	Endrin aldehyde	245, 347
42	PCB 163	360, 290
43	PCB 138	360, 290
44	p,p'-DDT	235, 165
45	Endosulfan sulfate	272, 389
46	Methoxychlor	227
47	PCB 180	396, 322
48	Benz[a]anthracene	228
49	Chrysene	228
50	PCB 170	396, 324
51	PCB 194	430, 358
52	Benzo[b]fluoranthene	252
53	Benzo[k]fluoranthene	252
54	Benzo[a]pyrene	252
55	Indeno[1,2,3-c,d]pyrene	276
56	Dibenz[a,h]anthracene	278
57	Benzo[g,h,i]perylene	276

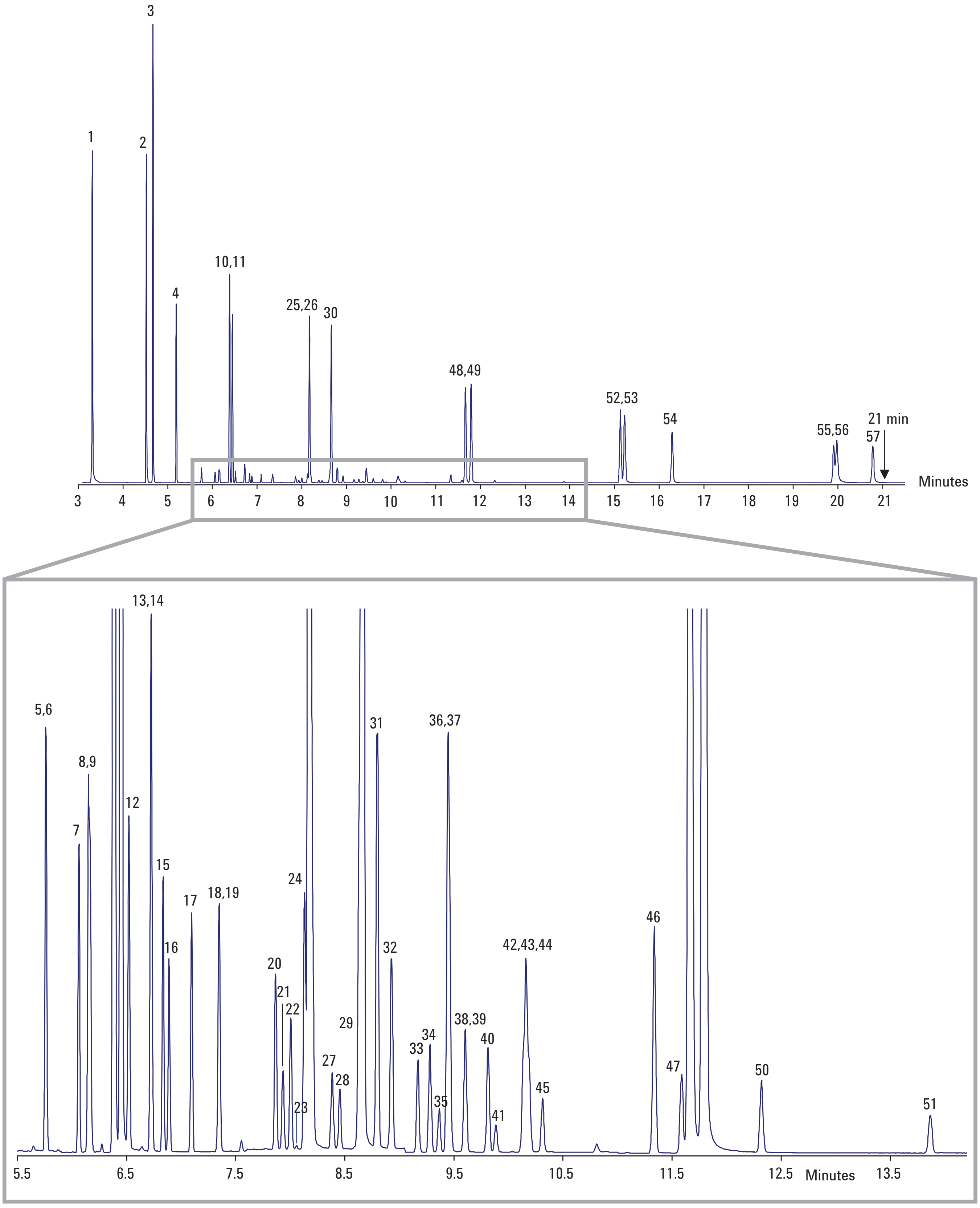

Figure 1. Total ion chromatogram multi-residue analysis using a VF-Xms column

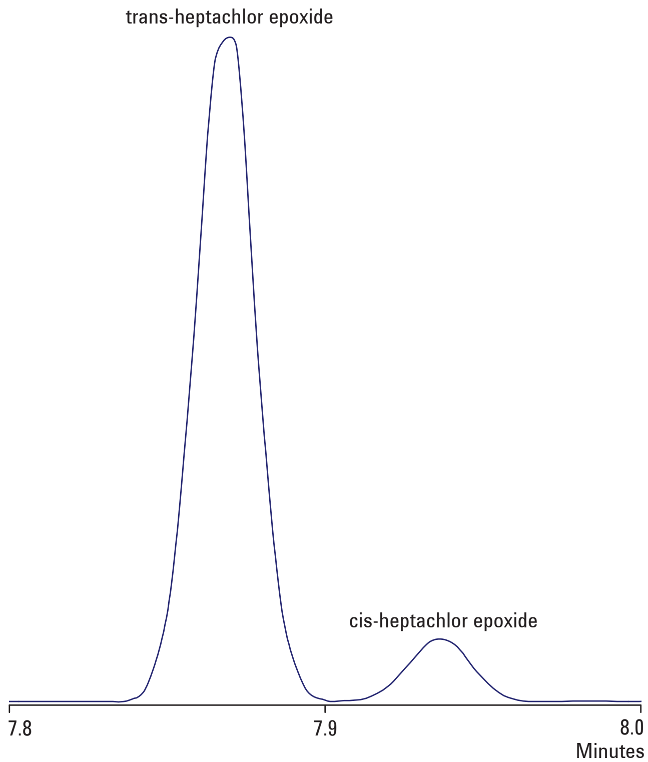

Figure 2. Selected ion chromatogram (m/z 353) of cis-heptachlor epoxide/ trans-heptachlor epoxide

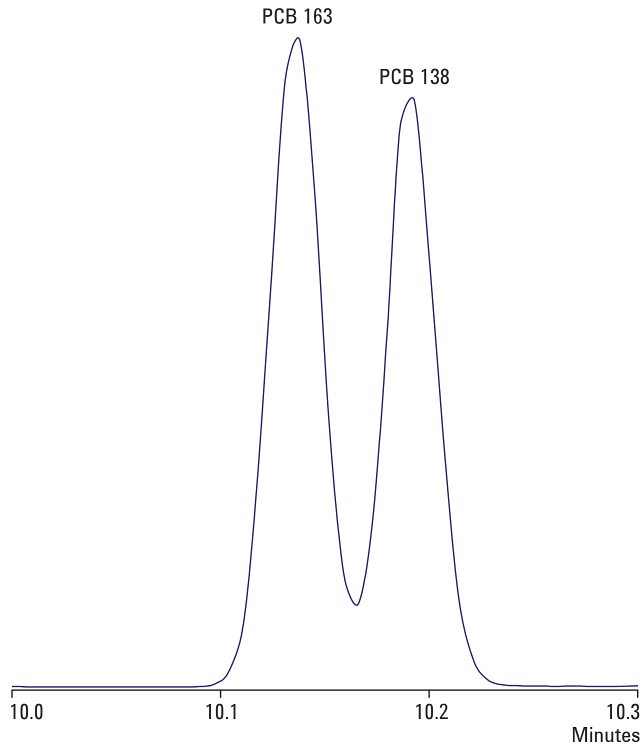

Figure 3. Selected ion chromatogram (m/z 360) of PCB 138/PCB 163

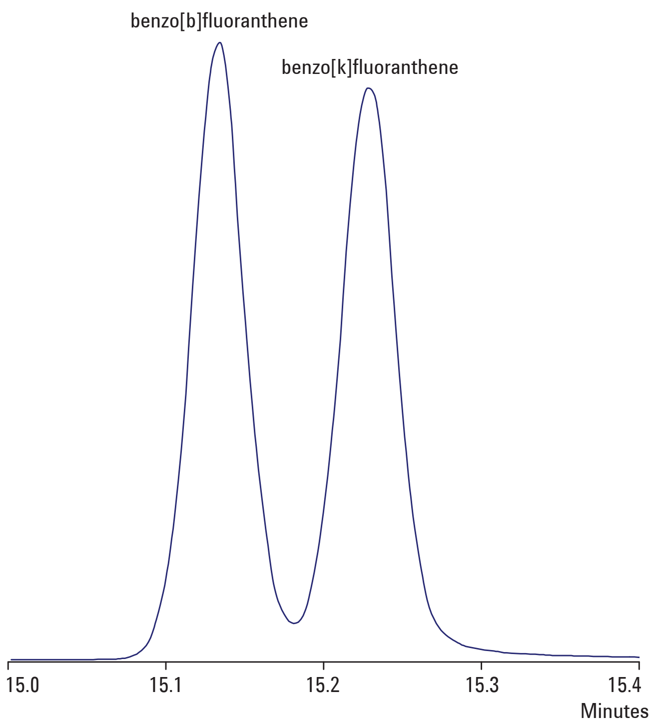

Figure 4. Zoomed total ion chromatogram of benzo[b]fluoranthene/benzo[k] fluoranthene

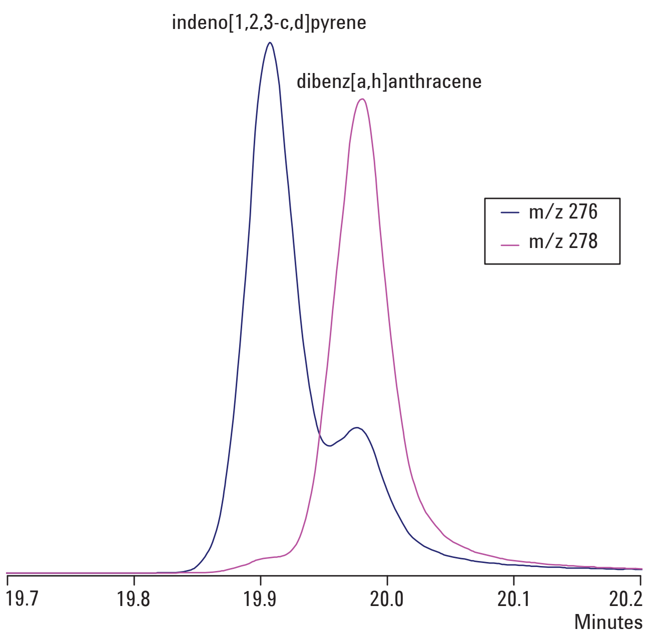

Figure 5. Selected ion chromatogram (m/z 276 and 278) of indeno[1,2,3-c,d]pyrene and dibenz[a,h]anthracene

Conclusion

A VF-Xms column and optimized oven program delivered a multi-residue analysis of 57 environmental pollutants in 21 minutes. This ultra-low bleed FactorFour column provides exceptionally high selectivity for pesticides, compared to more non-polar ‘ms’ type phases.

References

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Published in UK, October 14, 2010

SI-02443

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