Multi-residue Analysis of PAHs, PCBs and OCPs on an Agilent J&W FactorFour VF-5ms GC Column

Application Note

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Introduction

During multi-residue analyses, different groups of compounds are analyzed in a single run. Reducing time is one of the most important advantages of this approach. For environmental samples, different analyses usually have to be performed for each type of sample. To improve productivity, multi-residue analysis is often used, which is suitable for most sample types. This note describes a multi-residue method for polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs) and organochlorine pesticides (OCPs).

PAHs, PCBs and OCPs have different origins. PAHs are formed during incomplete combustion or pyrolysis of organic matter. PAHs are compounds that contain two or more aromatic rings. Due to their non-flammability, chemical stability, high boiling point, and electrical insulating properties, PCBs were used in hundreds of industrial and commercial applications, including electrical equipment, plasticizers, and pigments, amongst many others. PCBs are characterized by two phenyl groups, with varying numbers of chlorine atoms. OCPs are pesticides that primarily consist of carbon, hydrogen, and chlorine. Most OCPs break down slowly. They are typically very persistent in the environment, and are known for accumulating in sediments, plants and animals.

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

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Conditions

Technique:	GC/MS
Column:	VF-5ms, 30 m x 0.25 mm, df = 0.25 µm (Part number CP8944)
Sample Concentration:	PAHs 1 µg/mL, OCPs and PCBs 0.1 µg/mL
Injection Volume:	1 µL
Temperature:	60 °C (2 min), 20 °C/min, 175 °C, 5 °C/min, 250 °C, 10 °C/min, 325 °C/min (5 min)
Carrier Gas:	Helium, constant flow, 1 mL/min
Injector:	60 °C (0.4 min), 600 °C/min, 300 °C (15 min), 50 °C/min, 120 °C, splitless
Detector:	Quadrupole MS, EI in SIM, source 230 °C, transfer line 280°C

Results and Discussion

The VF-5ms column delivered a multi-residue analysis of PAHs, PCBs and OCPs in 32 minutes (Figure 1). Accurate quantification of the 57 compounds in a single run was challenging, because every group of compounds presented its own difficulties in separation.

For the PAH group two pairs were difficult to resolve. The first pair was benzo[b]fluoranthene/benzo[k]fluoranthene. These two compounds have the same mass and can therefore not be separated by MS alone. The second PAH pair was indeno[1,2,3-c,d]pyrene/dibenz[a,h]anthracene. These compounds have different masses, 276 and 278 respectively, but are difficult to resolve using just MS.

In the PCB and OCP group, PCB 138/PCB 163, and p,p′-DDD/o,p′-DDT, have the same mass spectra and cannot be resolved by MS when co-eluting. The OCP group has another pair that is difficult to resolve; cis-heptachlor epoxide/trans-heptachlor epoxide both have m/z 353 in their mass spectra. For SIM analysis, an extra m/z should be analyzed to confirm whether it is cis- or trans-heptachlor epoxide.

Figures 2 to 6 show the peak pairs that are difficult to resolve.

Table 1. Peak Identification for Figure 1

Peak	Compound	Ions
1	Naphthalene	128
2	Acenaphthylene	152
3	Acenaphthene	154
4	Fluorene	166
5	α-HCH	219, 181
6	Hexachlorobenzene	284, 249
7	β-HCH	219, 183
8	γ-HCH	219, 181
9	PCB 18	256, 186
10	Phenanthrene	178
11	Anthracene	178
12	δ-HCH	219, 181
13	PCB 28	258, 186
14	PCB 31	258, 186
15	PCB 20	256, 186
16	Heptachlor	272, 100
17	PCB 52	292, 220
18	Aldrin	263, 293
19	PCB 44	292, 220
20	trans-Heptachlor epoxide	353, 81
21	cis-Heptachlor epoxide	219, 183
22	Fluoranthene	202
23	PCB 155	360, 290
24	trans-Chlordane	373, 237
25	o,p′-DDE	246, 318
26	PCB 101	326, 256
27	Pyrene	202
28	cis-Chlordane	373, 237
29	Endosulfan I	241, 195
30	p,p′-DDE	246, 318
31	o,p′-DDD	235, 165
32	Dieldrin	263, 79
33	Endrin	263, 81
34	PCB 149	360, 290
35	PCB 118	326, 254
36	Endosulfan II	241, 195
37	p,p′-DDD	235, 165
38	o,p′-DDT	235, 165
39	Endrin-aldehyde	345, 250
40	PCB 153	360, 290
41	PCB 105	326, 254
42	Endosulfan sulfate	272, 387
43	p,p′-DDT	235, 165
44	PCB 163	360, 290
45	PCB 138	360, 290
46	Benz[a]anthracene	228
47	Chrysene	228
48	Methoxychlor	227
49	PCB 180	396, 324
50	PCB 170	394, 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

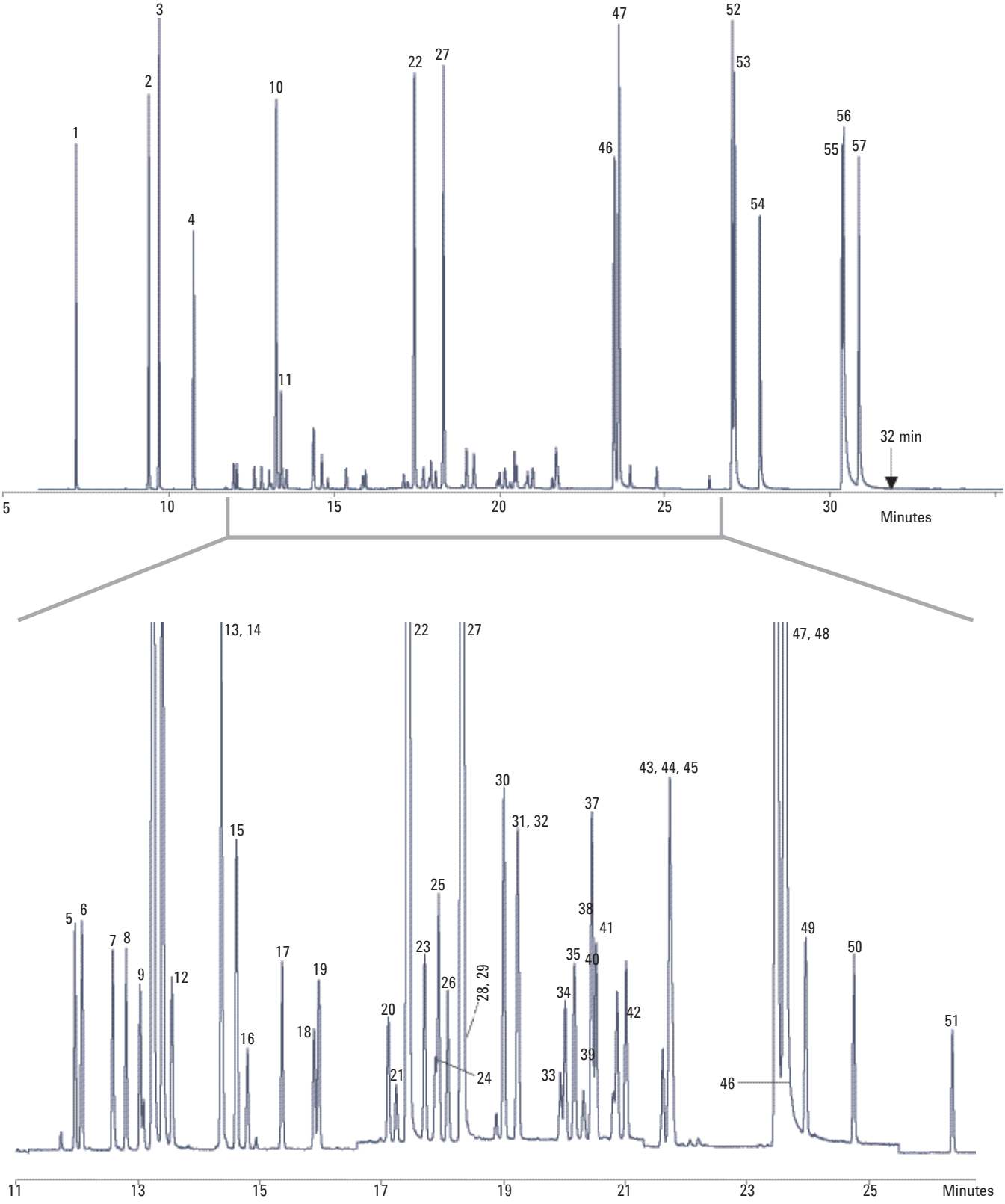

Figure 1. Total ion chromatogram multi-residue analysis on a VF-5ms column

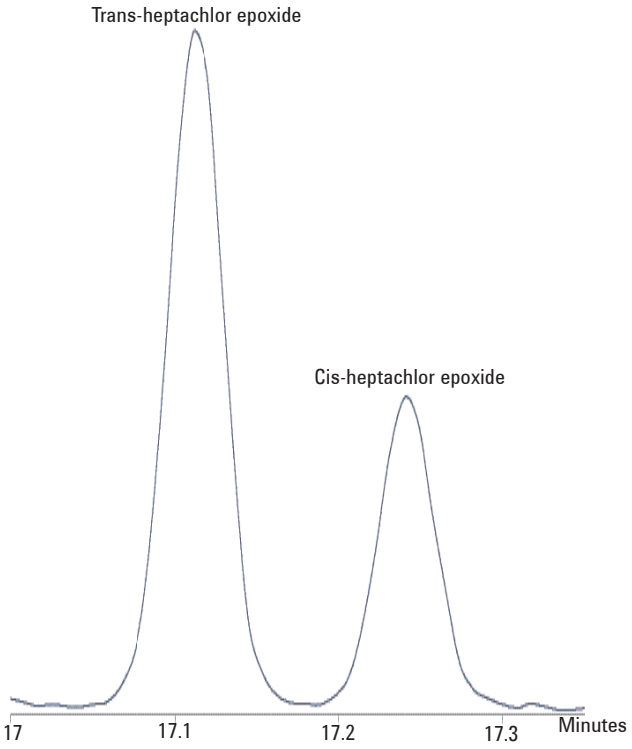

Figure 2. Zoomed total ion chromatogram of cis-heptachlor epoxide/trans-heptachlor epoxide

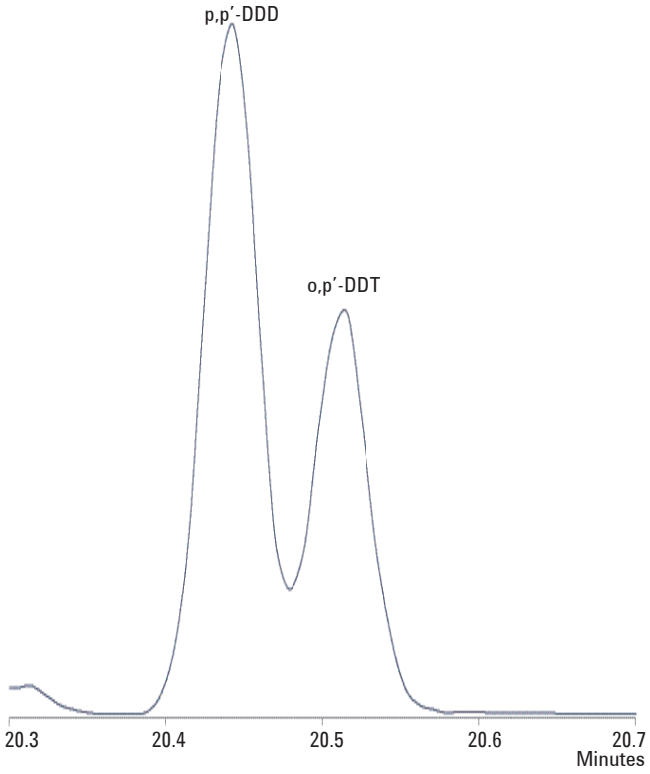

Figure 3. Selected ion chromatogram (m/z 235) of p,p′-DDD/o,p′-DDT

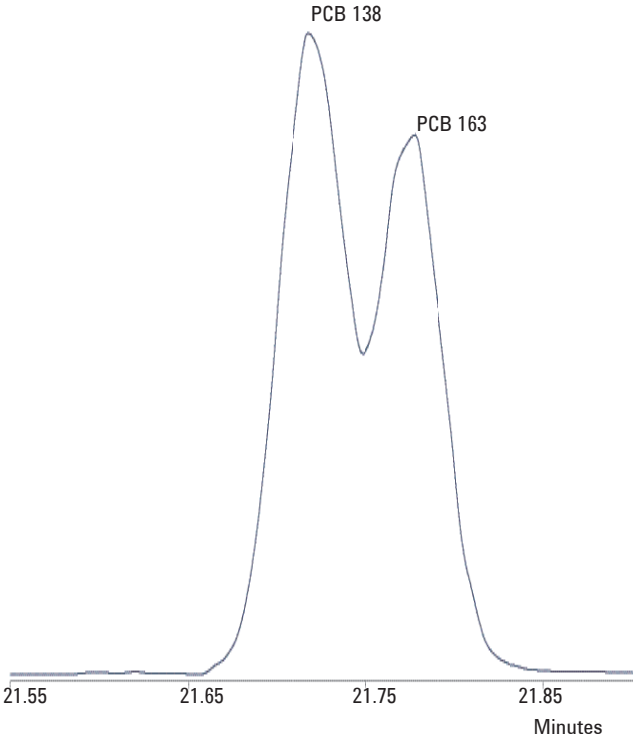

Figure 4. Selected ion chromatogram (m/z 360) of PCB 138/PCB 163

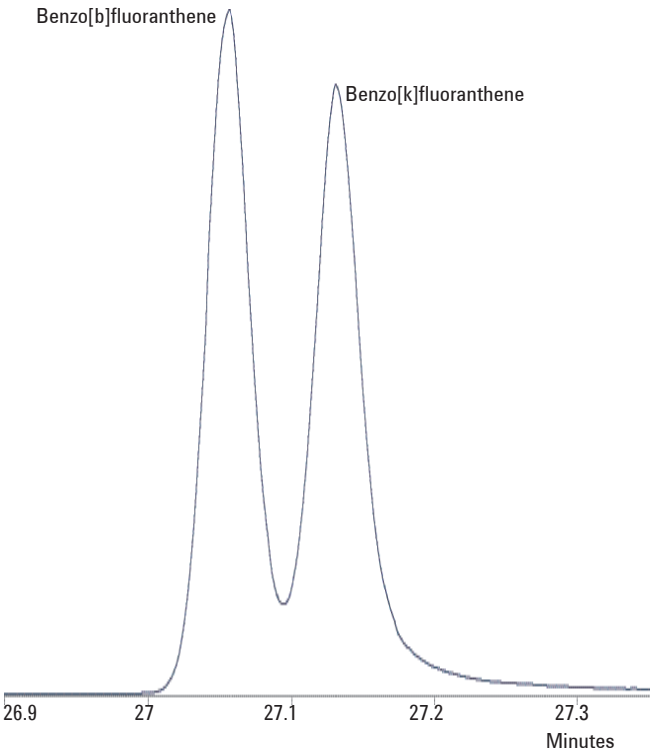

Figure 5. Zoomed total ion chromatogram of benzo[b]fluoranthene/ benzo[k]fluoranthene

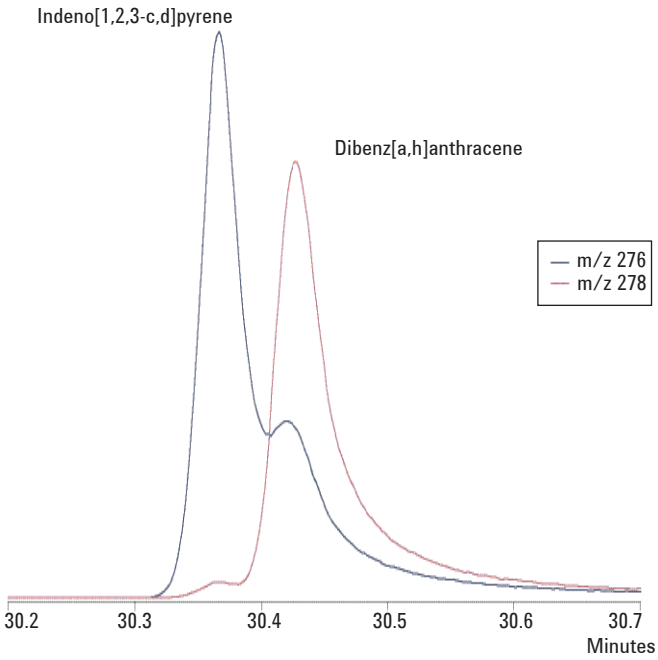

Figure 6. Selected ion chromatogram (m/z 276 and 278) of indeno[1,2,3-c,d]pyrene and dibenz[a,h]anthracene

Conclusion

The VF-5ms column and the optimized oven program separated 57 environmental pollutants in only 32 minutes in a multi-residue analysis. This multi-purpose column exhibits minimum column bleed for improved sensitivity, and provides excellent selectivity for aromatic compounds.

References

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