Multi-residue Analysis of PAHs, PCBs and OCPs using an Agilent J&W FactorFour VF-35ms Column

Application Note

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Introduction

Multi-residue analysis involves the separation of different groups of compounds in a single operation. One of the most important advantages of multi-residue analysis is the opportunity to screen and quantify a multitude of components in a short time span, reducing analysis costs. A multi-residue method for the separation of polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs) and organochlorine pesticides (OCPs) is described here.

PAHs, PCBs and OCPs have different sources and belong to different chemical classes. PAHs contain two or more aromatic rings and are formed during incomplete combustion or pyrolysis of organic matter. OCPs are pesticides containing mainly carbon, hydrogen and chlorine atoms. They break down slowly and can remain in the environment long after application, and bioaccumulate in organisms during prolonged exposure. PCBs are characterized by two phenyl groups, with varying numbers of chlorine atoms. PCBs are used in many industrial and commercial applications, for example, as plasticizers in paints, plastics and rubber products, and in pigments and dyes. All of these compounds are typically very persistent in sediments, plants and animals.

The 16 EPA PAHs,17 PCBs (including the 6 EU marker PCBs) and 24 common 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, a concentration difference that is common also in real environmental samples.

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Conditions

Technique:	GC/MS
Column:	VF-35ms, 30 m x 0.25 mm, df=0.25 µm (part number CP8877)
Sample Conc:	OCPs and PCBs 0.1 µg/mL, PAHs 1 µg/mL
Temperature:	75 °C (1 min), 20 °C/min, 220 °C, 5 °C/min, 260 °C (2 min), 7 °C/min, 305 °C, 2 °C/min, 330 °C
Injection Volume:	1.0 µL
Carrier Gas:	Helium, constant flow, 1.0 mL/min
Injection:	100 °C (0.4 min), 600 °C/min, 300 °C (15 min), 50 °C/min, 120 °C, splitless
Detection:	Quadrupole MS, EI in SIM, Source 230 °C, Transfer line 280 °C

Results and Discussion

The VF-35ms column delivered a multi-residue analysis of 57 PAHs, PCBs and OCPs in 32.5 minutes (Figure 1). For PAHs, two pairs are difficult to resolve, namely benzo[b] fluoranthene/benzo[k]fluoranthene and indeno[1,2,3-c,d] pyrene/dibenz[a,h]anthracene. The first pair has the same mass and therefore cannot be separated by MS alone. The second pair have different masses (276 and 278 respectively) and again are difficult to resolve using only MS.

For PCB and OCP groups, PCB 138/PCB 163 and p,p'-DDD/o,p'-DDT have the same mass spectra and cannot be separated by MS. The OCP group also has another pair difficult to resolve, namely cis-heptachlor epoxide and trans-heptachlor epoxide. These compounds both have the main m/z 353 in their mass spectra. Confirmation of both compounds therefore requires an additional m/z.

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

Table 1. Peak Identification and SIM ions

Peak	Compound	Ions
1	Naphthalene	128
2	Acenaphthylene	152
3	Acenaphthene	154
4	Fluorene	166
5	Hexachlorobenzene	284, 249
6	α-HCH	181, 219
7	PCB 18	256, 186
8	γ-HCH	181, 219
9	β-HCH	181, 219
10	Phenanthrene	178
11	Anthracene	178
12	PCB 28	256, 186
13	PCB 31	256, 186
14	Heptachlor	272, 100
15	δ-HCH	181, 219
16	PCB 20	256, 186
17	PCB 52	292, 220
18	Aldrin	66, 263
19	PCB 44	292, 220
20	trans-Heptachlor epoxide	353, 81
21	cis-Heptachlor epoxide	81, 183
22	PCB 155	360, 290
23	trans-Chlordane	373, 326
24	PCB 101	326, 254
25	o,p'-DDE	246, 318
26	cis-Chlordane	373, 237
27	Fluoranthene	202
28	Endosulfan I	195, 241
29	p,p'-DDE	246, 318
30	Dieldrin	79, 263
31	Pyrene	202
32	o,p'-DDD	235, 165
33	PCB 118	326, 254
34	PCB 149	360, 290
35	Endrin	263, 81
36	PCB 153	360, 290
37	o,p'-DDT	235, 165
38	p,p'-DDD	235, 165
39	Endosulfan II	241, 195
40	PCB 105	326, 254
41	PCB 163	360, 290
42	PCB 138	360, 290
43	p,p'-DDT	235, 165
44	Endrin-aldehyde	345, 67
45	Endosulfan sulfate	272, 387
46	PCB 180	396, 324
47	Methoxychlor	227
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

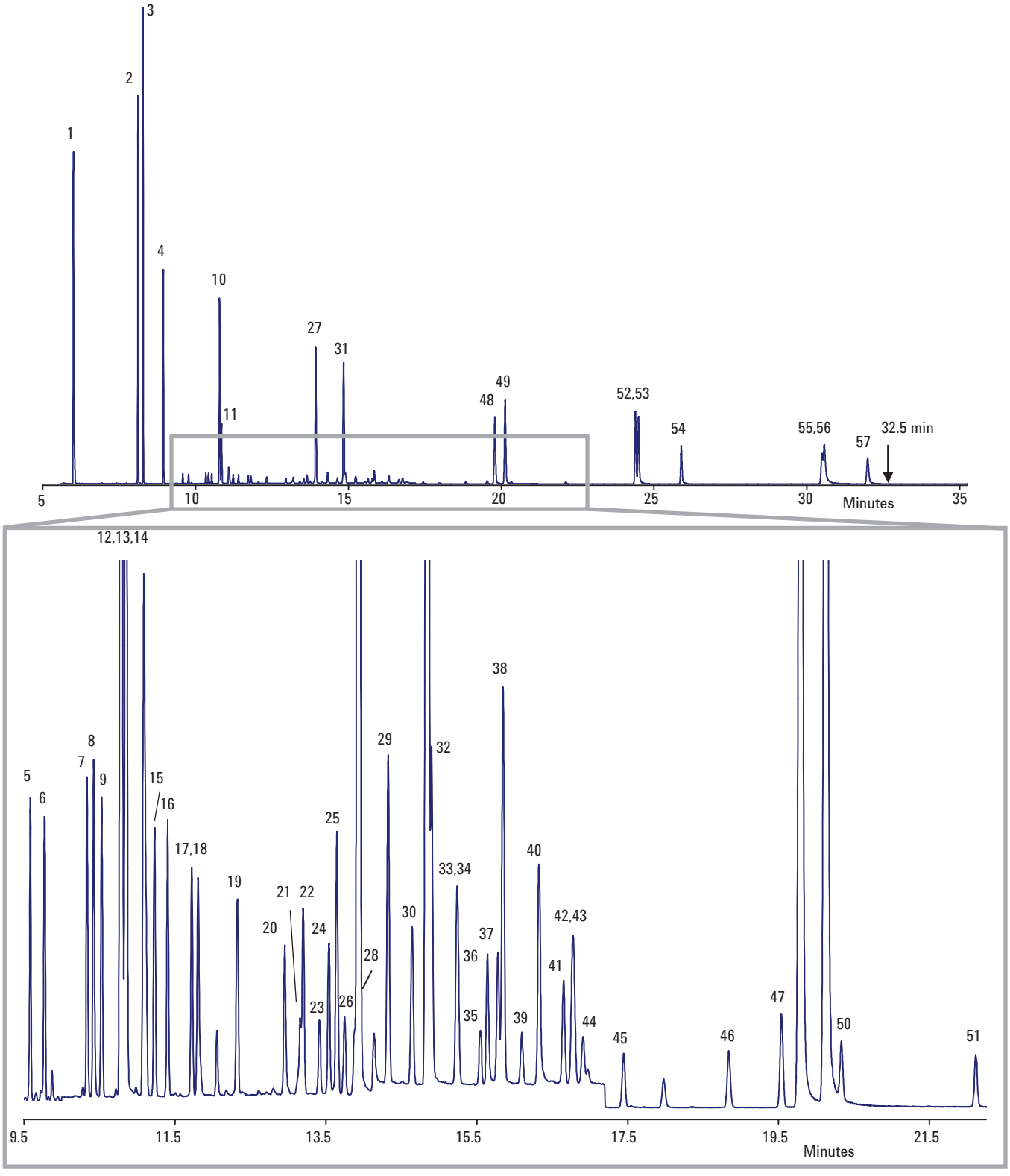

Figure 1. Total ion chromatogram multi-residue analysis on a VF-35ms column.

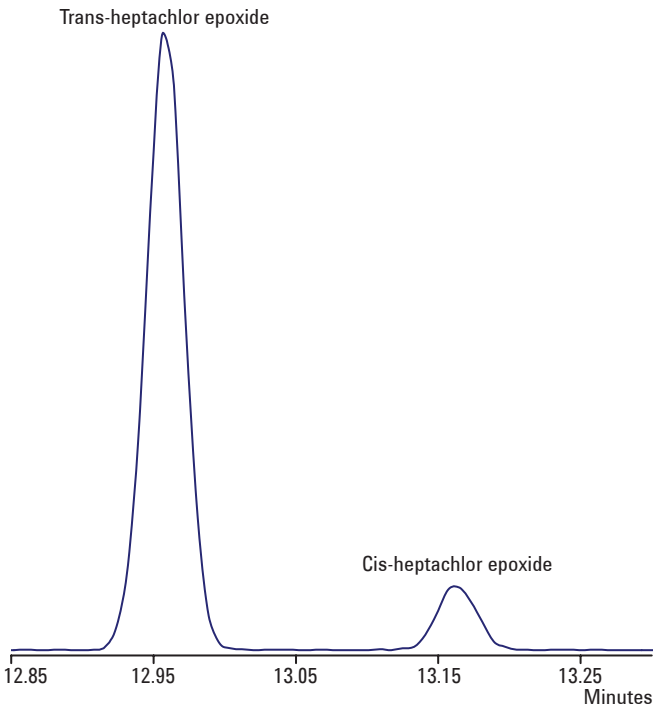

Figure 2. Zoomed total ion chromatogram of cis-heptachlor epoxide / trans-heptachlor epoxide

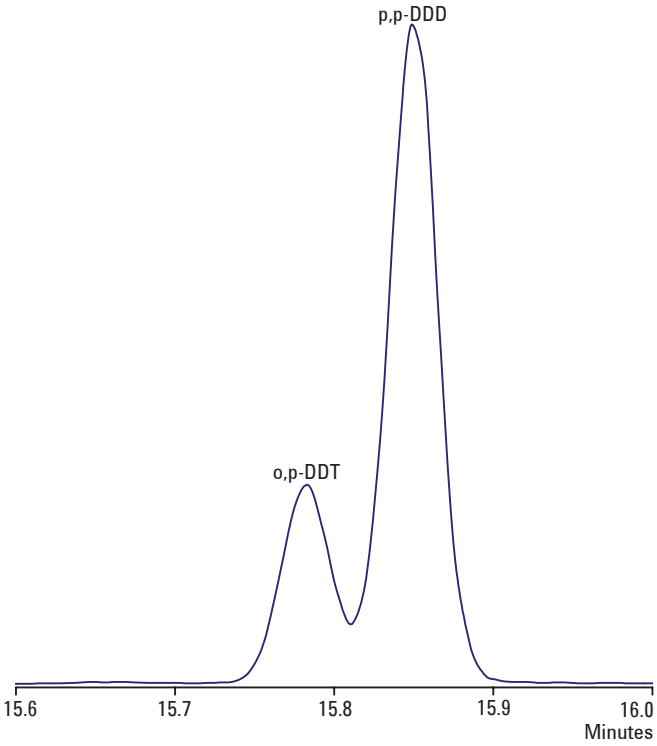

Figure 3. Selected ion chromatogram (m/z 235) of p,p'-DDD / o,p'-DDT

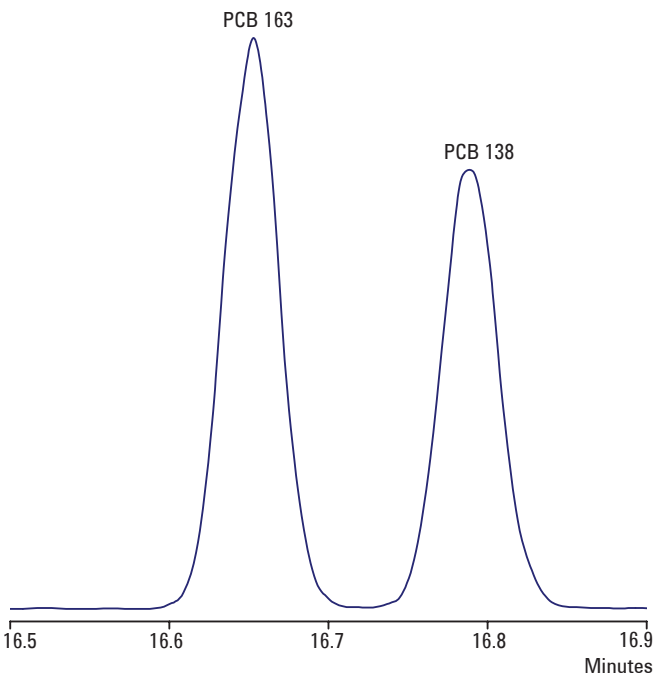

Figure 3. Selected ion chromatogram (m/z 360) of PCB 138/PCB 163

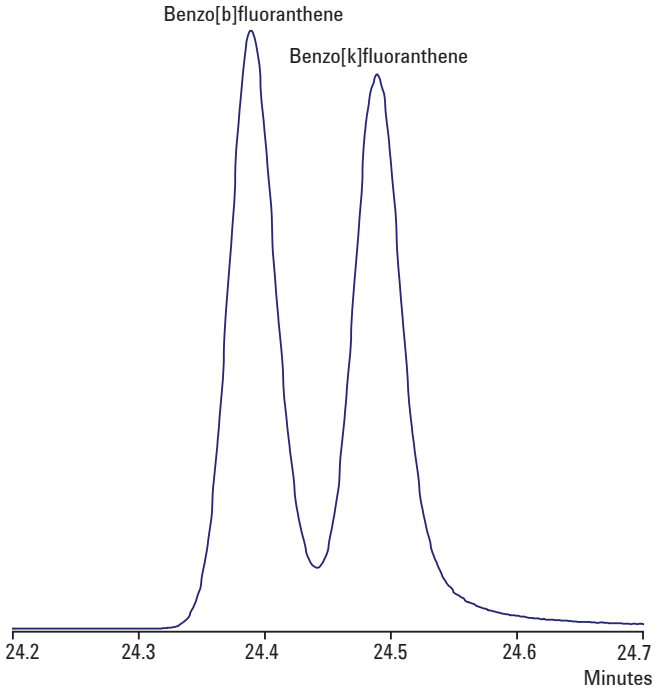

Figure 4. Zoomed total ion chromatogram of benzo[b]fluoranthene / benzo[k]fluoranthene

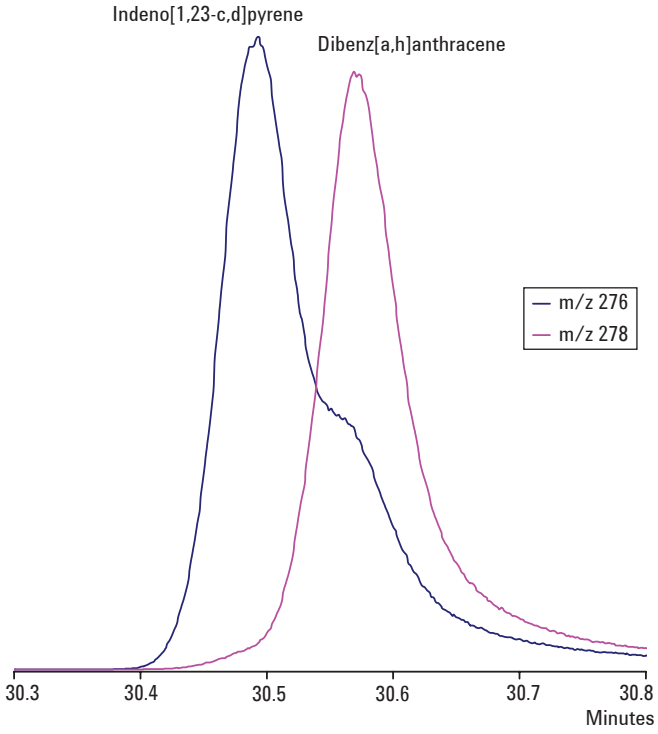

Figure 6. Selected ion chromatogram (m/z 276 and 278) of indeno[1,2,3-c,d]pyrene and dibenz[a,h]anthracene

Conclusion

Analyzing all of these compounds in a single run is normally problematic because every group presents its own difficulties in separation. However, with the VF-35ms column and optimized oven program, multi-residue separation is achieved in about 32 minutes. The VF-35ms column is based on a medium polarity, highly robust and inert phase, making it the ideal choice for demanding trace environmental and chemical analysis.

References

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www.agilent.com/chem

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