Multi-residue Analysis for PAHs, PCBs and OCPs on Agilent J&W FactorFour VF-200ms

Application Note

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Introduction

With multi-residue analysis, different groups of compounds are analyzed in a single run. Enhanced productivity is one of the most important advantages of this type of analysis. This application 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 and structures. PAHs contain two or more aromatic rings. They are formed during incomplete combustion or pyrolysis of organic matter. Characteristically, PCBs contain two phenyl groups, with varying number of chlorine atoms. Due to their non-flammability, chemical stability, high boiling point and electrical insulating properties, PCBs were used in hundreds of industrial and commercial applications, for example, in electrical, heat transfer and hydraulic equipment, as plasticizers in paints, plastics and rubber products, and in pigments, dyes and carbonless copy paper. OCPs are pesticides that primarily consist of carbon, hydrogen and chlorine. Most of them break down slowly and can remain in the environment long after application, and in organisms long after exposure. These compounds 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 can be found in real environmental samples.

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Materials and Methods

Technique:	GC/MS
Column:	VF-200ms, 30m x 0.25mm, df = 0.25µm (part number CP8858)
Sample Conc:	OCPs and PCBs 0.1 µg/mL, PAHs 1 µg/mL
Injection Volume:	1 µL
Temperature:	70 °C (1 min), 20 °C/min, 190 °C, 10 °C/min, 250 °C, 5 °C/min, 260 °C, 2 °C/min, 280 °C
Carrier Gas:	Helium, constant flow, 1 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-200ms column provided a multi-residue analysis of PAHs, PCBs and OCPs in 24 minutes (Figure 1). Analyzing 57 compounds in a single run is normally quite challenging because every group of compounds presents its own difficulties in separation.

In the PAH group, two pairs of isomers are difficult to resolve. The first pair is benzo[b]fluoranthene and benzo[k]-fluoranthene. These two compounds have the same mass and therefore cannot be separated by MS alone. Another PAH pair comprises indeno[1,2,3-c,d]pyrene and dibenz[a,h] anthracene. These compounds have different masses (278 and 276 respectively) but again are difficult to resolve with just MS.

In the PCB and OCP group, PCB 28/PCB 31 and p,p′-DDD/ o,p′-DDT have the same mass spectra and cannot be resolved by MS. 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 the presence of cis- or trans-heptachlor epoxide.

Figures 2 to 6 show the peak pairs that are difficult to resolve, although the VF-200ms is very capable of providing adequate separation.

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, 149
6	PCB 18	186, 256
7	α-HCH	183, 219
8	δ-HCH	181, 219
9	Phenanthrene	178
10	Anthracene	178
11	PCB 31	186, 256
12	PCB 28	186, 256
13	β-HCH	219, 183
14	PCB 20	186, 256
15	Heptachlor	272, 100
16	γ-HCH	183, 219
17	PCB 52	292, 220
18	Aldrin	66, 263
19	PCB 44	220, 292
20	PCB 155	360, 290
21	o,p'-DDE	246, 318
22	cis-Heptachlor epoxide	183, 353
23	PCB 101	326, 256
24	trans-Heptachlor epoxide	81, 353
25	trans-Chlordane	373, 237
26	cis-Chlordane	373, 237
27	Fluoranthene	202
28	p,p'-DDE	246, 318
29	Endosulfan I	137, 77
30	o,p'-DDD	235, 165
31	Pyrene	202
32	PCB 149	360, 180
33	o,p'-DDT	235, 165
34	PCB 118	326, 254
35	Dieldrin	79, 137
36	PCB 153	360, 290
37	PCB 105	326, 254
38	Endrin	137, 77
39	p,p'-DDD	235, 165
40	PCB 138	360, 290
41	PCB 163	360, 290
42	p,p'-DDT	235, 165
43	Endosulfan II	137, 77
44	Methoxychlor	227
45	PCB 180	394, 324
46	Endrinaldehyde	67, 345
47	PCB 170	394, 324
48	Benz[a]anthracene	228
49	Chrysene	228
50	Endosulfan sulfate	272, 387
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	278
56	Dibenz[a,h]anthracene	276
57	Benzo[g,h,i]perylene	276

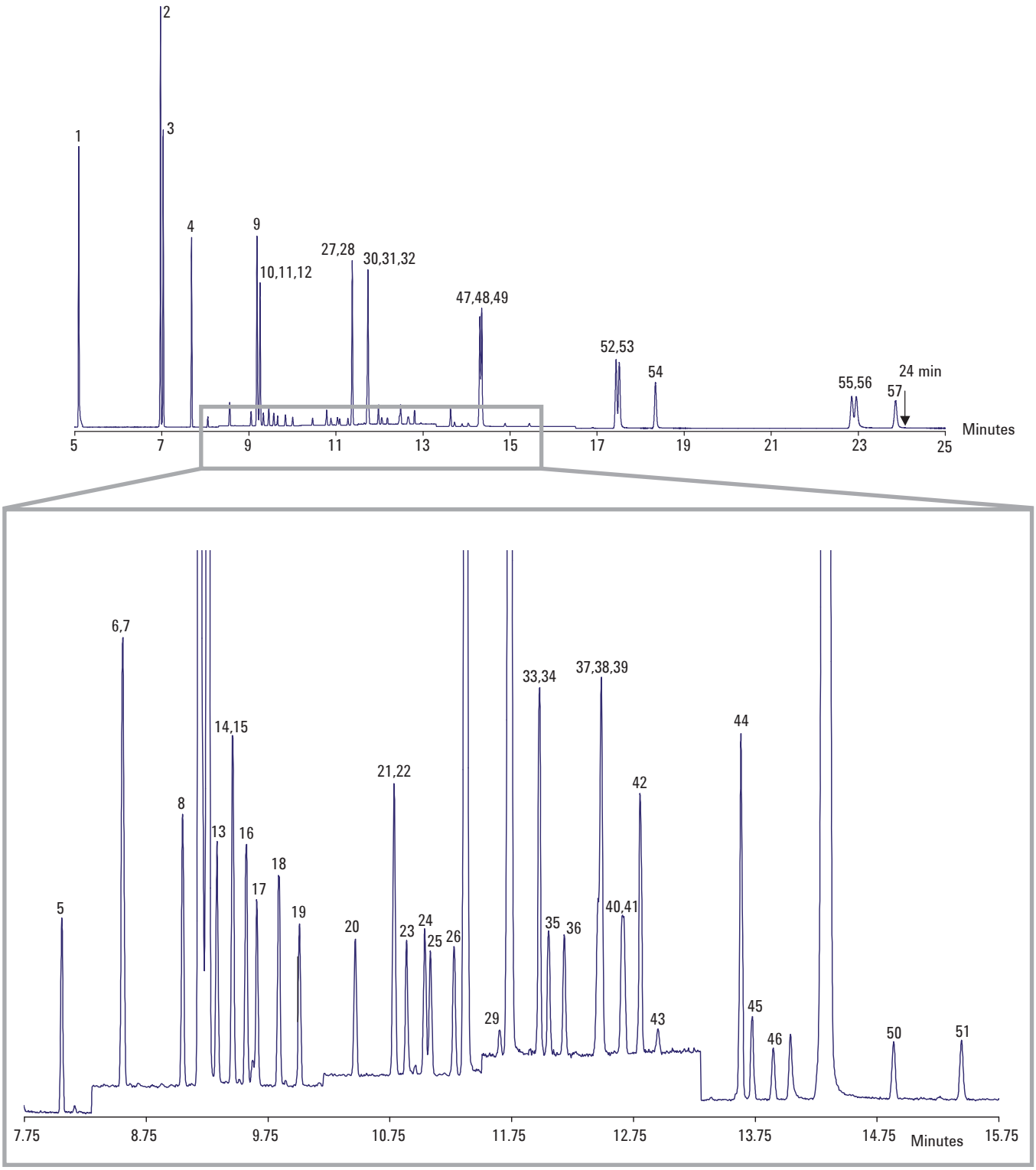

Figure 1. Total ion chromatogram (TIC) multi-residue analysis on a VF-200ms column

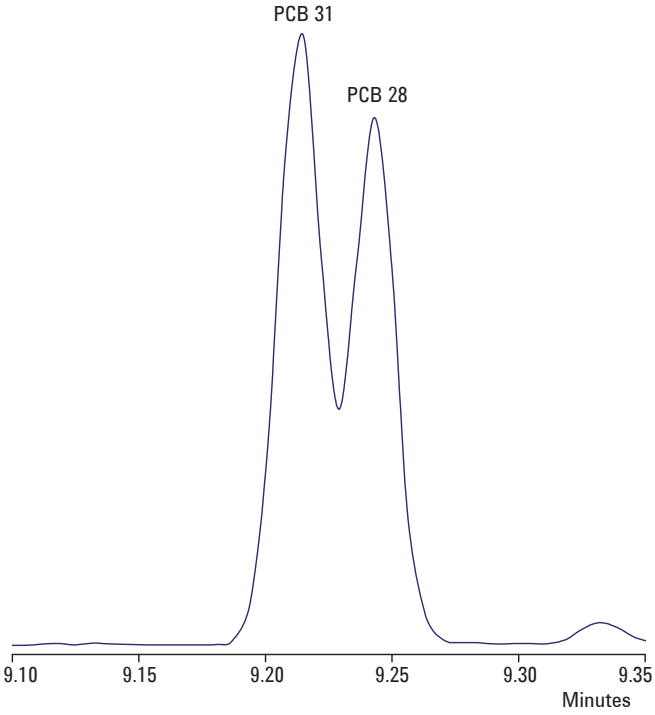

Figure 2. Selected ion chromatogram (m/z 256) of PCB 28/PCB 31

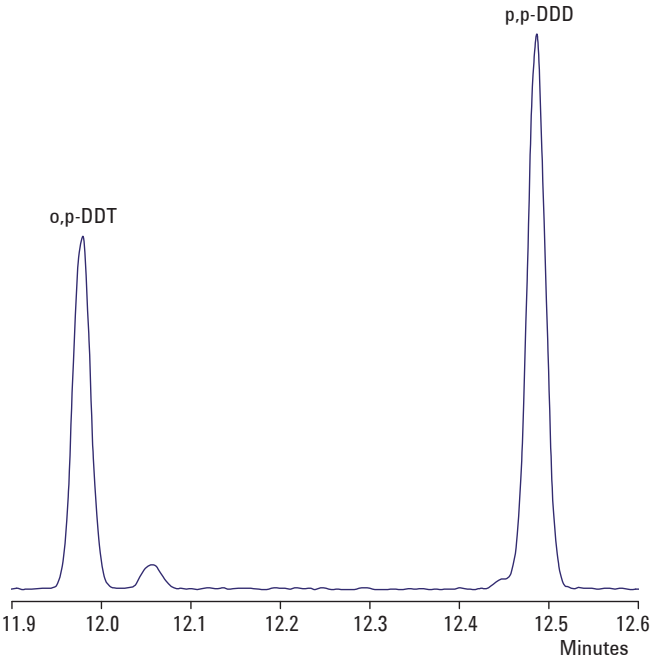

Figure 4. Separation of p,p'-DDD/o,p'-DDT (m/z 235)

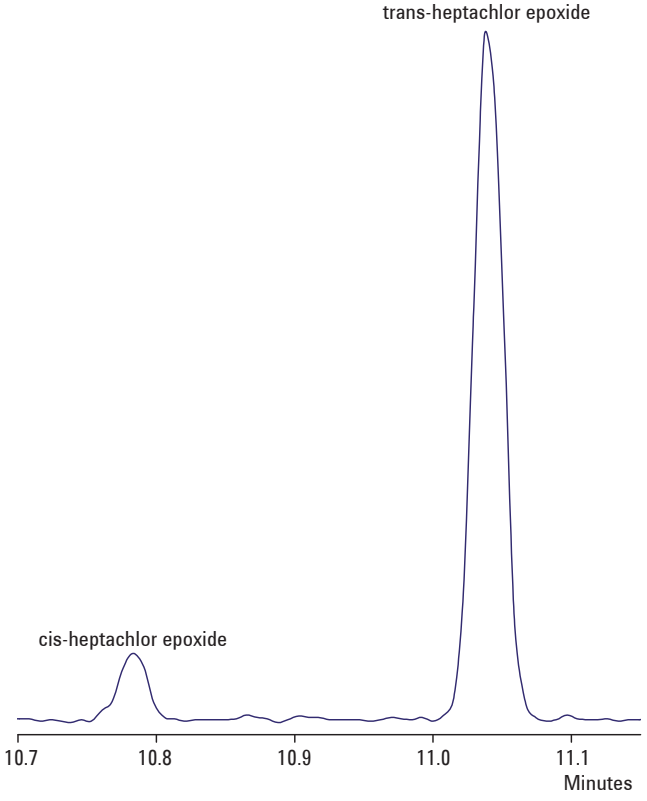

Figure 3. Selected ion chromatogram (m/z 353) of cis-heptachlor epoxide/ trans-heptachlor epoxide

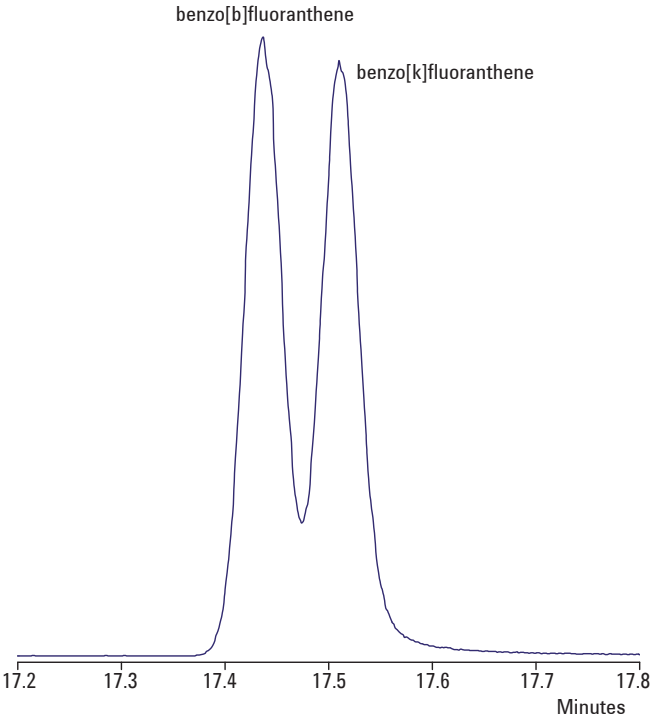

Figure 5. Separation of benzo[b]fluoranthene/benzo[k]fluoranthene (TIC)

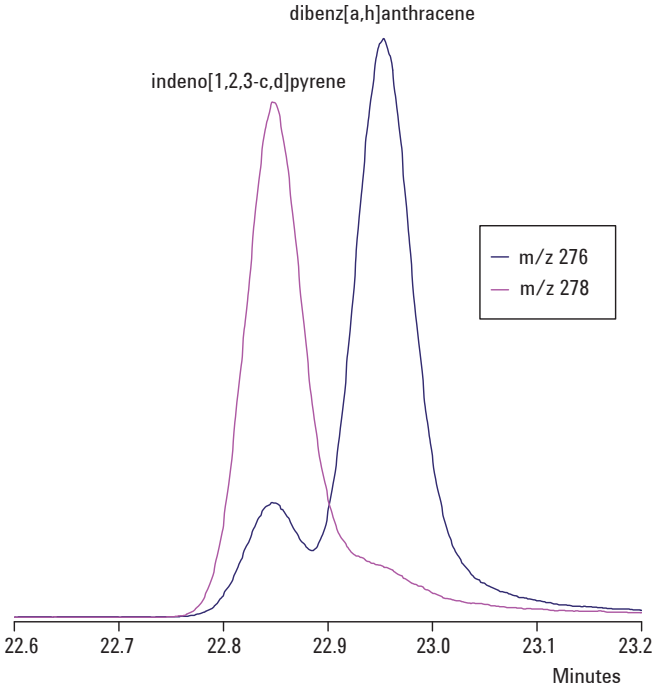

Figure 6. Selected ion chromatogram (m/z 276 and 278) of indeno-[1,2,3-c,d]pyrene and dibenz[a,h]anthracene

Conclusion

A J&W FactorFour VF-200ms column and optimized oven program delivered a multi-residue analysis of 57 environmental pollutants in 24 minutes. The VF-200ms column has a unique selectivity for polar compounds and is especially suited to electron rich, high dipole moment compounds, such as PAHs and chloro-containing molecules.

References

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www.agilent.com/chem

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