Fast Separation of EU and US EPA Regulated PAHs on Agilent J&W Select PAH GC Columns

Application Note

Author

John Oostdijk
Agilent Technologies, Inc.

Introduction

Several polycyclic aromatic hydrocarbons (PAHs) have the same mass, making them difficult to separate using GC/MS. To resolve these PAHs, the use of an appropriate column and an optimized oven program are necessary. In addition, speed of analysis is important for high productivity, but faster speeds often lead to lower resolution. With the 15 m x 0.15 mm ID version of the J&W Select PAH GC column, it is possible to perform high resolution PAH analysis at high speed with no loss of resolution. In this application note, we demonstrate high resolution analysis of 54 PAHs within 30 minutes.

PAHs are formed during incomplete combustion or pyrolysis of organic matter, during industrial processes and in cooking and food processing. Due to their carcinogenic properties, they are pollutants of concern and are subject to monitoring in food and environmental samples. However, different legislations describe different PAHs. The US Environmental Protection Agency (EPA) focuses on environmental PAH contamination whereas the EU legislation is more concerned with PAHs in foodstuffs.

Conditions

Technique:	GC/MS, Triple Quad
Column:	Select PAH, 15 m x 0.15 mm, df = 0.10 µm (part number CP7461)
Sample Conc:	Approx. 0.1-0.3 µg/mL
Injection Volume:	1 µL
Temperature:	70 °C (0.4 min), 70 °C/min, 180 °C, 7 °C/min, 230 °C (7 min), 50 °C/min, 280 °C (7 min), 30 °C/min, 350 °C (4 min)
Carrier Gas:	Helium, constant flow 1.2 mL/min
Injector:	300 °C, Splitless mode, 0.5 min @ 100 mL/min
Detector:	Triple Quad, EI in SIM mode, ion source 275 °C, transfer line 300 °C

Results and Discussion

There are three groups of peaks that are difficult to resolve when analyzing PAHs. Benz[a]anthracene, cyclopenta[c,d]pyrene, chrysene and triphenylene have different masses, m/z 226 and 228. However, the compounds in this group with m/z 228 also contain some m/z 226, making resolution problematic using only MS. The same complication occurs when separating indeno[1,2,3-c,d]pyrene, benzo[b]triphenylene and dibenz[a,h]anthracene with m/z 276 and 278.

The third set of difficult to resolve PAHs are the benzofluoranthene isomers, benzo[b]fluoranthene, benzo[j]fluoranthene and benzo[k]fluoranthene, which have the same mass and, as before, cannot be resolved with MS only.

Therefore, resolution of these isomers must be done chromatographically. This is straightforward and fast using the Select PAH column and the optimized oven program, as shown in Figure 1 (all PAHs) and Figures 2-8 (details and information).

Table 1. Peak identification of Figure 1

Peak	MW	Compound	EPA 610	SFC & EFSA PAHs (15+1)	CAS
1	136	Naphthalene-d8			1146-65-2
2	128	Naphthalene	x		91-20-3
3	142	2-Methylnaphthalene			91-57-6
4	142	1-Methylnaphthalene			90-12-0
5	152	Acenaphthylene	x		208-96-8
6	164	Acenaphthene-d10			15067-26-2
7	154	Acenaphthene	x		83-32-9
8	166	Fluorene	x		86-73-7
9	188	Phenanthrene-d10			1517-22-2
10	178	Phenanthrene	x		85-01-8
11	178	Anthracene	x		120-12-7
12	202	Fluoranthene	x		206-44-0
13	202	Pyrene	x		129-00-0
14	216	Benzo[a]fluorene			238-84-6
15	216	Benzo[b]fluorene			243-17-4
16	216	7H-Benzo[c]fluorene		x	205-12-9
17	234	Benzo[b]naphtho[2,1-d]thiophene			239-35-0
18	226	Benzo[g,h,i]fluoranthene			203-12-3
19	228	Benzo[c]phenanthrene			195-19-7
20	228	Benz[a]anthracene	x	x	56-55-3
21	226	Cyclopenta[c,d]pyrene		x	27208-37-3
22	240	Chrysene-d10			1719-03-5
23	228	Triphenylene			217-59-4
24	228	Chrysene	x	x	218-01-9
25	242	6-Methylchrysene			1705-85-7
26	242	5-Methylchrysene		x	3697-24-3
27	252	Benzo[b]fluoranthene	x	x	205-99-2
28	252	Benzo[k]fluoranthene	x	x	207-08-9
29	252	Benzo[j]fluoranthene		x	205-82-3
30	252	Benzo[a]fluoranthene			203-33-8
31	252	Benzo[e]pyrene			192-97-2
32	252	Benzo[a]pyrene	x	x	50-32-8
33	264	Perylene-d12			1520-96-3
34	252	Perylene			198-55-0
35	268	3-Methylcholanthrene			56-49-5
36	330	9,10-diphenylanthracene			216-105-1
37	279	Dibenzo[a,h]acridine			226-36-8
38	279	Dibenzo[a,j]acridine			224-42-0
39	278	Dibenz[a,j]anthracene			224-41-9
40	292	Dibenzo[a,h]anthracene D14			13250-98-1
41	278	Benzo[b]triphenylene			215-58-7
42	276	Indeno[1,2,3-cd]pyrene	x	x	193-39-5
43	278	Dibenz[a,h]anthracene	x	x	53-70-3
44	278	Benzo[b]chrysene			214-17-5
45	278	Picene			213-46-7
46	27	Benzo[g,h,i]perylene	x	x	191-24-2
47	276	Dibenzo[def,mno]chrysene			191-26-4
48	267	7H-Dibenzo[c,g]carbazole			194-59-2
49	302	Dibenzo[a,l]pyrene		x	191-30-0
50	302	Dibenzo[a,e]pyrene		x	192-65-4
51	300	Coronene			191-07-1
52	302	Benzo[b]perylene			197-70-6
53	302	Dibenzo[a,i]pyrene		x	189-55-9
54	302	Dibenzo[a,h]pyrene		x	189-64-0

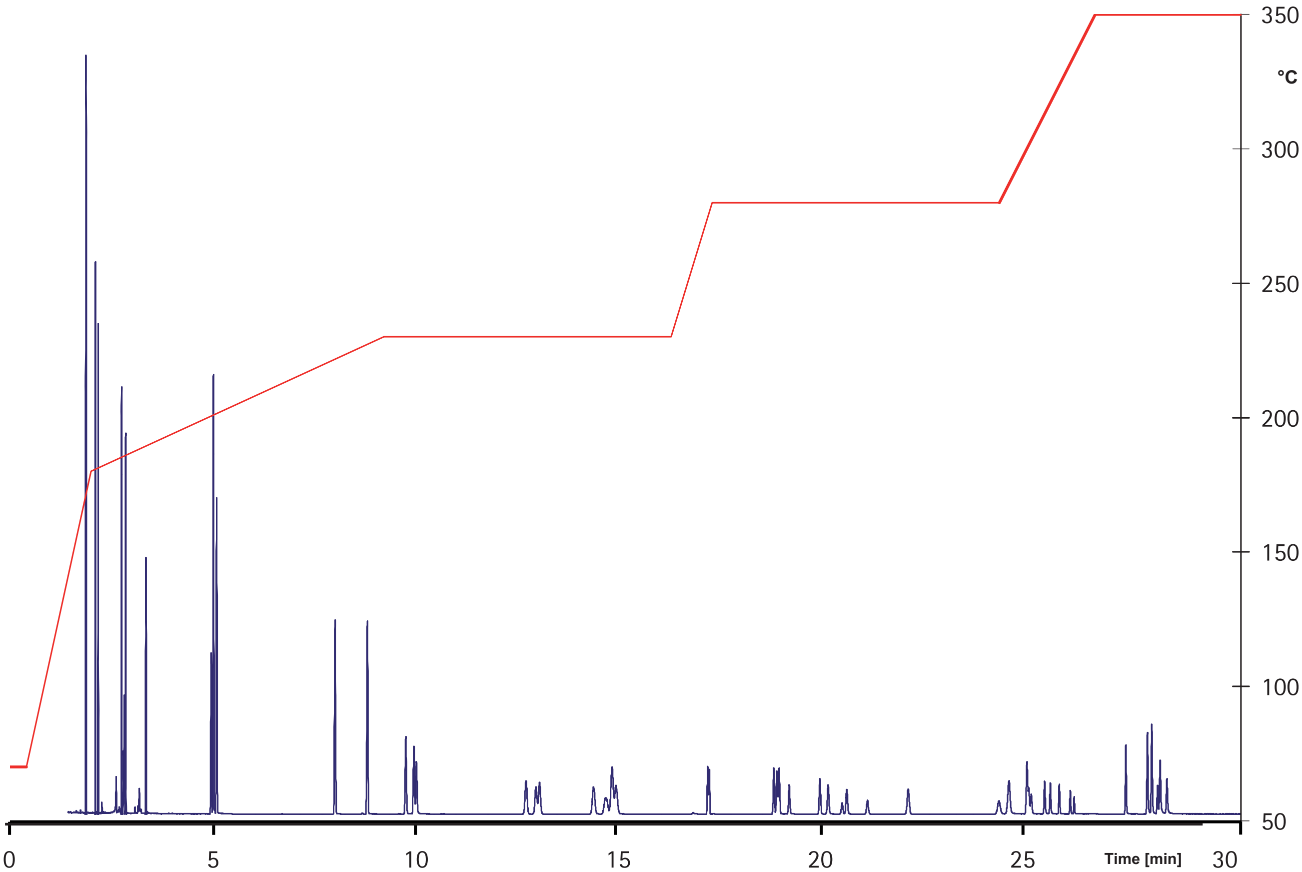

Figure 1. GC/MS analysis of EU and EPA PAHs on Select PAH 15 m x 0.15 mm x 0.10 µm

Table 2. Peak Identification for Figure 2

Peak	MW	Compound	EPA 610	CAS
1	136	Naphthalene-d8		1146-65-2
2	128	Naphthalene	x	91-20-3
3	142	2-Methylnaphthalene		91-57-6
4	142	1-Methylnaphthalene		90-12-0
5	152	Acenaphthylene	x	208-96-8
6	164	Acenaphthene-d10		15067-26-2
7	154	Acenaphthene	x	83-32-9
8	166	Fluorene	x	86-73-7

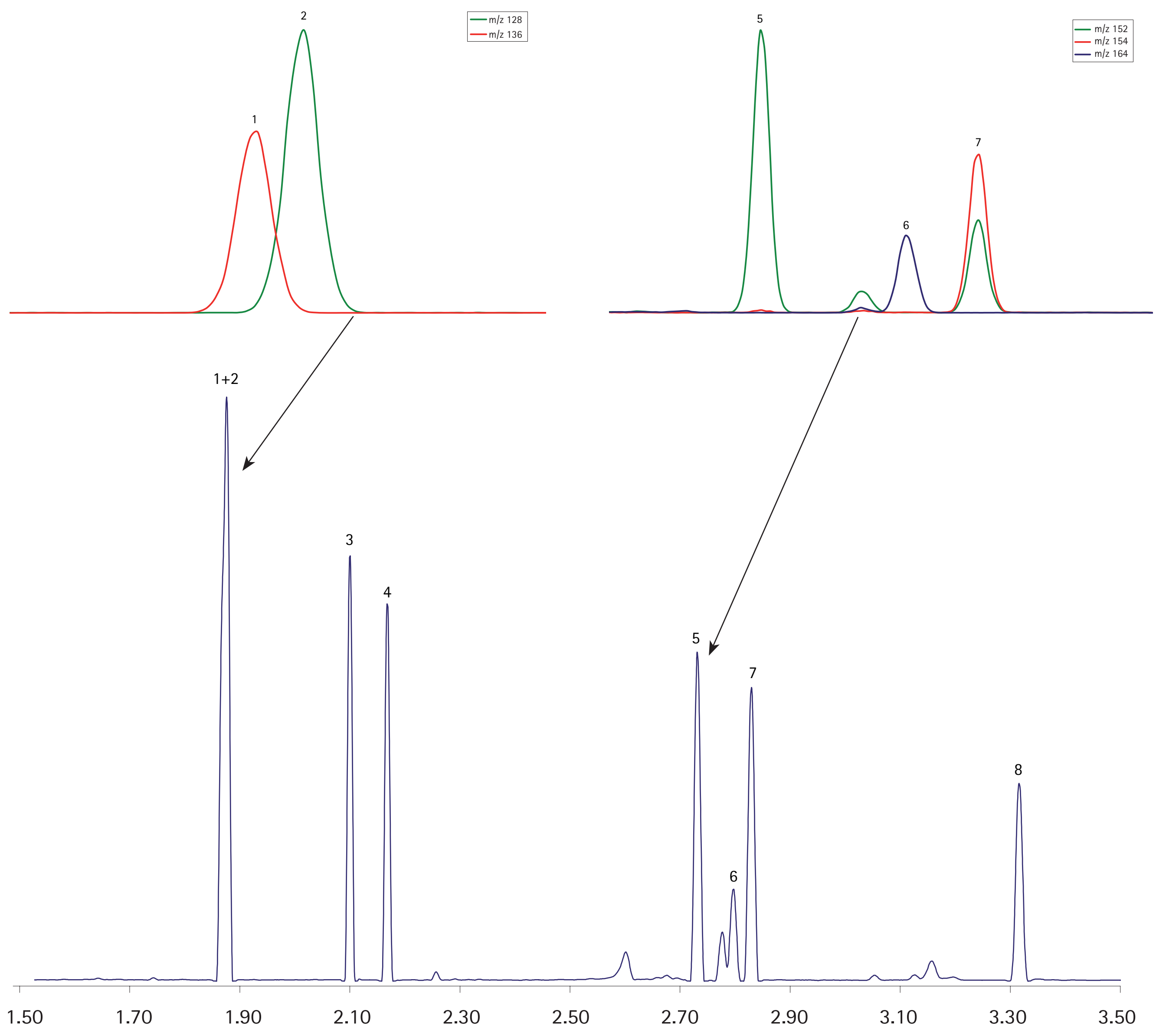

Figure 2. Details and identification of peaks 1 to 8

Table 3. Peak Identification for Figure 3

Peak	MW	Compound	EPA 610	CAS
9	188	Phenanthrene-d10		1517-22-2
10	178	Phenanthrene	x	85-01-8
11	178	Anthracene	x	120-12-7
12	202	Fluoranthene	x	206-44-0
13	202	Pyrene	x	129-00-0

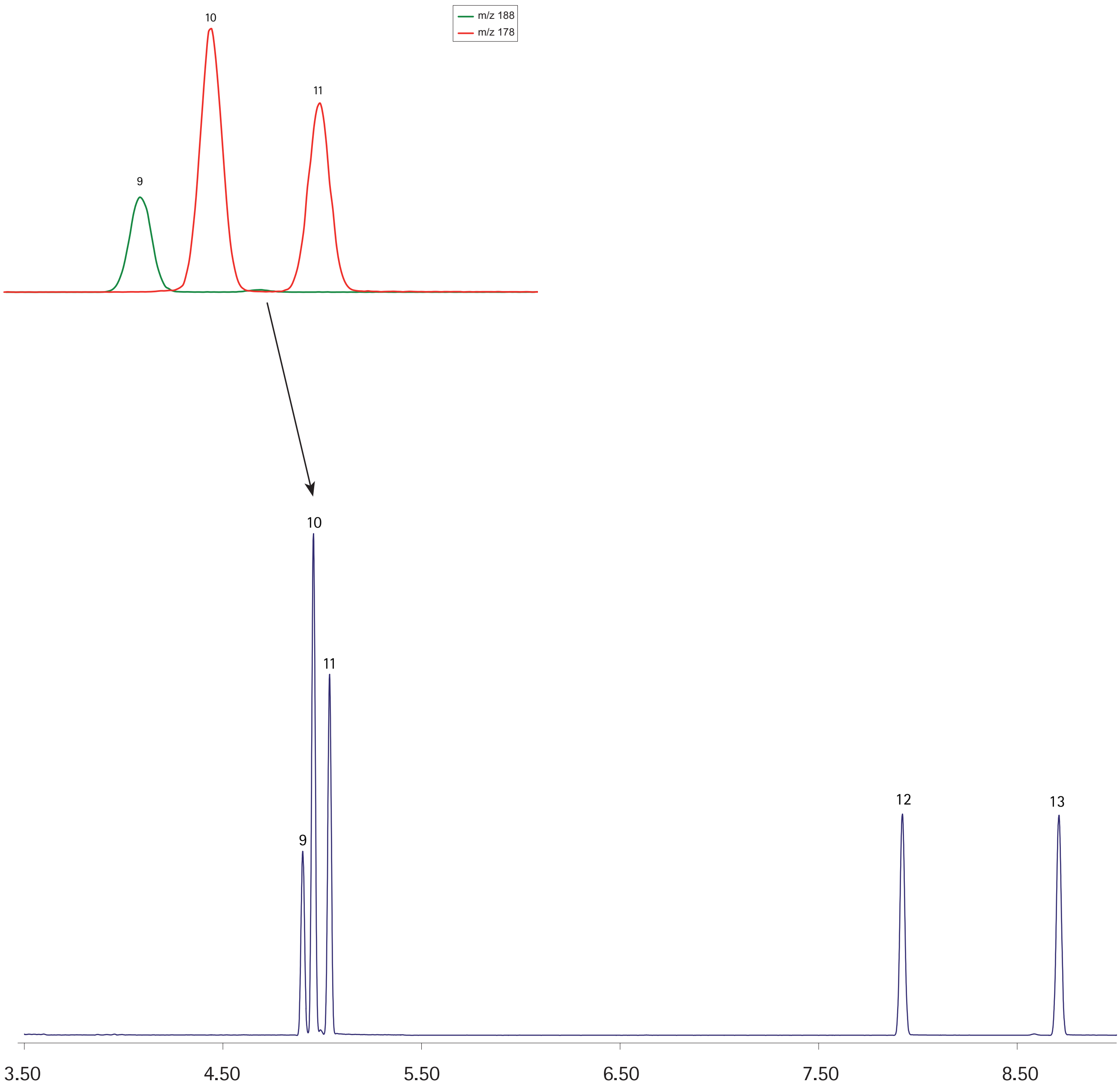

Figure 3. Details and identification of peaks 9 to 13

Table 4. Peak Identification for Figure 4

Peak	MW	Compound	EPA 610	SFC & EFSA PAHs (15+1)	CAS
14	216	Benzo[a]fluorene			238-84-6
15	216	Benzo[b]fluorene			243-17-4
16	216	7H-Benzo[c]fluorene		x	205-12-9
17	234	Benzo[b]naphtho[2,1-d] thiophene			239-35-0
18	226	Benzo[g,h,i]fluoranthene			203-12-3
19	228	Benzo[c]phenanthrene			195-19-7
20	228	Benz[a]anthracene	x	x	56-55-3
21	226	Cyclopenta[c,d]pyrene		x	27208-37-3
22	240	Chrysene-d10			1719-03-5
23	228	Triphenylene			217-59-4
24	228	Chrysene	x	x	218-01-9

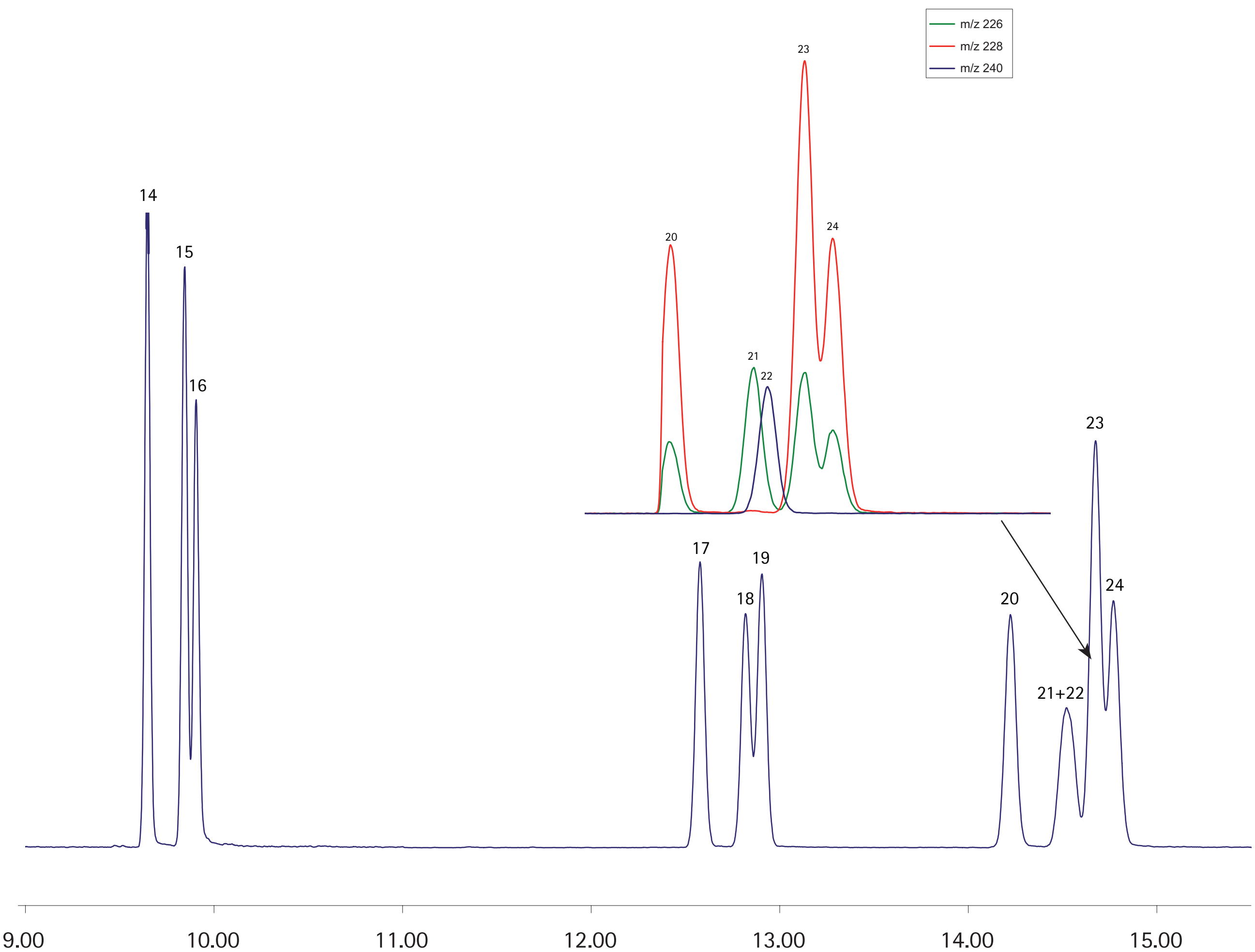

Figure 4. Details and identification of peaks 14 to 24

Table 5. Peak Identification for Figure 5

Peak	MW	Compound	EPA 610	SFC & EFSA PAHs (15+1)	CAS
25	242	6-Methylchrysene			1705-85-7
26	242	5-Methylchrysene		x	3697-24-3
27	252	Benzo[b]fluoranthene	x	x	205-99-2
28	252	Benzo[k]fluoranthene	x	x	207-08-9
29	252	Benzo[j]fluoranthene		x	205-82-3
30	252	Benzo[a]fluoranthene			203-33-8
31	252	Benzo[e]pyrene			192-97-2
32	252	Benzo[a]pyrene	x	x	50-32-8
33	264	Perylene-d12			1520-96-3
34	252	Perylene			198-55-0
35	268	3-Methylcholanthrene			56-49-5
36	330	9,10-diphenylanthracene			216-105-1

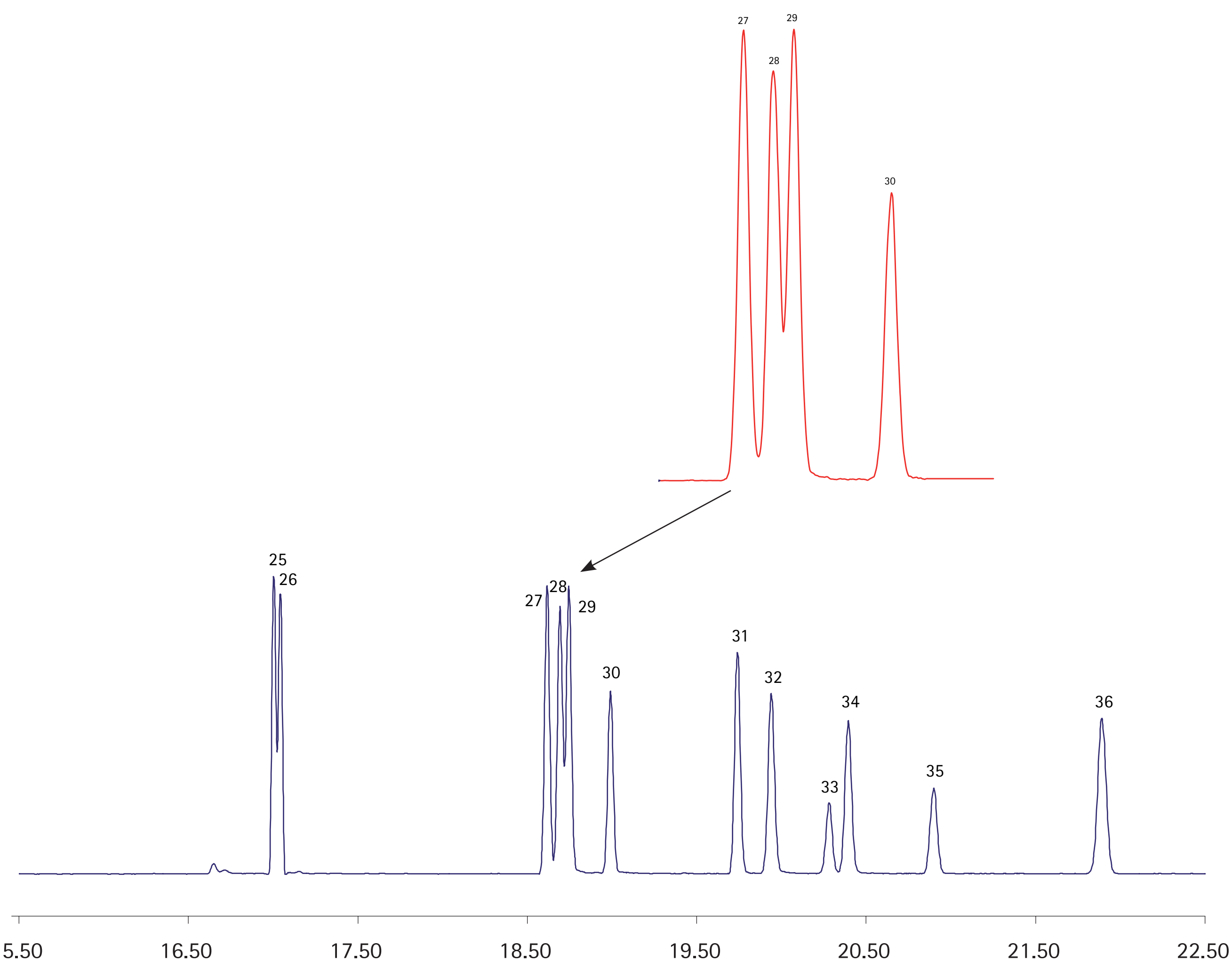

Figure 5. Details and identification of peaks 25 to 36

Table 6. Peak Identification for Figure 6

Peak	MW	Compound	EPA 610	SFC & EFSA PAHs (15+1)	CAS
37	279	Dibenzo[a,h]acridine			226-36-8
38	279	Dibenzo[a,j]acridine			224-42-0
39	278	Dibenz[a,j]anthracene			224-41-9
40	292	Dibenzo[a,h]anthracene D14			13250-98-1
41	278	Benzo[b]triphenylene			215-58-7
42	276	Indeno[1,2,3-c,d]pyrene	x	x	193-39-5
43	278	Dibenz[a,h]anthracene	x	x	53-70-3
44	278	Benzo[b]chrysene			214-17-5
45	278	Picene			213-46-7
46	27	Benzo[g,h,i]perylene	x	x	191-24-2
47	276	Dibenzo[def,mno]chrysene			191-26-4
48	267	7H-Dibenzo[c,g]carbazole			194-59-2
49	302	Dibenzo[a,l]pyrene		x	191-30-0
50	302	Dibenzo[a,e]pyrene		x	192-65-4
51	300	Coronene			191-07-1
52	302	Benzo[b]perylene			197-70-6
53	302	Dibenzo[a,i]pyrene		x	189-55-9
54	302	Dibenzo[a,h]pyrene		x	189-64-0

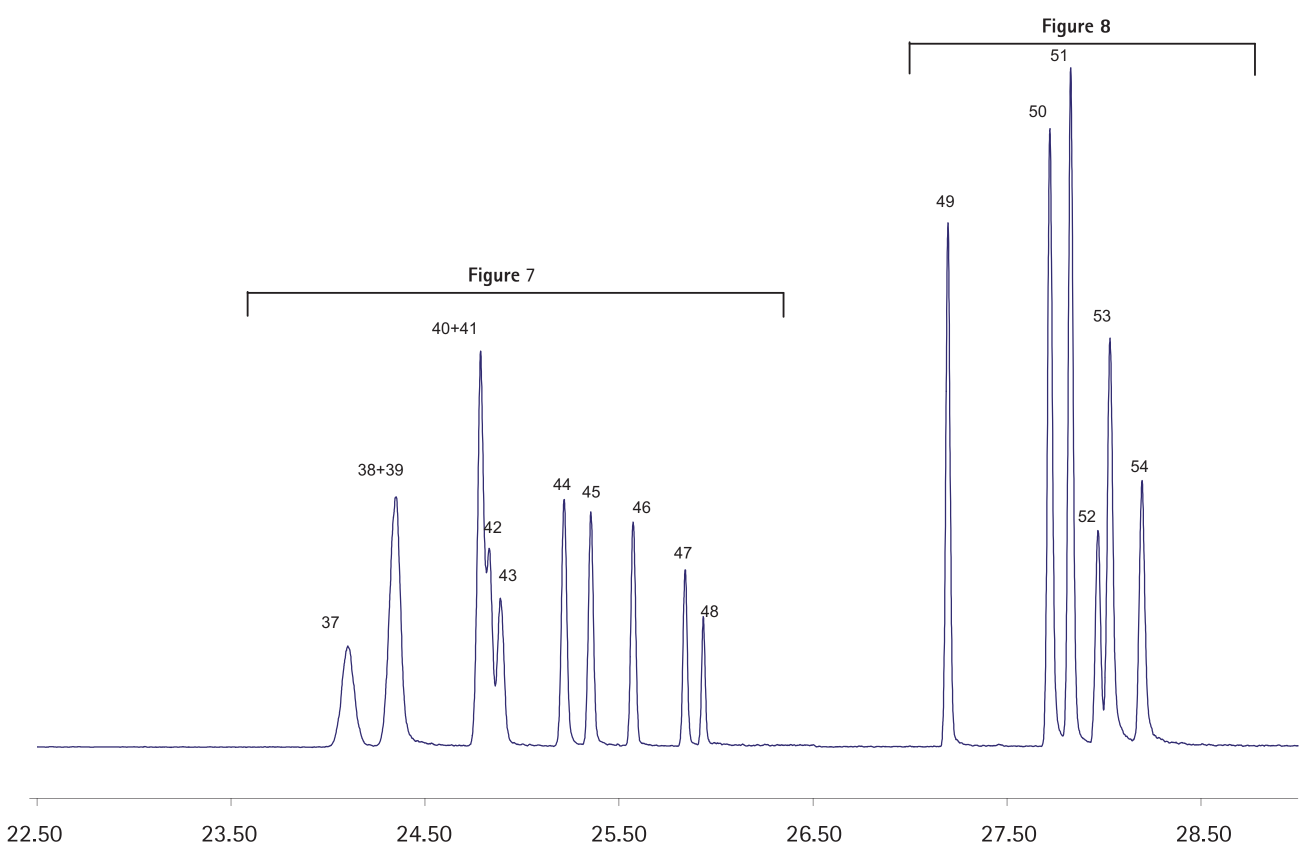

Figure 6. Details and identification of peaks 37 to 54

Table 7. Peak Identification for Figure 7

Peak	MW	Compound	EPA 610	SFC & EFSA PAHs (15+1)	CAS
37	279	Dibenzo[a,h]acridine			226-36-8
38	279	Dibenzo[a,j]acridine			224-42-0
39	278	Dibenz[a,j]anthracene			224-41-9
40	292	Dibenzo[a,h]anthracene D14			13250-98-1
41	278	Benzo[b]triphenylene			215-58-7
42	276	Indeno[1,2,3-c,d]pyrene	x	x	193-39-5
43	278	Dibenz[a,h]anthracene	x	x	53-70-3
44	278	Benzo[b]chrysene			214-17-5
45	278	Picene			213-46-7
46	27	Benzo[g,h,i]perylene	x	x	191-24-2
47	276	Dibenzo[def,mno]chrysene			191-26-4
48	267	7H-Dibenzo[c,g]carbazole			194-59-2

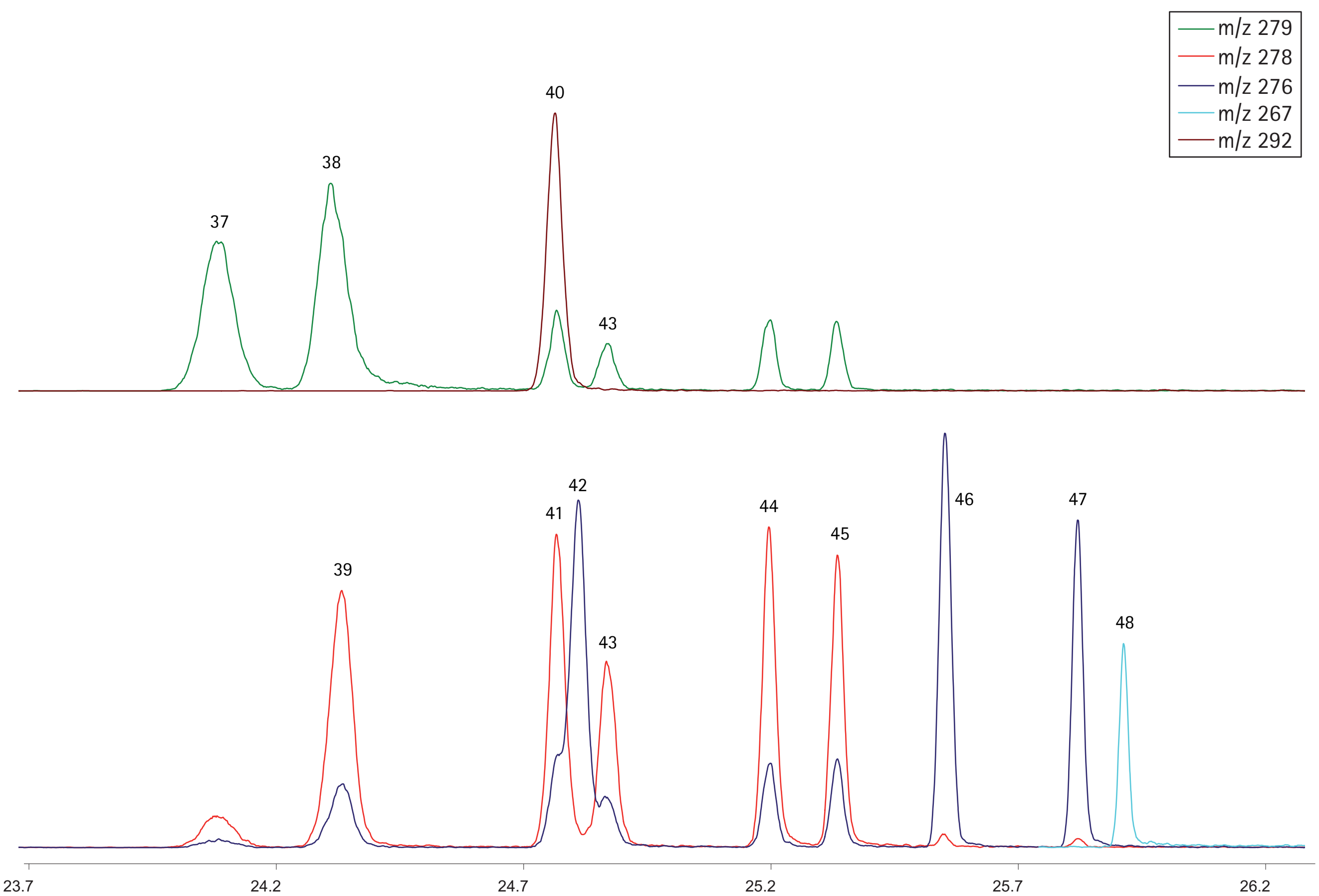

Figure 7. Details and identification of peaks 37 to 48

Table 8. Peak Identification for Figure 8

Peak	MW	Compound	EPA 610	SFC & EFSA PAHs (15+1)	CAS
49	302	Dibenzo[a,l]pyrene		x	191-30-0
50	302	Dibenzo[a,e]pyrene		x	192-65-4
51	300	Coronene			191-07-1
52	302	Benzo[b]perylene			197-70-6
53	302	Dibenzo[a,i]pyrene		x	189-55-9
54	302	Dibenzo[a,h]pyrene		x	189-64-0

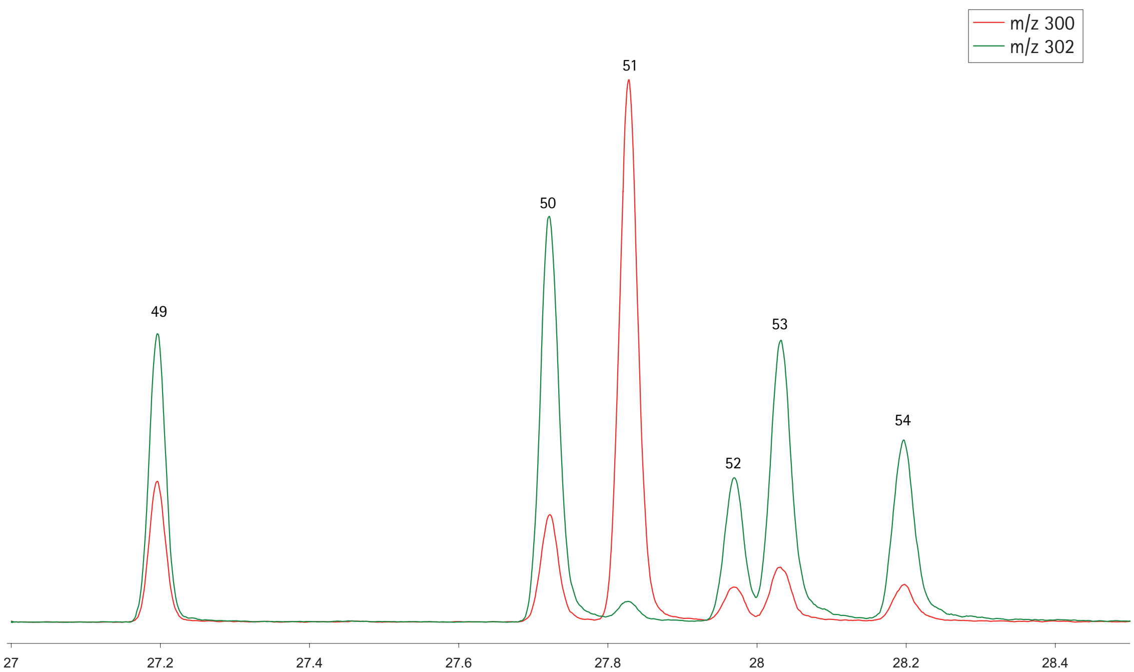

Figure 8. Details and identification of peaks 49 to 54

Conclusion

The J&W Select PAH column phase, as well as the GC oven program, affects the separation of difficult to resolve PAH sets. With the optimized oven program described in this application note, all EU and EPA PAHs, and known interferences, are resolved in a single run with a runtime of 29 min.

References

Ziegenhals K et al., (2008) Fast-GC/HRMS to quantify the EU priority PAH, J. Sep. Sci., 31, 1779-1786.

Anon, (2005) Report Joint FAO/Who Expert Committee on Food Additives, Sixty-fourth meeting, Rome, 8-17 February 2005.

Bordajandi LR et al., (2008) Optimisation of the GC-MS conditions for the determination of the 15 EU foodstuff priority polycyclic aromatic hydrocarbons, J. Sep. Sci., 31, 1769-1778.

Lerda D, (2009) Polycyclic Aromatic Hydrocarbons (PAHs) Factsheet. European Commission, Joint Research Centre, Institute for Reference Materials and Measurements, JRC 500871.

Poster DL et al., (2006) Analysis of polycyclic aromatic hydrocarbons (PAHs) in environmental samples: a critical review of gas chromatographic (GC) methods. Anal. Bioanal. Chem, 386, 859-881.

www.agilent.com/chem

Published in UK, October 11, 2010

SI-02259

Agilent Technologies