Separation of 27 EU and US EPA Regulated PAHs on Agilent J&W Select PAH Columns

Application Note

Author

John Oostdijk
Agilent Technologies, Inc.

Introduction

The difficulty in analyzing polycyclic aromatic hydrocarbons (PAHs) lies in the number of PAHs with the same mass. This makes their separation rather difficult using GC/MS, and so careful column selection together with an optimized oven program are necessary. We describe here an optimized oven program for the Select PAH column.

PAHs are compounds containing two or more aromatic rings. They are formed during incomplete combustion or pyrolysis of organic matter, industrial processes, and cooking and food processing. PAHs are therefore analyzed in environmental and food samples.

In PAH analysis, there is a difference between the European (EU) and American (EPA 610) legislation. The legislations both describe a different set of PAHs (Table 1). In particular, the EPA list is used for environmental samples, while the EU PAHs are analyzed in food samples. This application note demonstrates a GC/MS method that resolves all EU and EPA PAHs, and known interferences, with the Select PAH column (Figure 1).

Agilent Technologies

Conditions

Technique:	GC/MS, Triple Quad
Column:	Select PAH, 30 m x 0.25 mm, df = 0.15 µm (part number CP7462)
Sample:	Mixture of 27 PAHs, concentration approx. 1 µg/mL
Injection Volume:	1 µL
Temperature:	70 °C (0.70 min), 85 °C/min, 180 °C, 3 °C/min, 230 °C (7 min), 28 °C/min, 280 °C (10 min), 14 °C/min, 350 °C (3 min)
Carrier Gas:	Helium, constant flow 2 mL/min
Injector:	300 °C, Splitless mode, 1 min @ 50 mL/min
Detector:	Triple Quad, EI in SIM mode, ion source 275 °C, transfer line 300 °C

Results and Discussion

When performing this analysis, there are three sets of peaks that are difficult to resolve. The first set, benz[a]anthracene, cyclopenta[c,d]pyrene, chrysene and triphenylene, have different masses, m/z 226 and 228. The compounds with m/z 228 also contain some m/z 226 and this makes it difficult to resolve this set with MS alone. The same problem occurs when separating indeno[1,2,3-cd]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. These three isomers, benzo[b] fluoranthene, benzo[j]fluoranthene and benzo[k]fluoranthene, have the same mass and cannot be resolved with MS alone. For these reasons, the column has to resolve PAH isomers chromatographically, which is shown in Figures 1-4.

Table 1. Peak identification for Figure 1

Peak	MW	Compound	EPA 610	SFC[1] & EFSA[2] PAHs (15+1)	CAS
1	128	Naphthalene	x		91-20-3
2	152	Acenaphthylene	x		208-96-8
3	154	Acenaphthene	x		83-32-9
4	166	Fluorene	x		86-73-7
5	178	Phenanthrene	x		85-01-8
6	178	Anthracene	x		120-12-7
7	202	Fluoranthene	x		206-44-0
8	202	Pyrene	x		129-00-0
9	216	7H-Benzo[c]fluorene		x	205-12-9
10	228	Benzo[a]anthracene	x	x	56-55-3
11	226	Cyclopenta[c,d]pyrene		x	27208-37-3
12	228	Triphenylene			217-59-4
13	228	Chrysene	x	x	218-01-9
14	242	6-Methylchrysene			1705-85-7
15	242	5-Methylchrysene		x	3697-24-3
16	252	Benzo[b]fluoranthene	x	x	205-99-2
17	252	Benzo[k]fluoranthene	x	x	207-08-9
18	252	Benzo[j]fluoranthene		x	205-82-3
19	252	Benzo[a]pyrene	x	x	50-32-8
20	278	Benzo[b]triphenylene			215-58-7
21	276	Indeno[1,2,3-c,d]pyrene	x	x	193-39-5
22	278	Dibenz[a,h]anthracene	x	x	53-70-3
23	276	Benzo[g,h,i]perylene	x	x	191-24-2
24	302	Dibenzo[a,l]pyrene		x	191-30-0
25	302	Dibenzo[a,e]pyrene		x	192-65-4
26	302	Dibenzo[a,i]pyrene		x	189-55-9
27	302	Dibenzo[a,h]pyrene		x	189-64-0

[1]Scientific Committee on Food, one of the committees providing the European Commission with scientific advice on food safety

[2]European Food Safety Authority

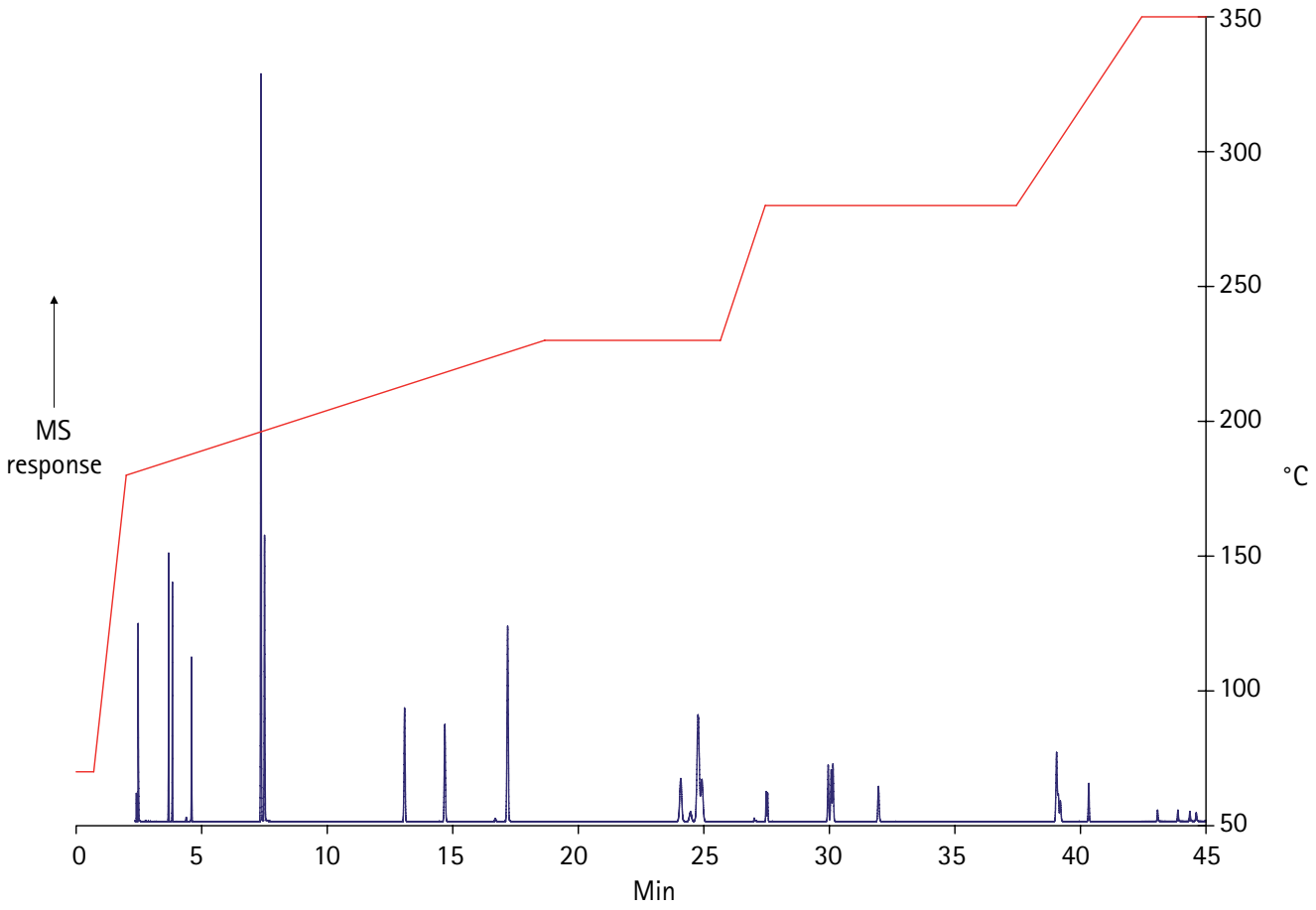

Figure 1. GC/MS analysis of 27 EU and EPA PAHs

Table 2. Peak Identification for Figure 2

Peak	MW	Compound	EPA 610	SFC & EFSA PAHs (15+1)	CAS
1	128	Naphthalene	x		91-20-3
2	152	Acenaphthylene	x		208-96-8
3	154	Acenaphthene	x		83-32-9
4	166	Fluorene	x		86-73-7
5	178	Phenanthrene	x		85-01-8
6	178	Anthracene	x		120-12-7
7	202	Fluoranthene	x		206-44-0
8	202	Pyrene	x		129-00-0
9	216	7H-Benzo[c] fluorene		x	205-12-9

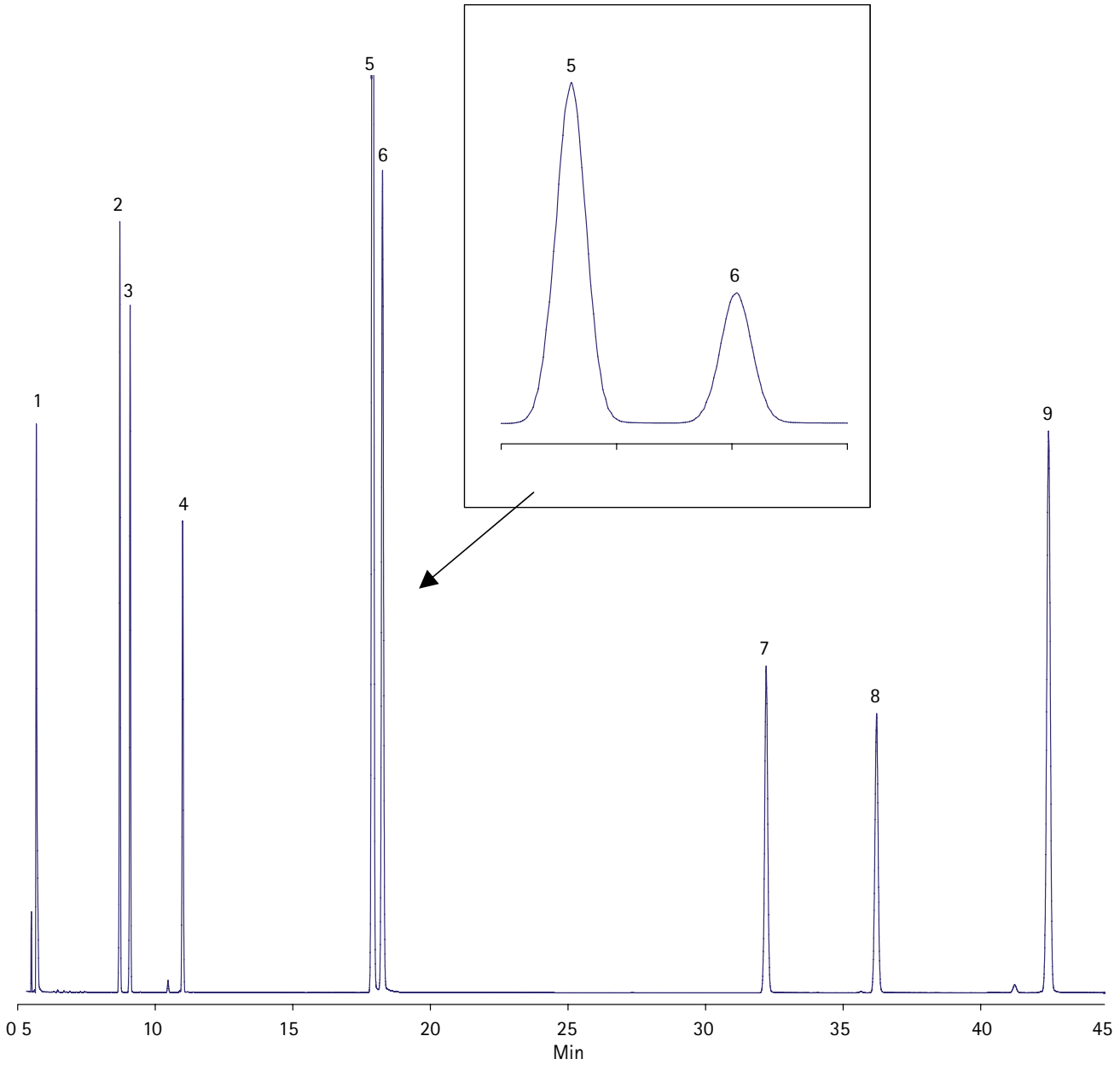

Figure 2. Details and identification of peak numbers 1 to 9

Table 3. Peak Identification for Figure 3

Peak	MW	Compound	EPA 610	SFC & EFSA PAHs (15+1)	CAS
10	228	Benzo[a]anthracene	x	x	56-55-3
11	226	Cyclopenta[c,d]pyrene		x	27208-37-3
12	228	Triphenylene			217-59-4
13	228	Chrysene	x	x	218-01-9
14	242	6-Methylchrysene			1705-85-7

Peak	MW	Compound	EPA 610	SFC & EFSA PAHs (15+1)	CAS
15	242	5-Methylchrysene		x	3697-24-3
16	252	Benzo[b]fluoranthene	x	x	205-99-2
17	252	Benzo[k]fluoranthene	x	x	207-08-9
18	252	Benzo[j]fluoranthene		x	205-82-3
19	252	Benzo[a]pyrene	x	x	50-32-8

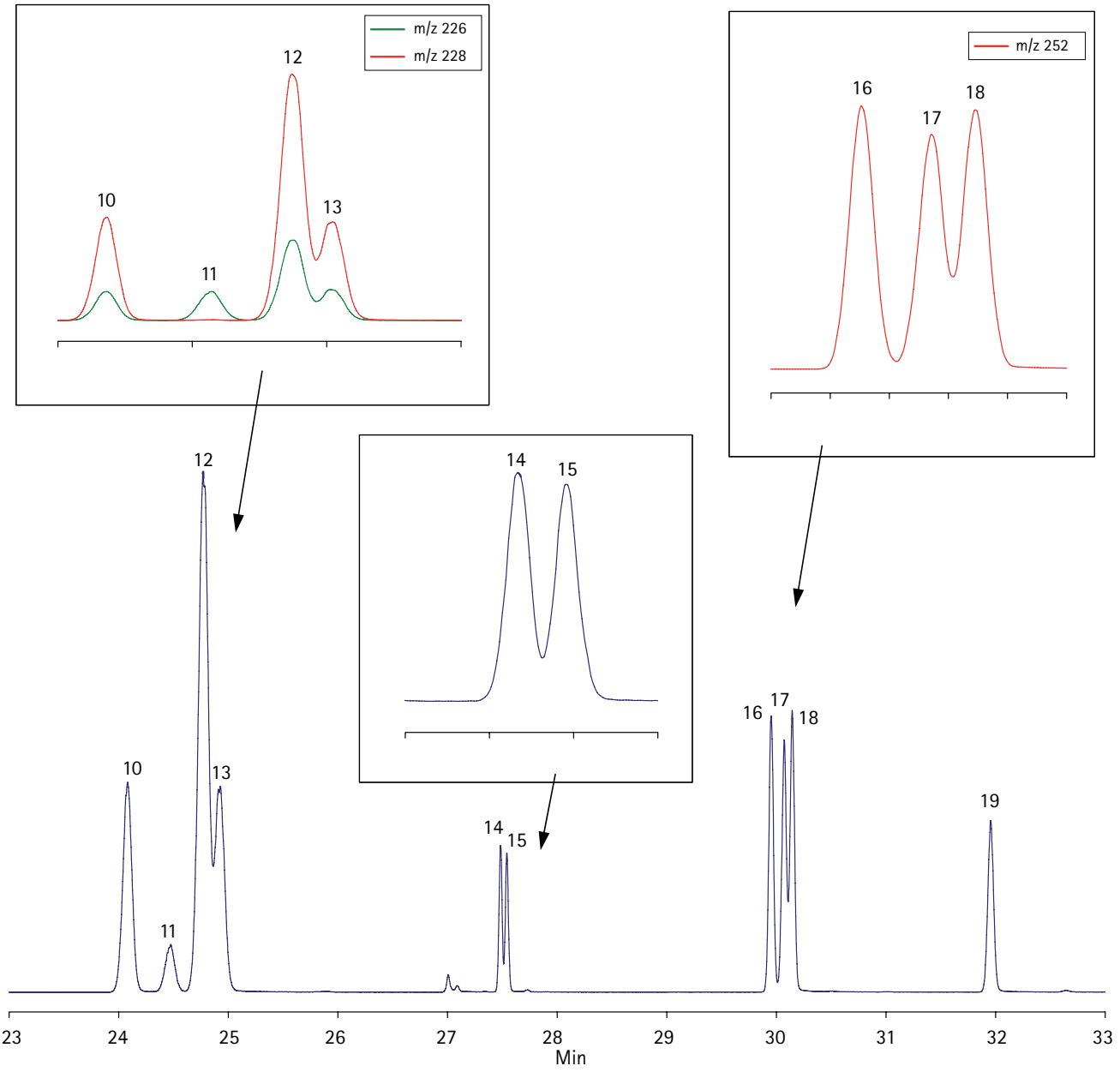

Figure 3. Details and identification of peak numbers 10 to 19

Table 4. Peak Identification for Figure 4

Peak	MW	Compound	EPA 610	SFC & EFSA PAHs (15+1)	CAS
20	278	Benzo[b]triphenylene			215-58-7
21	276	Indeno[1,2,3-c,d]pyrene	x	x	193-39-5
22	278	Dibenz[a,h]anthracene	x	x	53-70-3
23	276	Benzo[g,h,i]perylene	x	x	191-24-2
24	302	Dibenzo[a,l]pyrene		x	191-30-0
25	302	Dibenzo[a,e]pyrene		x	192-65-4
26	302	Dibenzo[a,i]pyrene		x	189-55-9
27	302	Dibenzo[a,h]pyrene		x	189-64-0

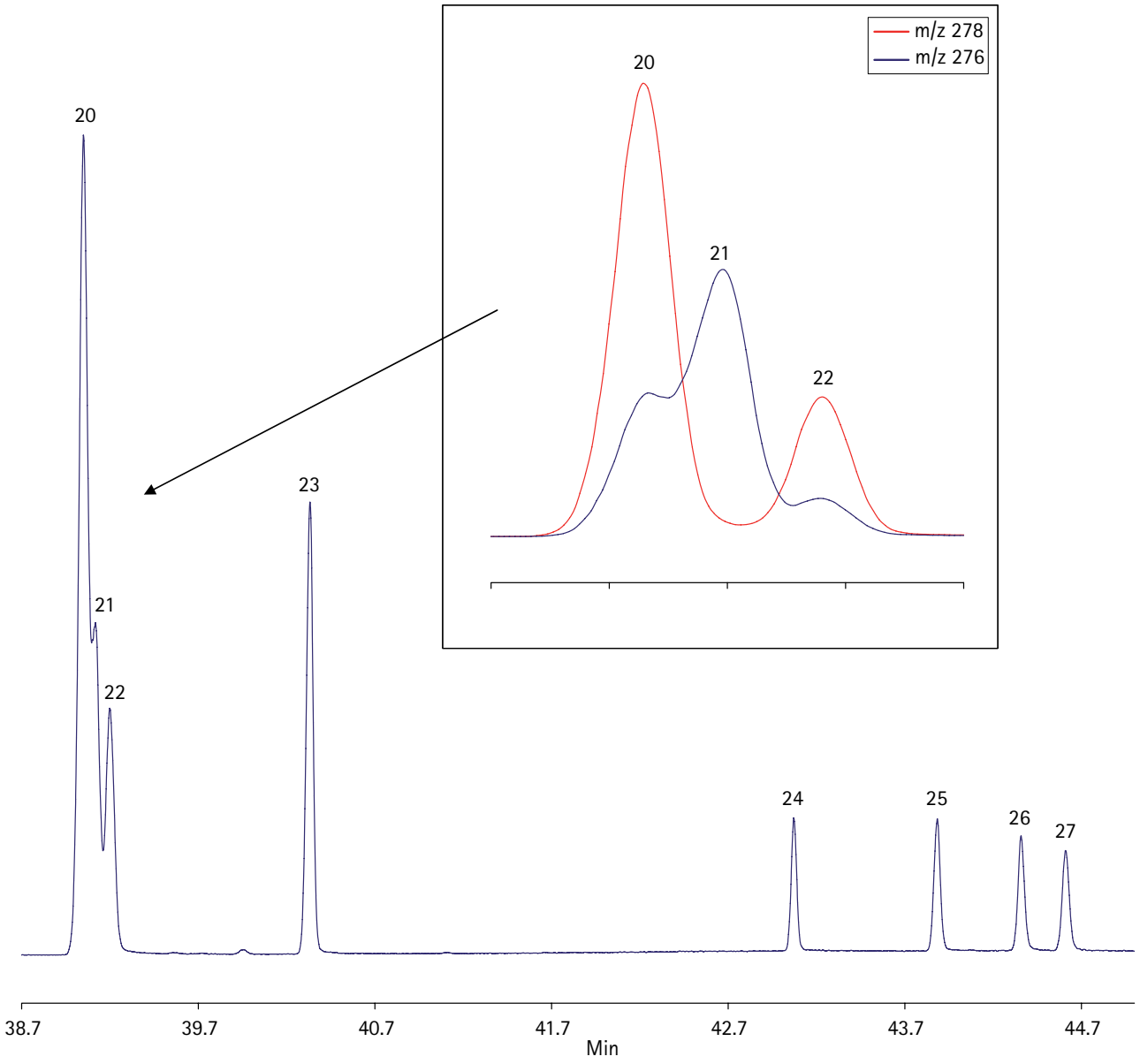

Figure 4. Details and identification of peaks 20 to 27

Conclusion

The Select PAH column phase, as well as the GC oven program, affect the separation of the difficult to resolve PAH sets. With the optimized oven program described here, all EU and EPA PAHs are resolved in a single run with a runtime of 45 min.

References

Anon (2005) Report JOINT FAO/WHO EXPERT COMMITTEE ON FOOD ADDITIVES, Sixty-fourth meeting, 8-17 February 2005. FAO/WHO, Rome, Italy.

Bordajandi LR, Dabrio M, Ulberth F and Emons H, (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. JRC 500871. European Commission, Joint Research Centre, Institute for Reference Materials and Measurements, Geel, Belgium.

Poster DL, Schantz MM, Sander LC and Wise SA, (2006) Analysis of polycyclic aromatic hydrocarbons (PAHs) in environmental samples: a critical review of gas chromatographic (GC) methods. Anal. Bioanal. Chem, 386, 859-881.

Ziegenhals K, Hubschmann HJ, Speer K, and Jira W, (2008) Fast-GC/HRMS to quantify the EU priority PAH. J. Sep. Sci., 31, 1779-1786.

www.agilent.com/chem

Published in UK, October 11, 2010

SI-02281

Agilent Technologies