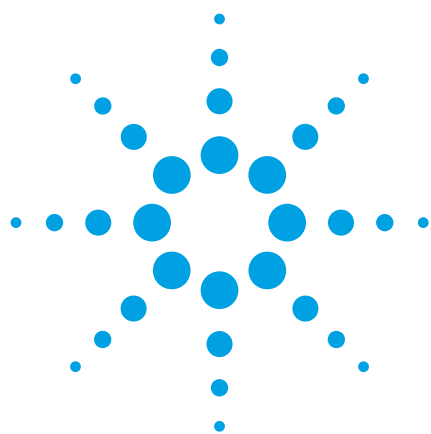




Fast Separation of 27 EU and US EPA Regulated PAHs on Agilent J&W Select PAH

Application Note

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Introduction

Polycyclic aromatic hydrocarbons (PAHs) are formed during incomplete combustion or pyrolysis of organic matter, industrial processes and cooking, and food processing. Due to their carcinogenic properties, they need to be analyzed in environmental and food samples. However, several PAHs are difficult to determine with GC/MS because they have the same mass, and co-elute. Enhanced column selectivity and an optimized oven program are necessary to resolve these PAHs, as described in this application note.

There is a difference between the European (EU) and American (EPA 610) legislation in the analysis of PAHs, with different sets of PAHs being targeted (Table 1). The EPA list is particularly used for environmental samples and the EU list for food samples. This application note describes a GC/MS method that resolves all EU and EPA PAHs, and their known interferences, with the Select PAH column (Figure 1).



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Conditions

Technique: GC/MS, Triple Quad
 Column: Select PAH, 15 m x 0.15 mm, df = 0.10 µm (part number CP7461)
 Sample: Mixture of 27 PAHs, concentration approx 1 µg/mL
 Injection Volume: 1.0 µL
 Temperature: 70 °C (0.40 min), 70 °C/min, 180 °C
 Program: 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
 Injection: 300 °C, Splitless mode, 0.5 min @ 100 mL/min
 Detection: Triple Quad, EI in SIM mode, ion source 275 °C, transfer line 300 °C

Results and Discussion

In PAH analysis there are three sets of peaks that are difficult to resolve because of similarities in compound mass. The first of these, comprising benz[a]anthracene, cyclopenta[c,d]pyrene, chrysene and triphenylene, has different masses of m/z 226 and 228. However, compounds with m/z 228 also contain some m/z 226, and this makes their resolution problematic by MS alone. The same issue occurs when separating indeno[1,2,3-c,d]pyrene, benzo[b]triphenylene and dibenz[a,h]anthracene, with m/z 276 and 278. Benzofluoranthene isomers are the third set of difficult-to-resolve PAHs, containing three isomers, benzo[b]fluoranthene, benzo[j]fluoranthene and benzo[k]fluoranthene. They have the same mass and cannot be resolved with MS only. Nonetheless, these isomers can be resolved chromatographically by the Select PAH GC column, as shown in the figures presented here.

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

Peak	MW	Compound	EPA 610	SFC ¹ & EFSA ² PAHs (15+1)	CAS
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

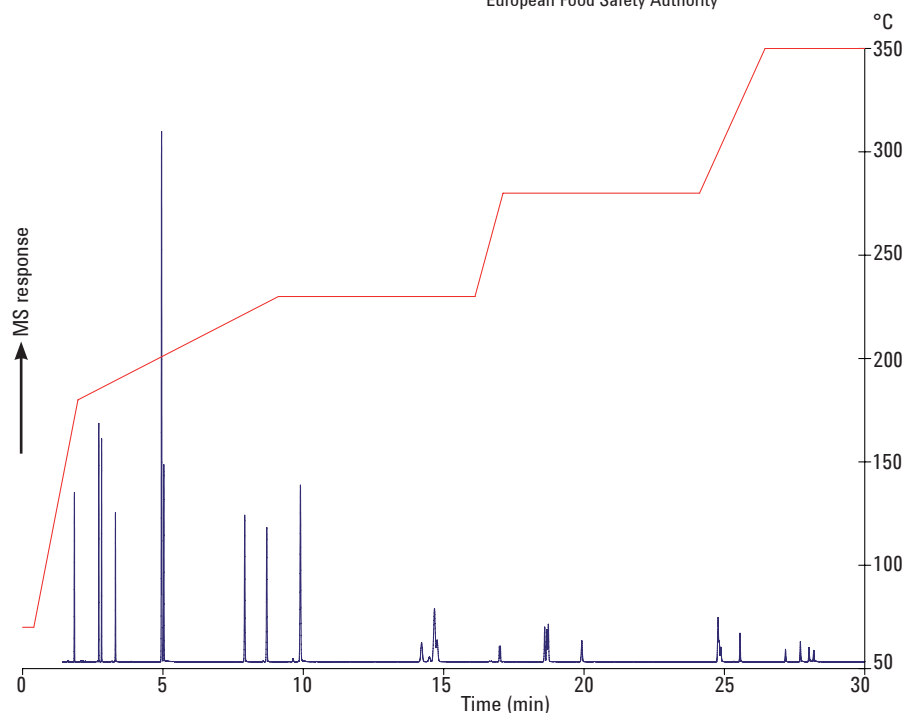


Figure 1. GC/MS analysis of 27 EU and EPA PAHs on Select PAH 15 m x 0.15 mm x 0.10 µm

Table 2. Peak Identification for Figure 2

Peak	MW	Compound	EPA 610	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

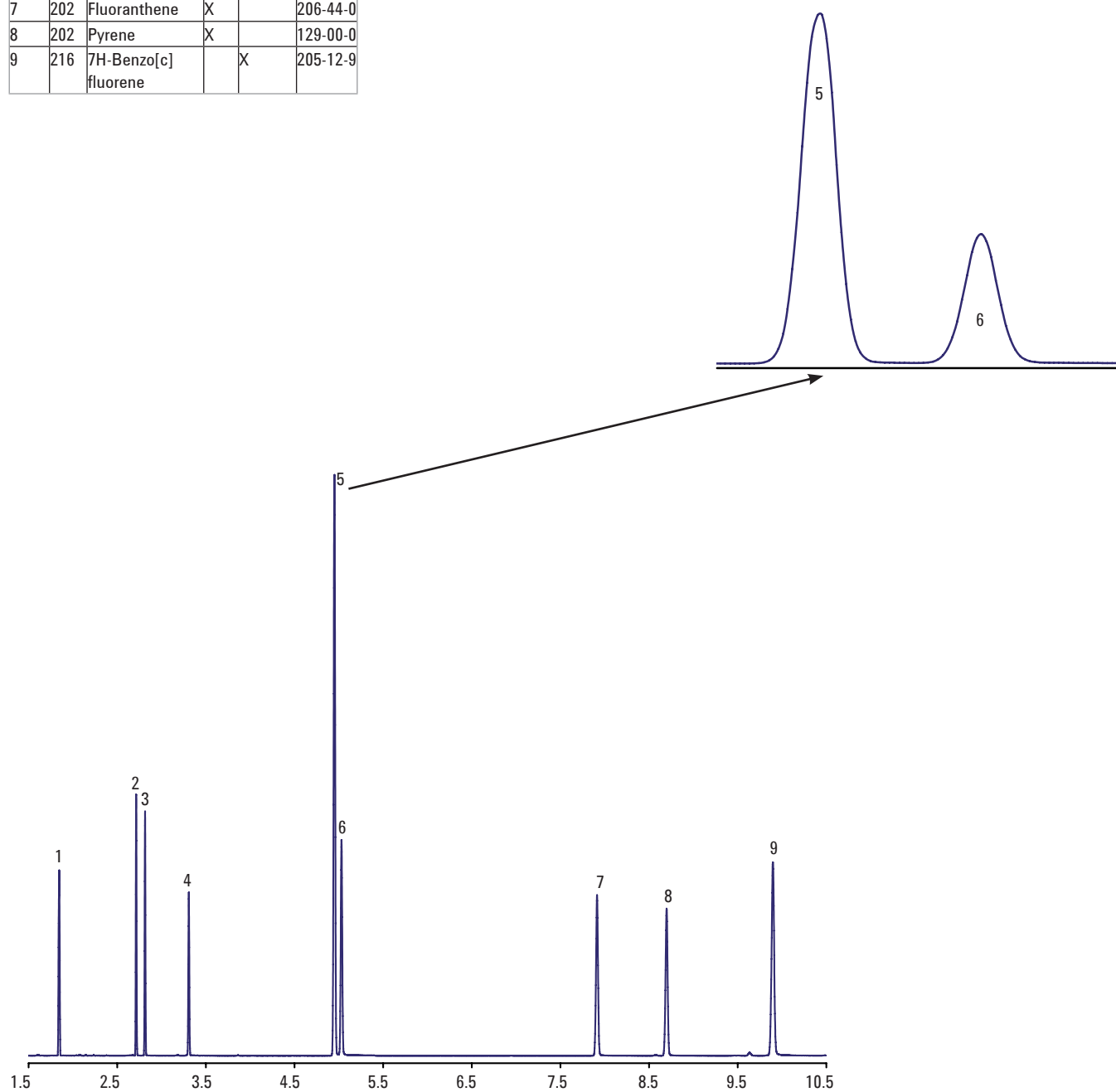


Figure 2. Details and identification of peaks 1 to 9

Table 3. Peak Identification for Figure 3

Peak	MW	Compound	EPA 610	SFC and EFSA PAHs (15+1)	CAS
10	188	Benzo[a]anthracene	X	X	56-55-3
11	178	Cyclopenta[c,d]pyrene		X	27208-37-3
12	178	Triphenylene			217-59-4
13	202	Chrysene	X	X	218-01-9
14	202	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

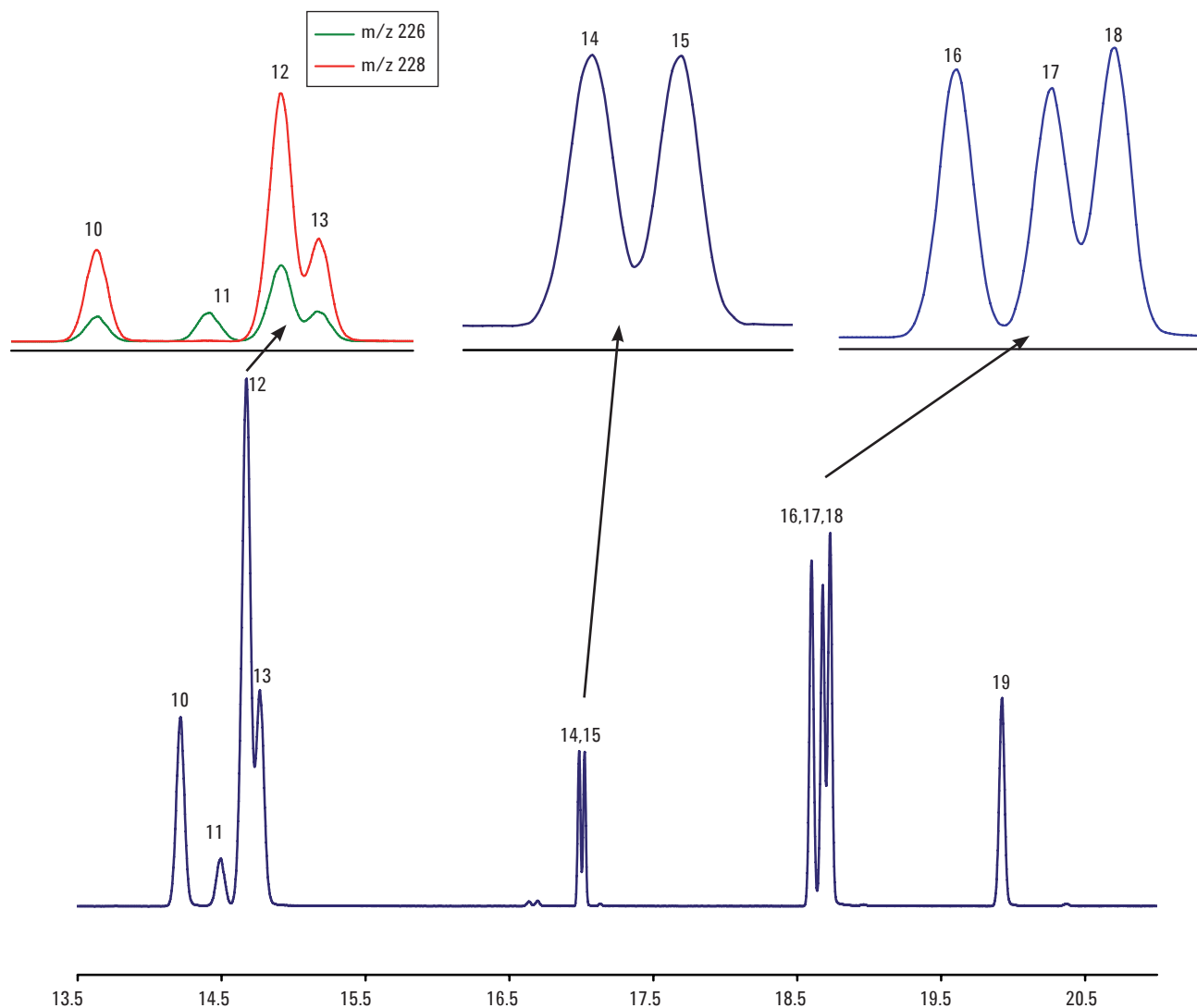


Figure 3. Details and identification of peaks 10 to 19

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

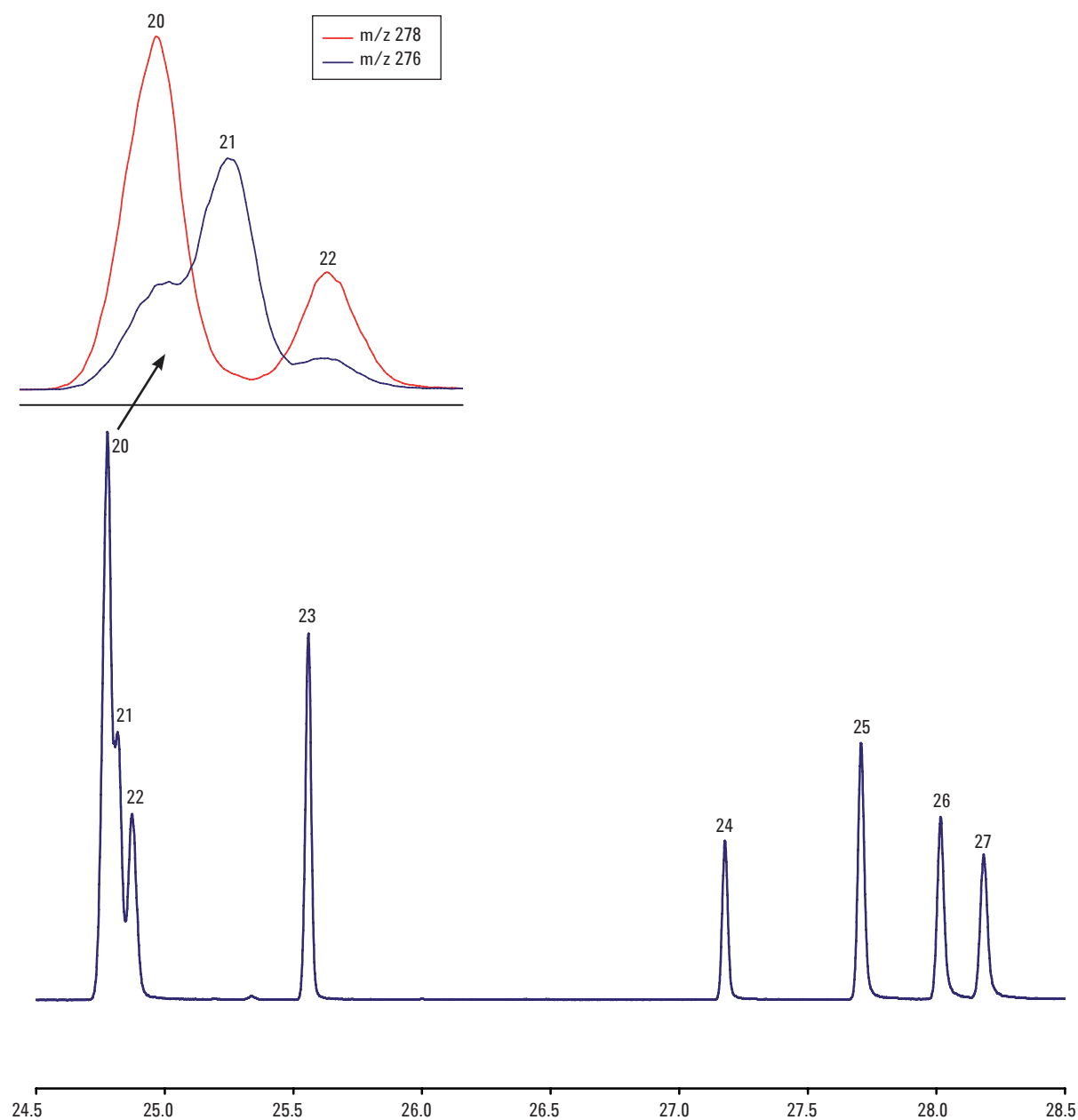


Figure 4. Details and identification of peaks 20 to 27

Conclusion

Using the Select PAH GC column with an optimized GC oven program resolves all 27 EPA and EU PAHs in a single run. The small dimensions of this column delivered a fast runtime of just 29 minutes, demonstrating a significant productivity advantage.

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.

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