

Separation of EU and EPA Regulated PAHs on Agilent J&W FactorFour VF-17ms Column

Application Note

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Introduction

The difficulty in analyzing polycyclic aromatic hydrocarbons (PAHs) is the number of PAHs with the same mass. This makes the separation of these PAHs with GC/MS rather difficult, and column selectivity and an optimized oven program are necessary to resolve these PAHs. In this application note an optimized oven program for the FactorFour VF-17ms column is described.

PAHs are compounds which contain 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 both environmental and food related matrices.

In PAH analysis there is a difference between the European (EU) and American (EPA) legislation. These regulatory bodies both describe a different set of PAHs (Table 1). The EPA list is especially used for environmental samples while the EU PAHs are analyzed in food samples. This application note shows a GC/MS method which resolves all EU and EPA PAHs with the VF-17ms column (Figure 1).



Conditions		Tabl
Technique:	GC/MS	
Column:	VF-17ms, 30 m x 0.25 mm, df = 0.15 µm (part number	Pea
	CP8981)	1
Temperature:	70 °C (1 min), 40 °C/min, 180	-
	°C, 2.5 °C/ min, 245 °C, 2.0	2
	°C/min, 270 °C (10 min), 8.0	3
	°C/min, 350 °C (5 min)	4
Carrier Gas:	Helium, constant flow 1 mL/	. <u>.</u>
	min	5
Injector:	300 °C, Splitless mode	6
Detector:	Quadrupole MS, El in SIM, ion source 275 °C, transfer	7
	line 300 °C	8
Sample:	Concentration approx. 1 µg/	-
Sample.	mL	9
Injection Volume:	πc 1 μL	10
injection volumo.	· F=	11

Table 1. Peak Identification for Figure 1

Peak	Peak Compound		EU PAH	EPA PAH
1	Naphthalene	128		Х
2	Acenaphthylene	152		Х
3	Acenaphthene	154		Х
4	Fluorene	166		Х
5	Phenanthrene	178		Х
6	Anthracene	178		Х
7	Fluoranthene	202		Х
8	Pyrene	202		Х
9	Benzo(c)fluorene	216	Х	
10	Benz(a)anthracene	228	Х	Х
11	Cyclopenta(c,d) pyrene	226	x	
12	Triphenylene	228		
13	Chrysene	228	Х	Х
14	6-Methylchrysene	242		
15	5-Methylchrysene	242	Х	

Peak	Compound	мw	EU PAH	EPA PAH
16	Benzo(b) fluoranthene	252	X	Х
17	Benzo(k) fluoranthene	252	X	Х
18	Benzo(j)fluoranthene	252	Х	
19	Benzo(a)pyrene	252	Х	Х
20	Indeno(1,2,3-cd) pyrene	276	X	Х
21	Benzo(b) triphenylene	278		
22	Dibenz(a,h) anthracene	278	X	Х
23	Benzo(g,h,i)perylene	276	Х	Х
24	Dibenzo(a,l)pyrene	302	х	
25	Dibenzo(a,e)pyrene	302	Х	
26	Dibenzo(a,i)pyrene	302	Х	
27	Dibenzo(a,h)pyrene	302	x	

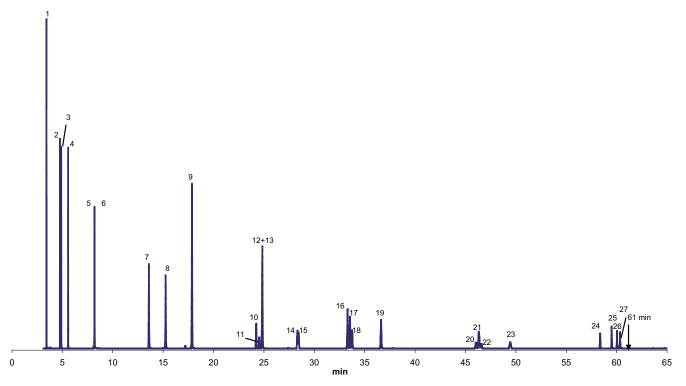


Figure 1. GC/MS analysis of EU and EPA PAHs on VF-17ms 30 m x 0.25 mm x 0.15 μm

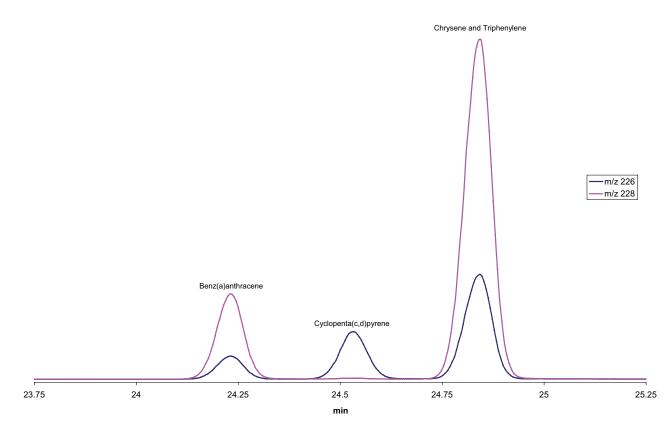


Figure 2. Separation of m/z 226 and 228

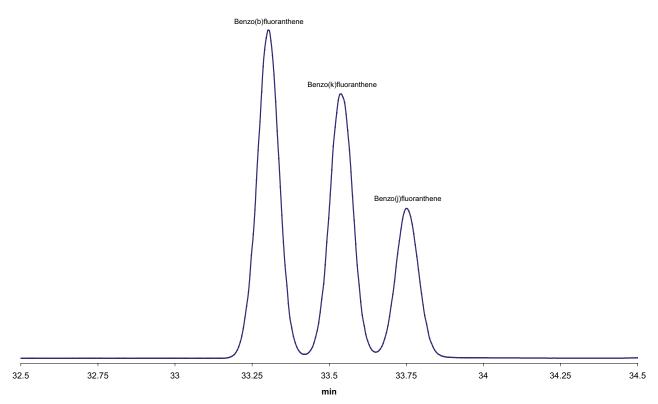


Figure 3. Separation of Benzofluoranthenes (m/z 252)

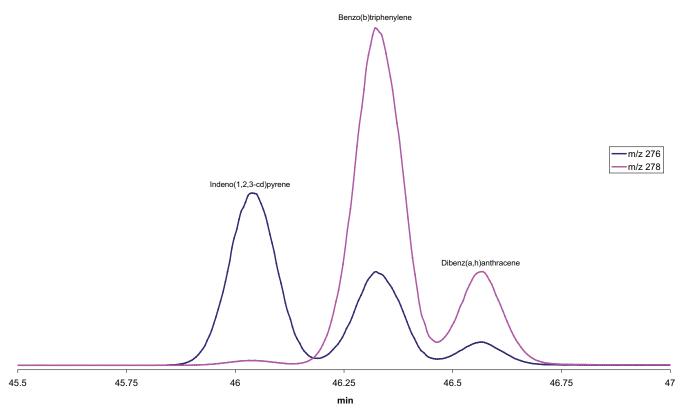


Figure 4. Separation of m/z 276 and 278

Results and Discussion

When performing this analysis there are 3 sets of peaks which are difficult to resolve. The first set, Benz(a) anthracene, Cyclopenta(c,d)pyrene, Chrysene and Triphenylene, has 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 only with MS (Figure 2). The same problem occurs when separating Indendo(1,2,3cd)pyrene, Benzo(b)triphenylene and Dibenz(a,h)anthracene with m/z 276 and 278 (Figure 4).

The third set of PAHs which are difficult to resolve are the Benzofluoranthene isomers. These three isomers, Benzo(b) fluoranthene, Benzo(j)fluoranthene and Benzo(k)fluoranthene, have the same mass and can't be resolved with MS only (Figure 3).

For the above reasons the column has to resolve these components chromatographically which is showed in the figures.

Conclusion

The VF-17ms 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 in this application note all EU and EPA PAHs are resolved. It should be taken in mind that Triphenylene can cause a false positive because of the co-elution with Chrysene.

References

Report Joint FAO/Who Expert Committee on Food Additives, Sixtyfourth meeting, Rome, 8-17 February 2005.

Polycyclic Aromatic Hydrocarbons (PAHs) factsheet, European Commission, Joint Research Centre, Institute for Reference Materials and Measurements.

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