

# Fast GC Analysis of EPA and EU Regulated PAHs

## Application Note

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### Introduction

After assessing 33 PAHs, the European Commission Scientific Committee on Food (SCF) in 2002 found that due to their toxicity, 15 of them were of major concern to human health and should therefore be monitored. The FAO/WHO Expert committee on Food Additives identified benzo(c)fluorine in 2005 as an additional compound that should be analyzed. These 15+1 EU priority PAHs differ from the 16 PAHs covered under the US EPA regulations.

With the increase of the number of target PAH analytes and the fact that many PAHs and their isomers exhibit identical MS mass fragmentation, the chromatographic separation and selectivity of the stationary phase for the analysis of PAHs has become more important.

This application note illustrates the successful use of an Agilent J&W FactorFour VF-17ms column from Agilent Technologies, Inc. for the fast GC/MS analysis of most PAHs listed in the EU and EPA regulations (Figure 1).



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## Conditions

Technique: GC/MS  
Column: VF-17ms 20 m x 0.15 mm df = 0.15 mm (p/n CP5884), retention gap 2.5 m x 0.53 mm (p/n CP8009)  
Temperature: 90 °C (1 min), 78 °C/min, 130 °C (0.4 min), 21 °C/min, 190 °C (1.90 min), 15.7 °C/min, 320 °C (12 min)  
Carrier Gas: Helium, constant flow 1 mL/min  
Injector: Splitless mode, pulsed pressure 50 psi for 0.2 min, 280 °C  
Detector: Quadrapole MS, EI in SIM, ion source 230 °C, transfer line 280 °C  
Sample: Concentration 5 ppm, isotope ISTDs 0.5 ppm, solvent toluene  
Injection Vol: 1 µL

## Results

Critical peak pairs such as the benzofluoranthene isomers (m/z 252) are almost baseline resolved (Figure 3). Although the VF-17ms stationary phase exhibits an excellent selectivity towards difficult to separate PAHs, the coelution of some peak pairs cannot be avoided. Indeno[1,2,3-cd]pyrene (m/z 276) and dibenzo[a,h]anthracene (m/z 278) coelute but can be distinguished by their difference in molecular masses (Figure 4). Crysene cannot be

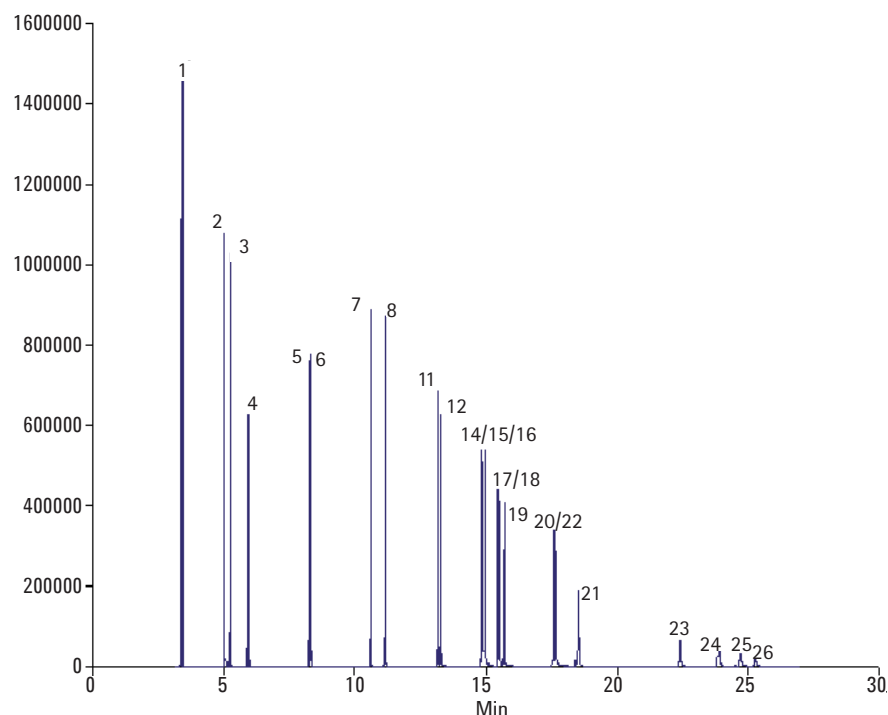
unambiguously quantified due to the possible coelution with triphenylene and identical quantifying ions (m/z 228), but is well resolved from benzo(a)anthracene (Figure 5). The four EU priority dibenzopyrenes (m/z 302) are well separated by VF-17ms (Figure 6). However, due to the large number of isomers with the same molecular mass, possible interferences may influence results.

The temperature program was carefully optimized to obtain optimal results in terms of separation and speed of analysis.

## Discussion

The VF-17ms column efficiently separates all 16 US regulated PAHs as well as the 15+1 EU priority PAHs. The 50% phenyl groups incorporated in the liquid phase provide additional selectivity based on  $\pi/\pi$  stacking interactions and electrostatic supported separation mechanisms. The smaller 0.15 mm ID column delivers fast analysis within 25 minutes without

compromizing the resolution efficiency needed for the separation of critical PAH peak pairs. Quantification of the 23 PAHs of this particular analysis is carried out by means of isotope dilution using deuterium labelled PAH internal standards. Perylene D12 is used as an additional standard to correct for response variations between standard and sample analysis due to matrix influences during injection.



### Peak Identification

	PAH	Abbreviation	MW	EU PAHs	EPA PAHs
1	Napthalene	Nap	128		x
2	Acenaphthylene	Acy	152		x
3	Acenaphthene	Acp	154		x
4	Fluorene	Flr	166		x
5	Phenanthrene	Phen	178	x	
6	Anthracene	Ant	178	x	
7	Fluoranthene	Flt	202		x
8	Pyrene	Pyr	202		x
9	Benzo[c]fluorene	B[c]F	216	x	
10	Cyclopenta[cd]pyrene	CCP	226	x	
11	Benzo[a]anthracene	BaA	228	x	x
12	Chrysene	Chr	228	x	x
13	5-Methylchrysene	5-MC	242	x	
14	Benzo[b]fluoranthene	BbF	252	x	x
15	Benzo[k]fluoranthene	BkF	252	x	x
16	Benzo[j]fluoranthene	BjF	252	x	
17	Benzo[e]pyrene	BeP	252		
18	Benzo[a]pyrene	BaP	252	x	x
19	Perylene	Per	252		
20	Indeno[1,2,3-cd]pyrene	IcdP	276	x	x
21	Benzo[ghi]perylene	BghiP	276	x	x
22	Dibenzo[a,h]anthracene	DahA	278	x	x
23	Dibenzo[a,i]pyrene	DaiP	302	x	
24	Dibenzo[a,j]pyrene	DajP	302	x	
25	Dibenzo[a,e]pyrene	DaeP	302	x	
26	Dibenzo[a,h]pyrene	DahP	302	x	

Figure 1. Fast GC/MS analysis of EU and EPA PAHs on VF-17ms 20 m x 0.15 mm x 0.15 µm

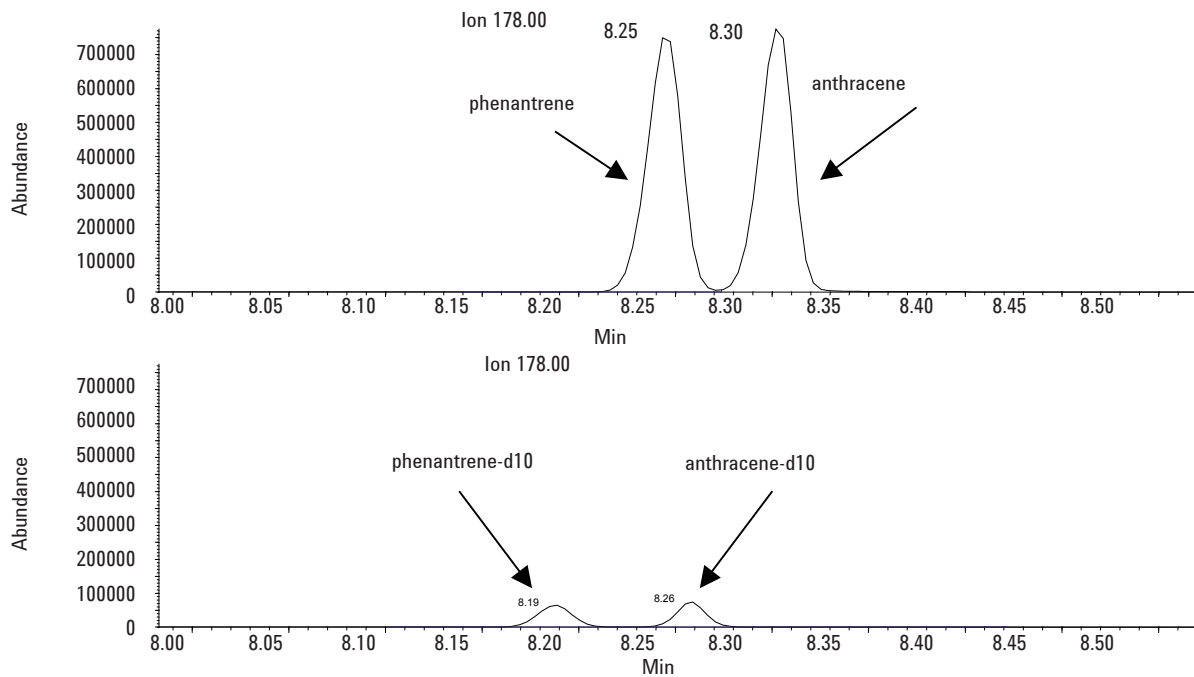


Figure 2. Phenanthrene and Fluoranthene both with  $m/z$  178 are efficiently separated

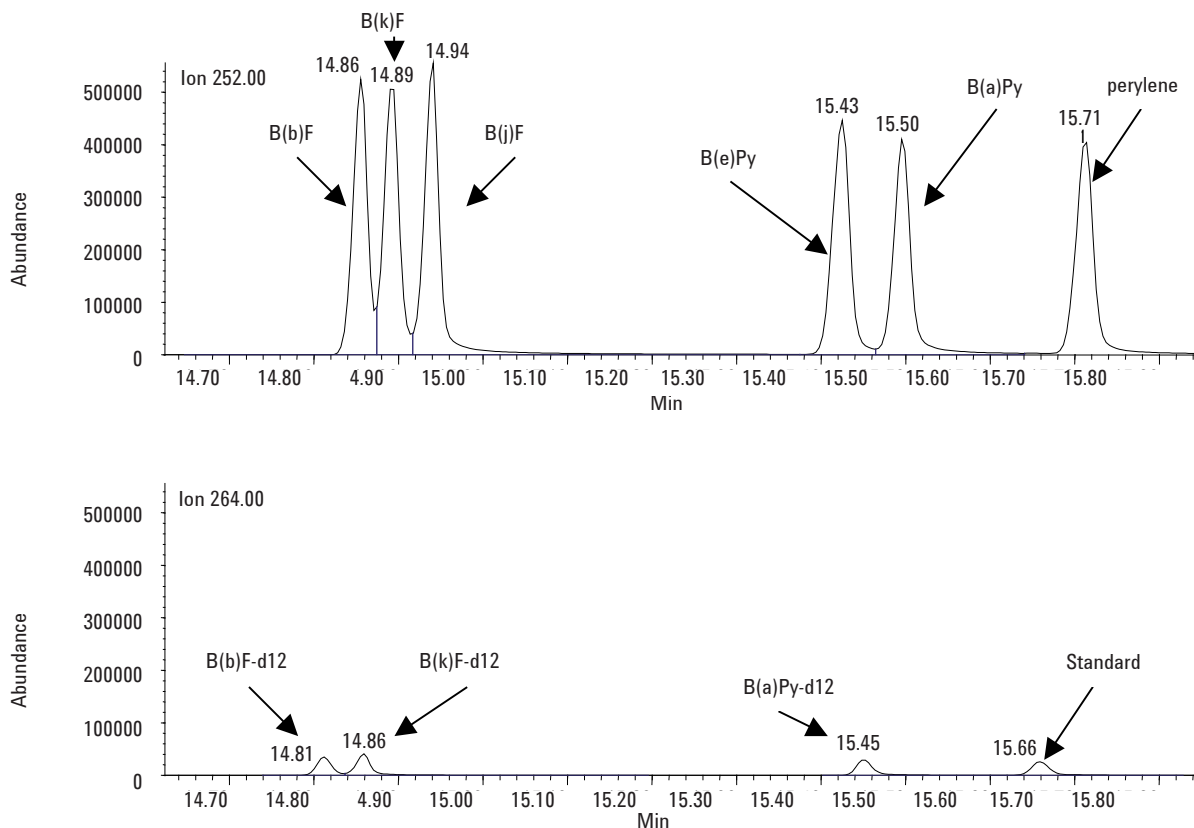
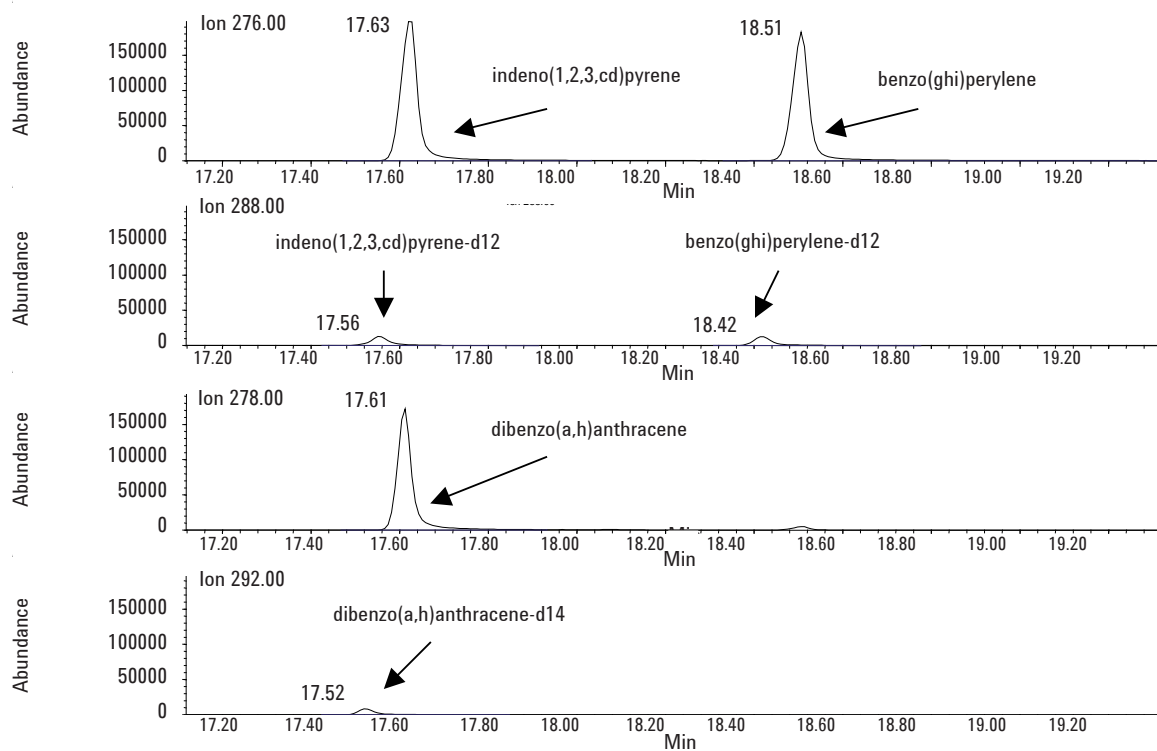
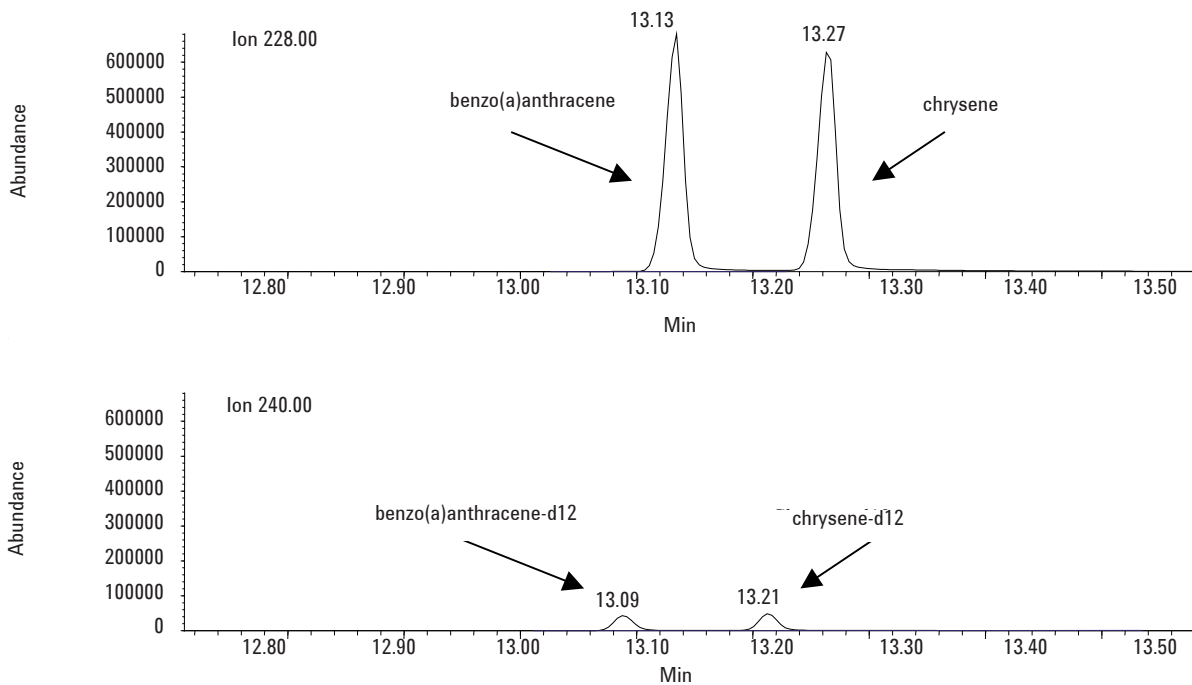


Figure 3. The most critical separations of the PAHs with  $m/z$  252. The benzofluoranthenes isomers are almost baseline resolved



**Figure 4.** The VF-17ms is able to separate indeno(1,2,3,cd) pyrene from dibenzo(a,h)anthracene using different temperature programs, in this analysis they are discriminated by their difference in molecular masses



**Figure 5.** Benzo(a)anthracene and chrysene are well resolved

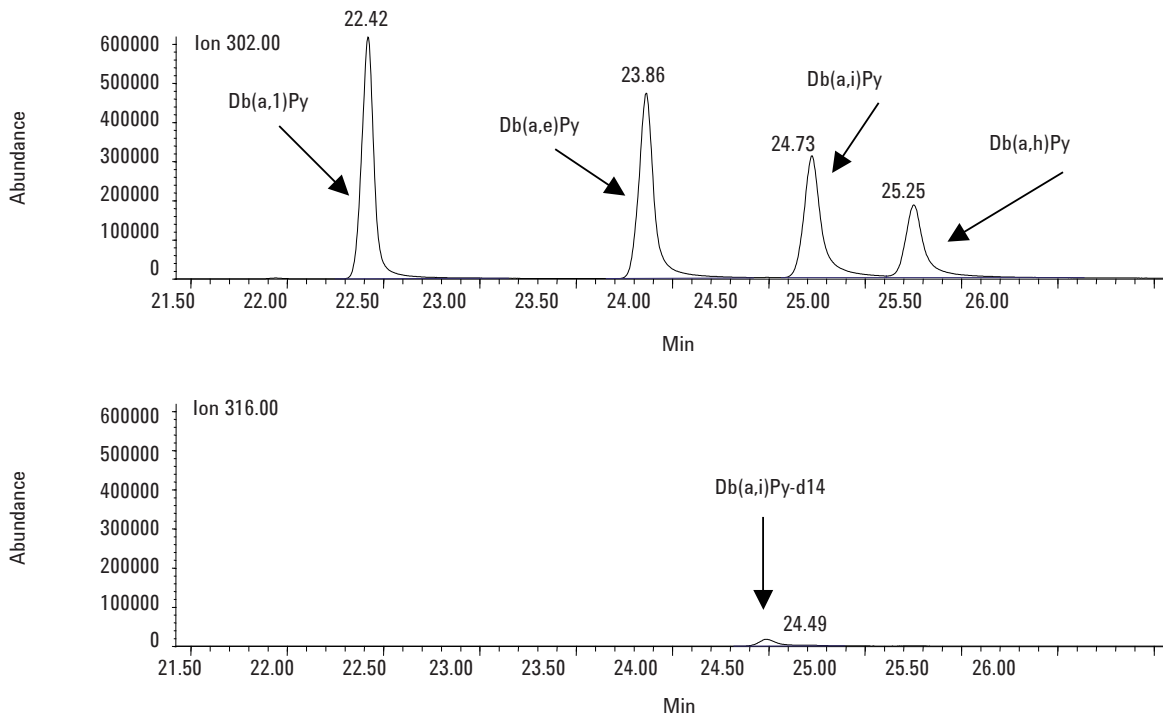


Figure 6. The last target compound dibenzo(a,h)pyrene elutes in about 25 minutes

## References

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