Analysis of PAH4 on Agilent J&W Select PAH

Application Note

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Introduction

Prior to 2010, the European Food Safety Authority (EFSA) regulated only benzo[a] pyrene as being a marker for carcinogenic polycyclic aromatic hydrocarbons (PAHs) in food samples. However, during 2007 and 2008 the EFSA Panel on Contaminants in the Food Chain (CONTAM) studied over 10,000 results for PAH levels in different food commodities. CONTAM concluded that in 30% of all samples, other carcinogenic PAHs were present, though these samples were negative for benzo[a] pyrene. Of all samples that did not show benzo[a]pyrene, the most commonly found of all individual PAHs was chrysene. CONTAM therefore concluded that benzo[a] pyrene was not a suitable marker for PAHs in food, and advised the use of the sum of benzo[a]pyrene, chrysene, benz[a]anthracene and benzo[b]fluoranthene (PAH4) instead. In 2010, the European Commission decided to follow the advice of CONTAM and changed the regulations on PAH analysis in food.

However, there are potential interferences when analyzing PAH4 with gas chromatography. Some of these interferences are isomers, which cannot be resolved using GC/MS, resulting in false positives and inaccurate results. The advanced selectivity of the Select PAH column, and an optimized oven program, provides separation of these PAH isomers, enhancing data quality and providing more straightforward data interpretation. In addition, results are reliable and productivity is improved.

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Materials and Methods

Technique:	GC/MS
Column:	Select PAH, 30 m x 0.25 mm, df = 0.15 µm (part number CP7462)
Sample Conc:	Approx 1 µg/mL
Injection Volume:	1 µL
Temperature:	70 °C (0.5 min), 60 °C/min, 210 °C, 5 °C/min, 250 °C, 10 °C/min, 280 °C (3 min), 10 °C/min, 350 °C (3 min)
Carrier Gas:	Helium, constant flow, 2 mL/min
Injection:	100 °C, 180 °C/min, 300 °C (20 min), splitless
Detection:	Triple Quad MS, EI in SIM, source 275 °C, transfer line 300 °C

Table 1. Peak Identification and SIM ions (PAHs in bold form PAH4 group)

Peak	Compound	Ions
1	Benzo[g,h,i]fluoranthene	226
2	Benzo[c]phenanthrene	228
3	Benz[a]anthracene	228
4	Cyclopenta[c,d]pyrene	226
5	Triphenylene	228
6	Chrysene	228
7	Benzo[b]fluoranthene	252
8	Benzo[k]fluoranthene	252
9	Benzo[j]fluoranthene	252
10	Benzo[a]fluoranthene	252
11	Benzo[e]pyrene	252
12	Benzo[a]pyrene	252
13	Perylene	252

Results and Discussion

The selectivity of the Select PAH column and the optimized oven program delivered excellent separation of the target PAHs and interferences. Figure 1 shows the chromatogram of PAH4 and nine interfering PAHs, in 37 minutes. Figures 2 to 4 reveal details of the separation of the four PAHs.

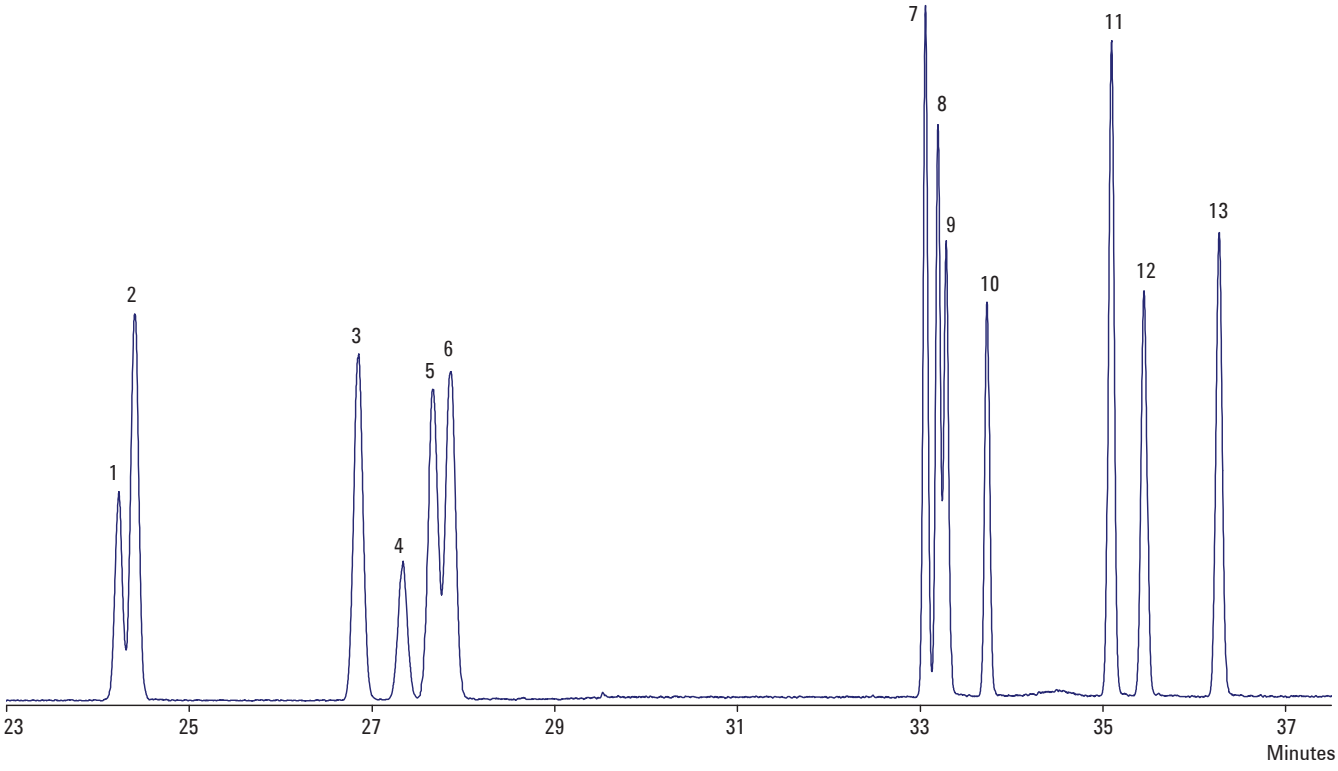

Figure 1. Chromatogram of PAHs analyzed on a Select PAH column

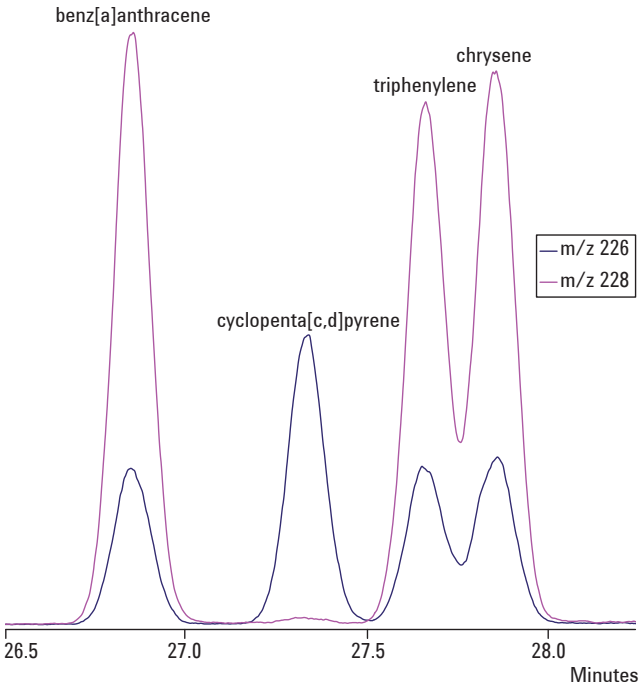

Figure 2. Separation of benz[a]anthracene and chrysene from their interfering PAHs

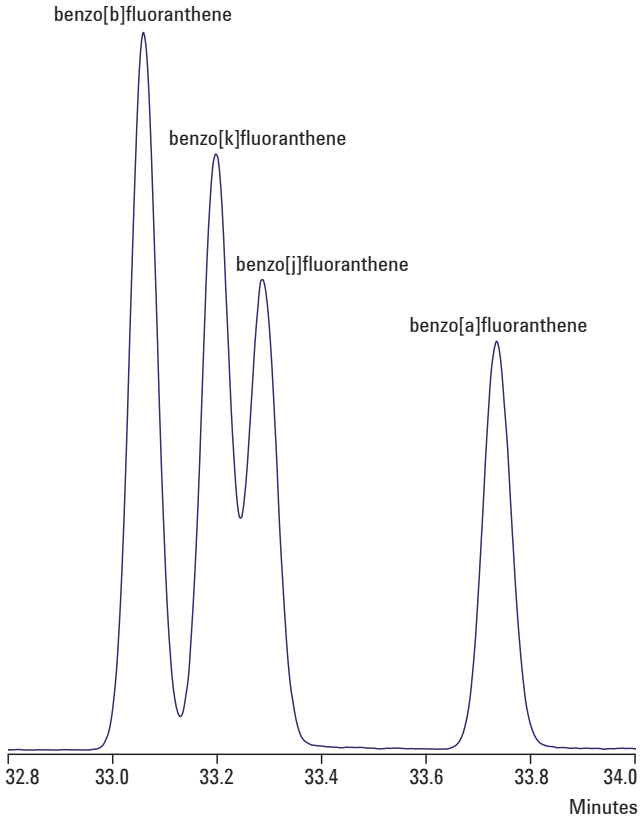

Figure 3. Separation of benzo[b]fluoranthene from its interfering PAHs

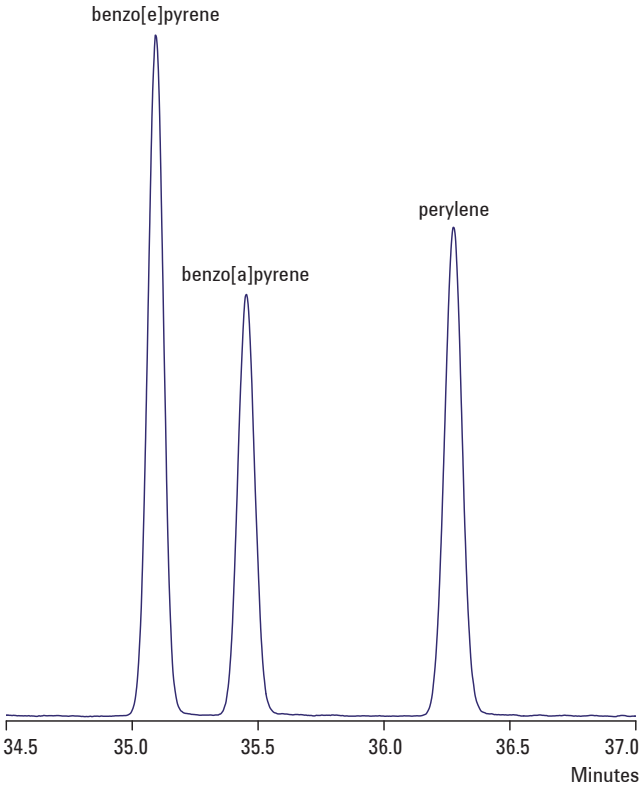

Figure 4. Separation of benzo[a]pyrene from its interfering PAHs

Conclusion

The Select PAH column provided excellent resolution for the analysis of PAH4 in less than 40 minutes. This column can also be used for a broader range of PAHs, providing high performance by combining very good separation of isomers with high productivity.

The Select PAH column, with a maximum programmed temperature of 350 °C, enabled quick bake-out at the end of the analysis, stripping the sample matrix off the column.

References

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EFSA (2008) Scientific Opinion of the Panel on Contaminants in the Food Chain on a Request from the European Commission on Polycyclic Aromatic Hydrocarbons in Food. EFSA Journal, 724, 1-114.

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

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