

APPLICATIONS

Rapid Analysis of 24 Regulated Polycyclic Aromatic Hydrocarbons (PAHs) on a Zebron™ ZB-PAH-EU GC Column

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He has a PhD in Analytical Chemistry and over 15 years experience in chromatographic method development and troubleshooting. Ramkumar loves to write poems, read Shakespeare, and attend Shakespeare plays.

Introduction

Polycyclic Aromatic Hydrocarbons (PAHs) are organic (highly hydrophobic) compounds that contain multiple aromatic rings (e.g. Naphthalene, Chrysene, and Benzo[b]fluoranthene). PAHs often result from the incomplete combustion of organic substances, such as wood, coal, and oil, and are found as pollution in air, water, and soil. Amongst the PAHs are some of the most toxic compounds known. Including some that are carcinogenic, mutagenic, and teratogenic.

In the United States, the EPA (Environmental Protection Agency) has listed 16 priority PAHs. The European Union (EU) has regulated a series of PAHs found in food matrices. This list is referred to as the EU 15+1 list (**Table 1**).

In this study, we utilized the Zebron ZB-PAH-EU GC column selectivity to resolve 24 priority PAH components. Various column dimensions were explored to get optimal separation and fast analysis. ZB-PAH-EU GC columns, 10 meter x 0.10 mm x 0.08 µm provided extreme fast analysis of 24 PAH components within 16 minutes.

Table 1.

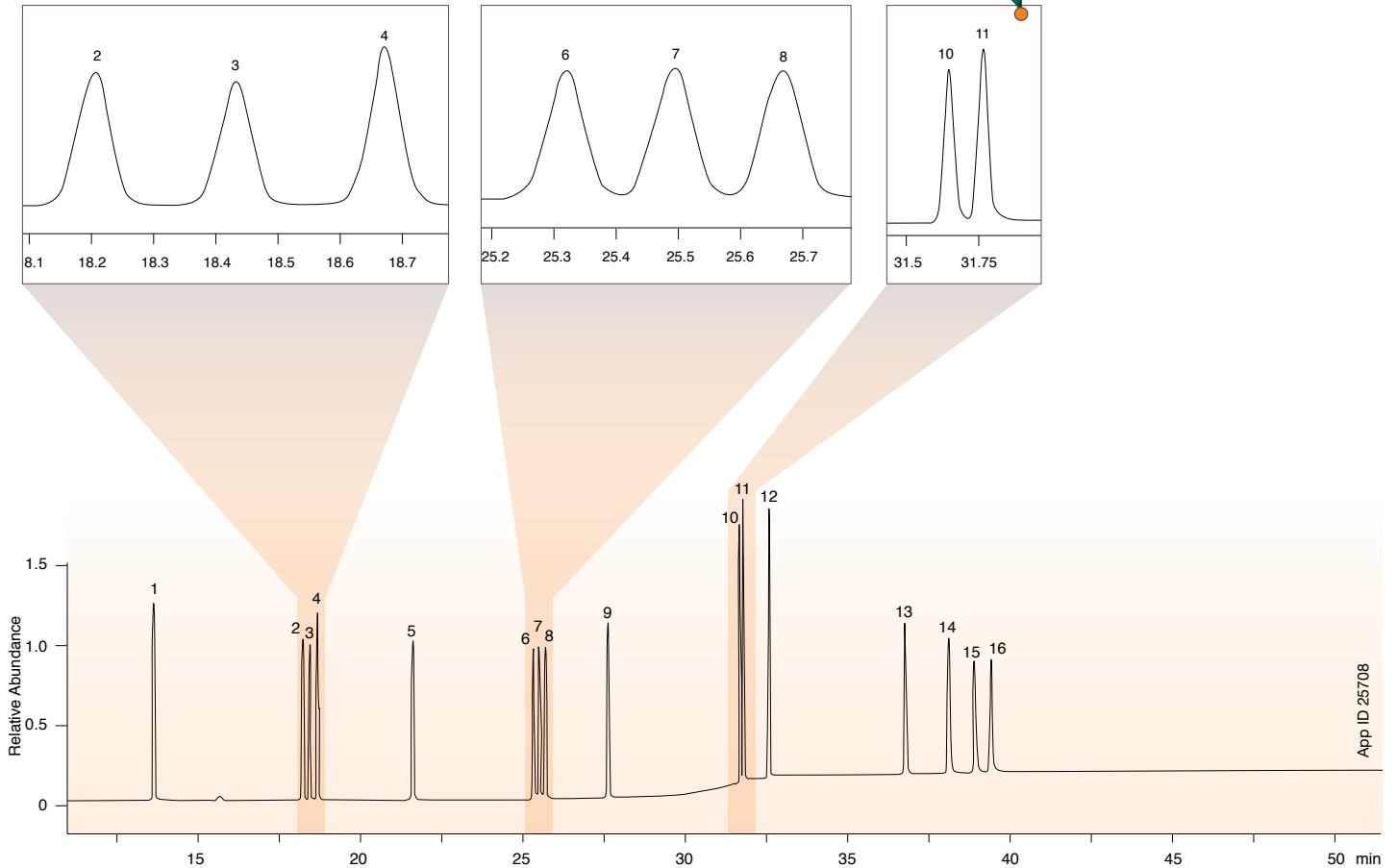
EU and US EPA Regulated PAH Compounds

Peak #	Sample	EU 15+1	EPA
1	Naphthalene		•
2	Acenaphthylene		•
3	Acenaphthene		•
4	Fluorene		•
5	Phenanthrene		•
6	Anthracene		•
7	Fluoranthene		•
8	Pyrene		•
9	Benzo[c]fluorene	•	
10	Benz[a]anthracene	•	•
11	Cyclopenta[c,d]pyrene	•	
12	Chrysene	•	
13	5-Methylchrysene	•	
14	Benzo[b]fluoranthene	•	•
15	Benzo[k]fluoranthene	•	•
16	Benzo[j]fluoranthene	•	
17	Benzo[a]pyrene	•	•
18	Indeno[1,2,3-cd]pyrene	•	•
19	Dibenz[a,h]anthracene	•	•
20	Benzo[g,h,i]perylene	•	•
21	Dibenzo[a,l]pyrene	•	
22	Dibenzo[a,e]pyrene	•	
23	Dibenzo[a,i]pyrene	•	
24	Dibenzo[a,h]pyrene	•	



Figure 1.
EU 15+1 PAHs using a 30 meter Zebtron™ ZB-PAH-EU GC column.

Excellent Resolution of
EU PAH Critical Pairs



App ID 25708

Column: Zebtron ZB-PAH-EU
Dimensions: 30 meter x 0.25 mm x 0.20 um
Part No.: ZHG-G043-10
Injection: Split 5:1 @ 330 °C, 1 µL
Recommended Liner: Zebtron PLUS Single Taper Z-Liner™
Liner Part No.: AG2-4B13-05 (for Shimadzu® 2010 GC)
Carrier Gas: Helium @ 24 psi (constant pressure)
Oven Program: 45 °C for 0.8 min to 200 °C @ 45 °C/min to 226 °C @ 3 °C/min for 0 min to 320 °C @ 10 °C/min for 20 min
Detector: MSD, 50-500 m/z
Transfer line Temperature: 300 °C
Source Temperature: 300 °C

Sample:

1. Benzo[c]fluorene
2. Benz[a]anthracene
3. Cyclopenta[c,d]pyrene
4. Chrysene
5. 5-Methylchrysene
6. Benzo[b]fluoranthene
7. Benzo[k]fluoranthene
8. Benzo[j]fluoranthene
9. Benzo[a]pyrene
10. Indeno[1,2,3-cd]pyrene
11. Dibenzo[a,h]anthracene
12. Benzo[g,h,i]perylene
13. Dibenzo[a,l]pyrene
14. Dibenzo[a,e]pyrene
15. Dibenzo[a,i]pyrene
16. Dibenzo[a,h]pyrene

Analysis of EU 15+1 and EPA 610 PAHs on Various Columns

Figure 2a.

Zebtron™ ZB-PAH-EU 30 meter x 0.25 mm x 0.20 μm

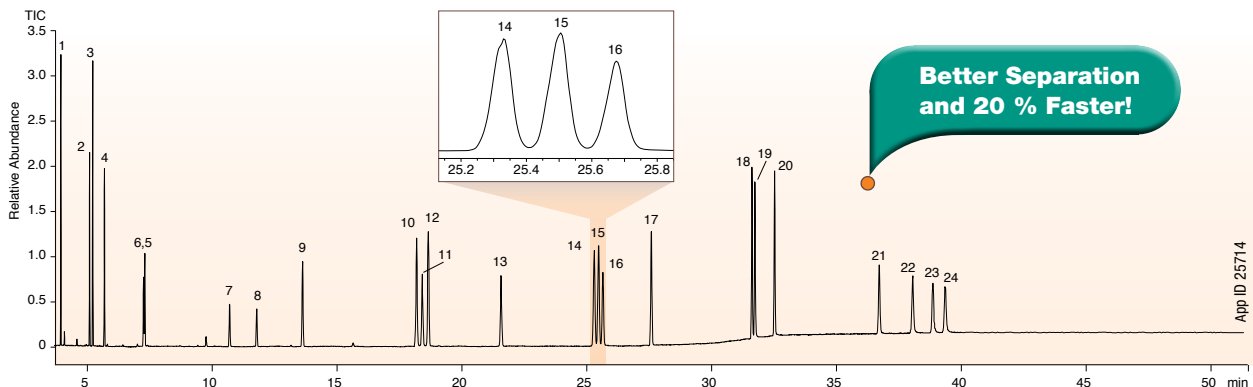


Figure 2b

Popular Brand A 30 meter x 0.25 mm x 0.25 μm

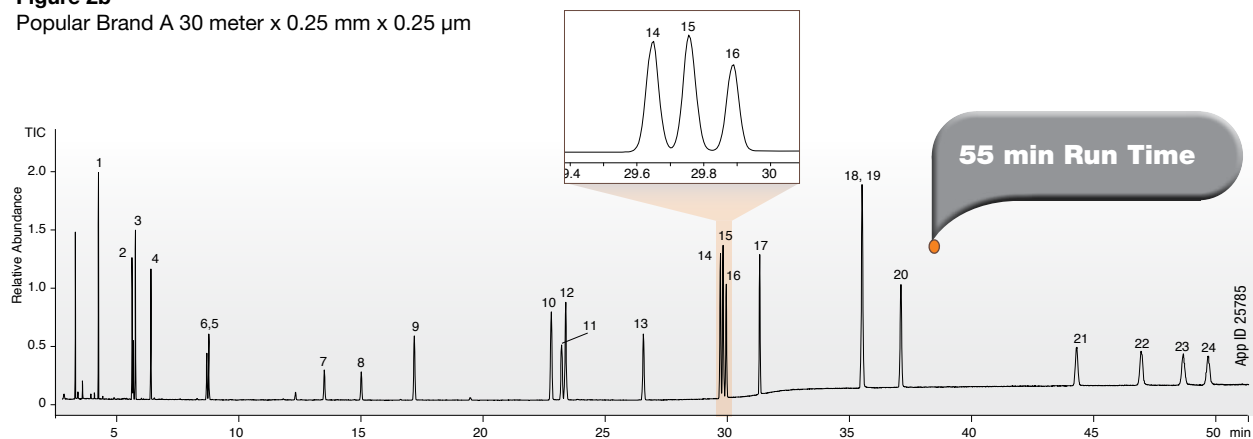
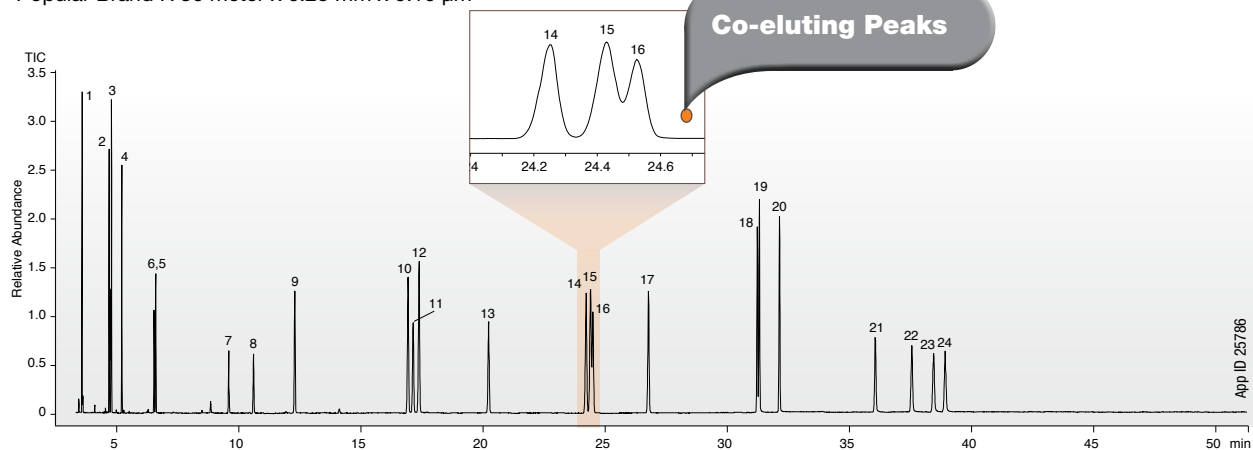


Figure 2c

Popular Brand R 30 meter x 0.25 mm x 0.10 μm



Comparative separations may not be representative of all applications.

Column: As indicated

Dimension: As indicated

Injection: Split 5:1 @ 330 °C, 1.0 μL

Recommended Liner: Zebtron PLUS Single Taper Z-Liner™

Liner Part No.: AG2-4B13-05

Carrier Gas: Helium @ 24 psi (constant pressure)

Oven Program: 45 °C for 0.8 min to 200 °C @ 45°C/min to 226 °C @ 3 °C/min for 0 min to 320 °C @ 10°C/min for 20 min

Detector: MSD @ 300 °C

Detector Mode: Scan 50-500 m/z

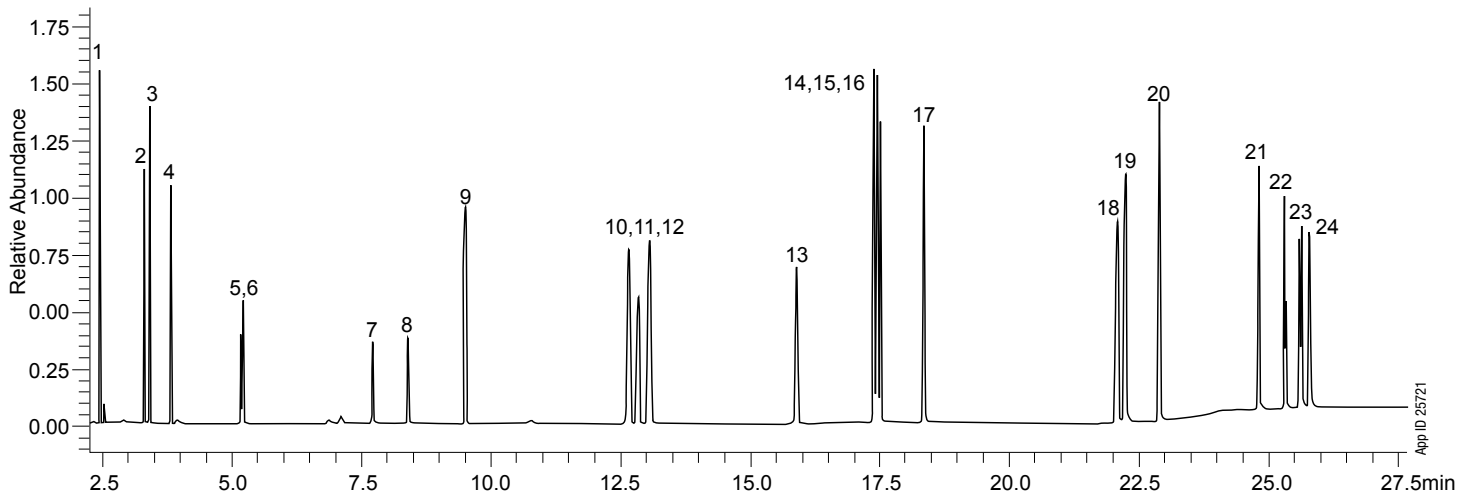
Transfer line Temperature: 300 °C

Sample: Analytes 10 ppm in Toluene

- | | |
|---------------------------|----------------------------|
| 1. Naphthalene | 13. 5-Methylchrysene |
| 2. Acenaphthylene | 14. Benzo[b]fluoranthene |
| 3. Acenaphthene | 15. Benzo[k]fluoranthene |
| 4. Fluorene | 16. Benzo[j]fluoranthene |
| 5. Phenanthrene | 17. Benzo[a]pyrene |
| 6. Anthracene | 18. Indeno[1,2,3-cd]pyrene |
| 7. Fluoranthene | 19. Dibenzo[a,h]anthracene |
| 8. Pyrene | 20. Benzo[g,h,i]perylene |
| 9. Benzo[c]fluorene | 21. Dibenzo[a,i]pyrene |
| 10. Benz[a]anthracene | 22. Dibenzo[a,e]pyrene |
| 11. Cyclopenta[c,d]pyrene | 23. Dibenzo[a,i]pyrene |
| 12. Chrysene | 24. Dibenzo[a,h]pyrene |

Figure 3.

Fast Separation of EU 15+1 and EPA 610 PAHs using a Zebtron™ ZB-PAH-EU 20 meter GC column.



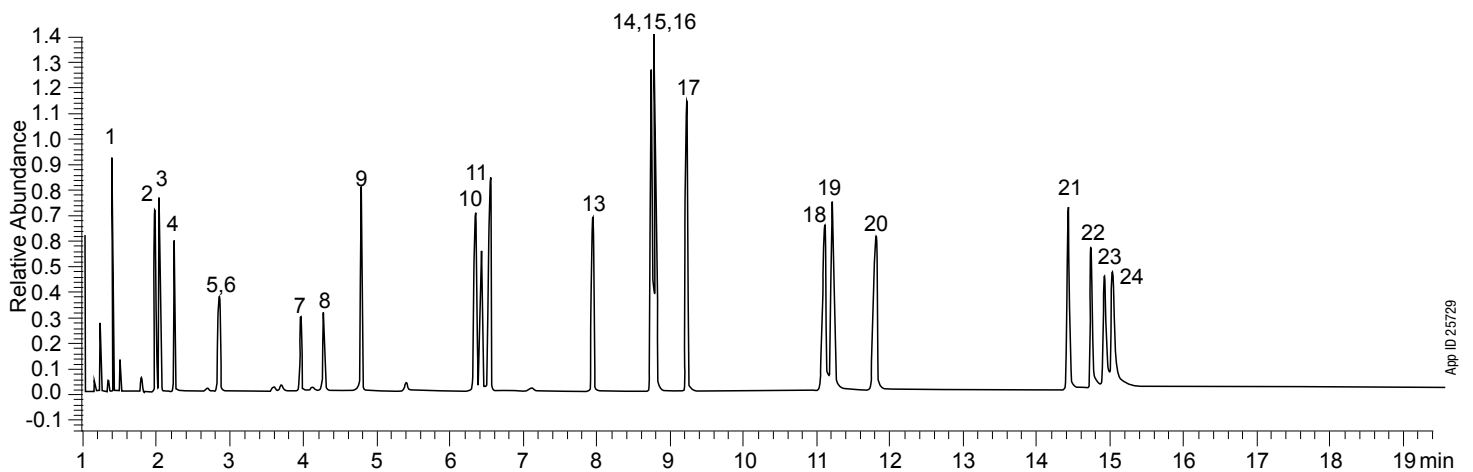
Column: Zebtron ZB-PAH-EU
Dimension: 20 meter x 0.18 mm x 0.14 μm
Part No.: [7FD-G043-47](#)
Injection: Split 5:1 @ 330 °C, 1.0 μL
Recommended Liner: Zebtron PLUS Single Taper Z-Liner™
Liner Part No.: [AG2-4B13-05](#)
Carrier Gas: Helium @ 1.75 mL/min (constant flow)
Oven Program: 70°C for 0.8 min to 180°C @ 70°C/min to 230°C @ 7°C/min for 6 min to 280°C @ 40°C/min for 5 min to 335°C @ 25°C/min for 5 min
Detector: MSD @ 300 °C
Detector Mode: Scan 100-500 m/z
Detector Temperature: 300 °C

Sample: Analytes 10 ppm in Toluene

- | | |
|---------------------------|----------------------------|
| 1. Naphthalene | 13. 5-Methylchrysene |
| 2. Acenaphthylene | 14. Benzo[b]fluoranthene |
| 3. Acenaphthene | 15. Benzo[k]fluoranthene |
| 4. Fluorene | 16. Benzo[j]fluoranthene |
| 5. Phenanthrene | 17. Benzo[a]pyrene |
| 6. Anthracene | 18. Indeno[1,2,3-cd]pyrene |
| 7. Fluoranthene | 19. Dibenzo[a,h]anthracene |
| 8. Pyrene | 20. Benzo[g,h,i]perylene |
| 9. Benzo[c]fluorene | 21. Dibenzo[a,i]pyrene |
| 10. Benz[a]anthracene | 22. Dibenzo[a,e]pyrene |
| 11. Cyclopenta[c,d]pyrene | 23. Dibenzo[a,i]pyrene |
| 12. Chrysene | 24. Dibenzo[a,h]pyrene |

Figure 4.

Faster Separation of EU 15+1 and EPA 610 PAHs using a Zebtron ZB-PAH-EU 10 meter GC column.



Column: Zebtron ZB-PAH-EU
Dimension: 10 meter x 0.10 mm x 0.08 μm
Part No.: [7CB-G043-59](#)
Injection: Split 5:1 @ 330 °C, 1.0 μL
Recommended Liner: Zebtron PLUS Single Taper Z-Liner™
Liner Part No.: [AG2-4B13-05](#)
Carrier Gas: Helium @ 0.88 mL/min (constant flow)
Oven Program: 70°C for 0.4 min to 180°C @ 140°C/min to 230°C @ 14°C/min for 3 min to 280°C @ 85°C/min for 5 min to 330°C @ 40°C/min for 5 min
Detector: MSD @ 300 °C
Detector Mode: Scan 100-500 m/z
Detector temperature: 300 °C

Sample: Analytes 10 ppm in Toluene

- | | |
|---------------------------|----------------------------|
| 1. Naphthalene | 13. 5-Methylchrysene |
| 2. Acenaphthylene | 14. Benzo[b]fluoranthene |
| 3. Acenaphthene | 15. Benzo[k]fluoranthene |
| 4. Fluorene | 16. Benzo[j]fluoranthene |
| 5. Phenanthrene | 17. Benzo[a]pyrene |
| 6. Anthracene | 18. Indeno[1,2,3-cd]pyrene |
| 7. Fluoranthene | 19. Dibenzo[a,h]anthracene |
| 8. Pyrene | 20. Benzo[g,h,i]perylene |
| 9. Benzo[c]fluorene | 21. Dibenzo[a,i]pyrene |
| 10. Benz[a]anthracene | 22. Dibenzo[a,e]pyrene |
| 11. Cyclopenta[c,d]pyrene | 23. Dibenzo[a,i]pyrene |
| 12. Chrysene | 24. Dibenzo[a,h]pyrene |

Results and Discussion

Presented in **Table 1** is the list of PAH compounds included in EPA 610 and EU 15+1. Combination of these two lists provides the 24 PAH analytes. Since many of the PAH components are isomers, it is essential to have a column selectivity that can recognize small changes in the electron density around the polycyclic compounds. Zebtron™ ZB-PAH-EU GC columns has proprietary selectivity, specially designed to resolve PAH components. In addition, these columns undergo Engineered Self Cross-linking (ESC™) to provide low column bleed and highly reproducible separation from run to run. In addition, ESC helps the ZB-PAH-EU GC column in attaining 340/360°C maximum temperature. **Figure 1** represents the separation of EU 15+1 PAH on a 30 meter ZB-PAH-EU column with complete resolution of EU 15+1 priority PAHs. Critical pairs such as benz[a]anthracene, cyclopenta[c,d]pyrene, chrysene, benzo[b]fluoranthene, benzo[k] fluoranthene, benzo[j]fluoranthene, indeno[1,2,3-cd] pyrene, and dibenz[a,h]anthracene are completely resolved allowing for more accurate detection and quantification. **Figure 2a, 2b** and **2c** represent the comparison of separation of 24 PAH compounds that includes EU 15+1 and EPA 610 list. With a 30 meter length ZB-PAH-EU, 24 PAH compounds were separated within 40 minutes. This was compared against Popular Brand A 30 meter x 0.25 mm x 0.25 µm (**Figure 2b**) and Popular Brand R 30 meter x 0.25 mm x 0.10 µm. (**Figure 2c**) ZB-PAH-EU GC column provided shortest run time compared to Popular Brand A. While Popular Brand R provided similar run time, certain critical pairs like benzo[k]fluoranthene-benzo[j]fluoranthene and indeno[1,2,3-cd] pyrene-dibenz[a,h]anthracene were not completely resolved.

Another set of challenging analytes are the four dibenzopyrene isomers. Due to their high molecular weight, these isomers are prone to inlet discrimination and poor peak shape. The Zebtron ZB-PAH-EU GC column provides a thinner film stationary phase and high maximum temperature limit of 340/360°C temperatures which lead to improved peak shape for these later eluting analytes.

Further, to explore faster run time for high-throughput analysis, a 20 meter x 0.18 mm x 0.14 µm and 10 meter x 0.10 mm x 0.08 µm dimensions of ZB-PAH-EU GC columns were explored. The 20 meter ZB-PAH-EU GC column separated 24 PAH compounds within 27 minutes (**Figure 3**), while with the 10 meter column dimension the separation was achieved within 16 minutes (**Figure 4**). Such short run times are extremely useful for high throughput laboratories that need fast analysis.

Conclusion

The Zebtron ZB-PAH GC column resolves all 24 EU 15+1 and EPA 610 regulated PAHs in under 16 minutes, including the benzo[b,j,k] fluoranthene isomers which are difficult to separate. The combination of proprietary stationary phase, unique column dimensions, high efficiency and higher maximum temperature provides optimal separation of critical priority PAH compounds with short run time.

Zebtron™ ZB-PAH-EU GC column

Length (m)	ID(mm)	df(µm)	Temp. Limits °C	Part No.
10	0.10	0.08	40 to 340/360	7CB-G043-59
20	0.18	0.14	40 to 340/360	7FD-G043-47
30	0.25	0.20	40 to 340/360	7HG-G043-10
60	0.25	0.20	40 to 340/360	7KG-G043-10

Zebtron™ ZB-PAH-CT GC column

Length (m)	ID(mm)	df(µm)	Temp. Limits °C	Part No.
20	0.18	0.14	40 to 320/340	7FD-G044-47
30	0.25	0.20	40 to 320/340	7HG-G044-10
40	0.18	0.14	40 to 320/340	7PD-G044-47



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