Fast Analysis of Regulated PAHs by GC-MS using Zebron™ ZB-PAH-EU and Zebron ZB-PAH-CT

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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 contamination in air, water and soil. Furthermore, PAHs enter the food chain via deposition or transfer from air, water and soil or food processing. 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. The list of EPA, EU(15+1) PAH and EFSA4 PAH are listed in **Table 1**. In addition, there are various regulations for PAH in plastic, rubber and electronic components. Listed below are common challenges associated with PAH separation.

Separation of Critical PAH	High efficiency and high selectivity for aromatic compounds are essential to provide higher resolution
Long Analysis Time	High efficiency dimension and optimal stationary phase chemistry are necessary for cutting down the run time
Ghost Peaks and Matrix Effects	Higher temperature limits and extensive crosslinking are necessary for high temperature GC bakeout
False Positives	Alternate selectivity and high efficiency dimensions are necessary to avoid false positives

Materials and Methods

In this study, we utilized two unique GC columns: Zebron ZB-PAH-EU and ZB-PAH-CT to resolve PAH components. Various column dimensions were explored to get optimal separation, fast and high-throughput analysis. While both column chemistries provided faster run time compared to other commercially available PAH columns, ZB-PAH-EU offered a maximum temperature of 340/360°C. In addition, it resolved 24 priority PAH components in less than 16 minutes. ZB-PAH-CT offered highest resolution for critical pair Chrysene and Triphenylene, which helps to minimize false positives in PAH analysis.

FIGURE 1.
Zebron ZB-PAH-EU & ZB-PAH-CT Benefits

ZB-PAH-EU
- Up to 70% Faster PAH Analysis
- Elevated Temperature Stability (340/360 °C)

ZB-PAH-CT
- Enhanced Resolution for Chrysene and Triphenylene (PAH Interferences)
- Increased Benzo[b,k]fluoranthene Separation

TABLE 1.
PAH Isomers Listed in Various Regulations

PAHs	EPA PAH	EU(15+1) PAH	EFSA4 PAH
Naphthalene	x		
Acenaphthylene	x		
Acenaphthene	x		
Fluorene	x		
Phenanthrene	x		
Anthracene	x		
Fluoranthene	x		
Pyrene			
Benzo[c]fluorene	x	x	
Cyclopenta[cd]pyrene	x	x	
Benz[a]anthracene		x	x
Chrysene		x	x

PAHs (continued)	EPA PAH	EU(15+1) PAH	EFSA4 PAH
5-Methylchrysene		x	
Benzo[b]fluoranthene	x	x	x
Benzo[k]fluoranthene	x	x	
Benzo[j]fluoranthene		x	
Benzo[a]pyrene	x	x	x
Indeno[1,2,3-cd]pyrene	x	x	
Benzo[g,h,i]perylene	x	x	
Dibenz[a,h]anthracene	x	x	
Dibenzo[a,l]pyrene		x	
Dibenzo[a,i]pyrene		x	
Dibenzo[a,e]pyrene		x	
Dibenzo[a,h]pyrene		x	

FIGURE 2.
Complete Separation of EU(15+1) Isomers on Zebron ZB-PAH-EU GC Column

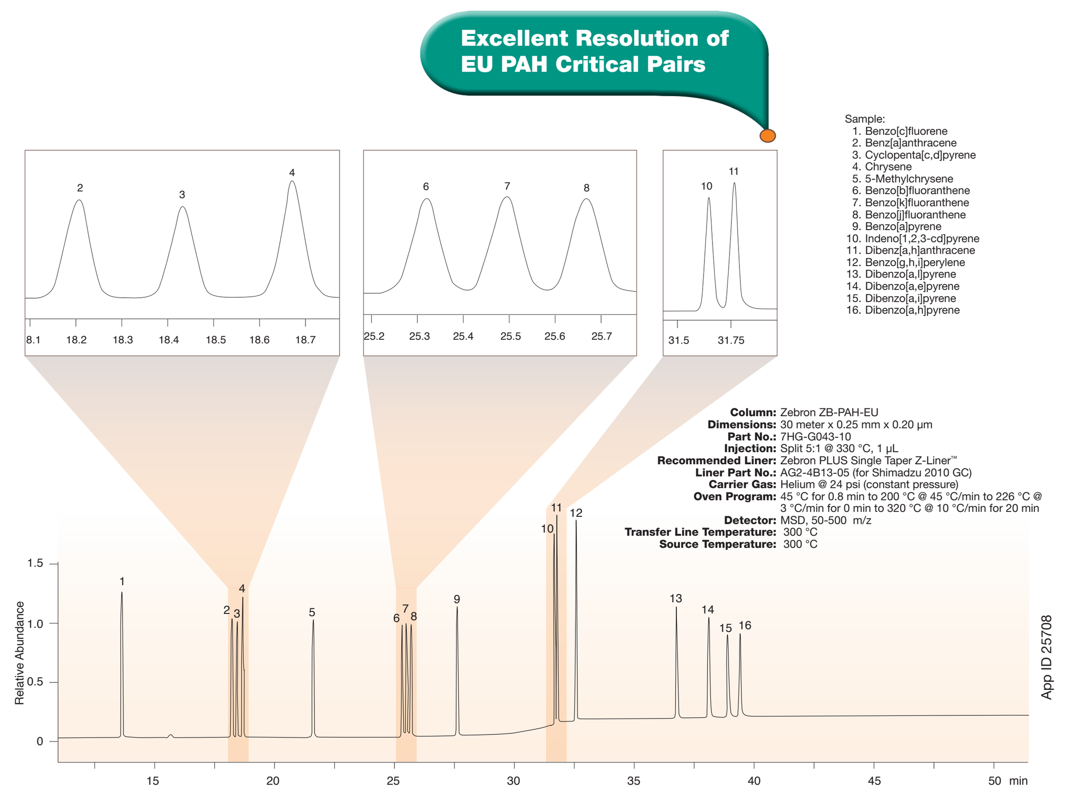


FIGURE 3.
Fast PAH Analysis with Optimal Dimension on Zebron ZB-PAH-EU GC Columns

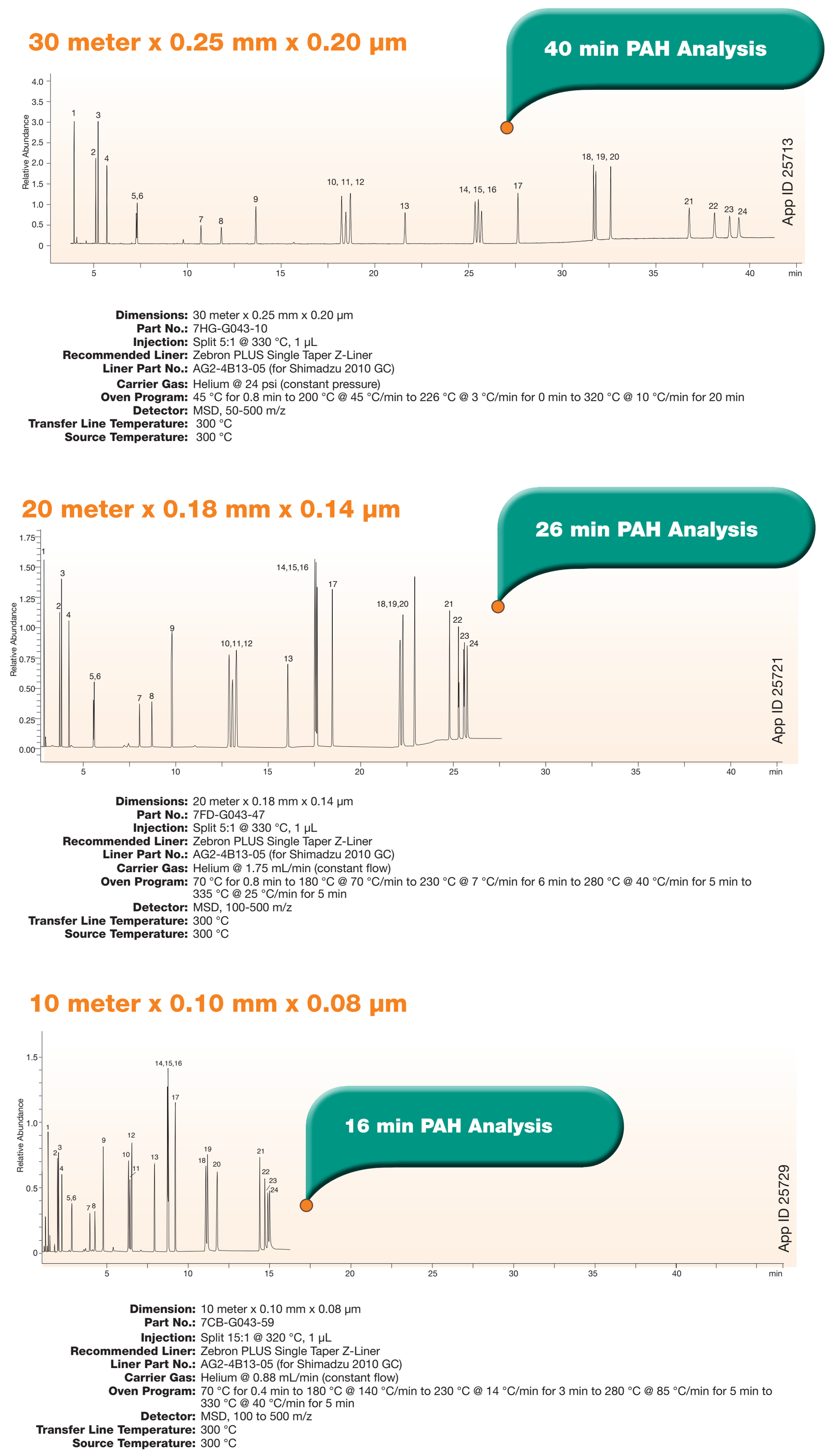
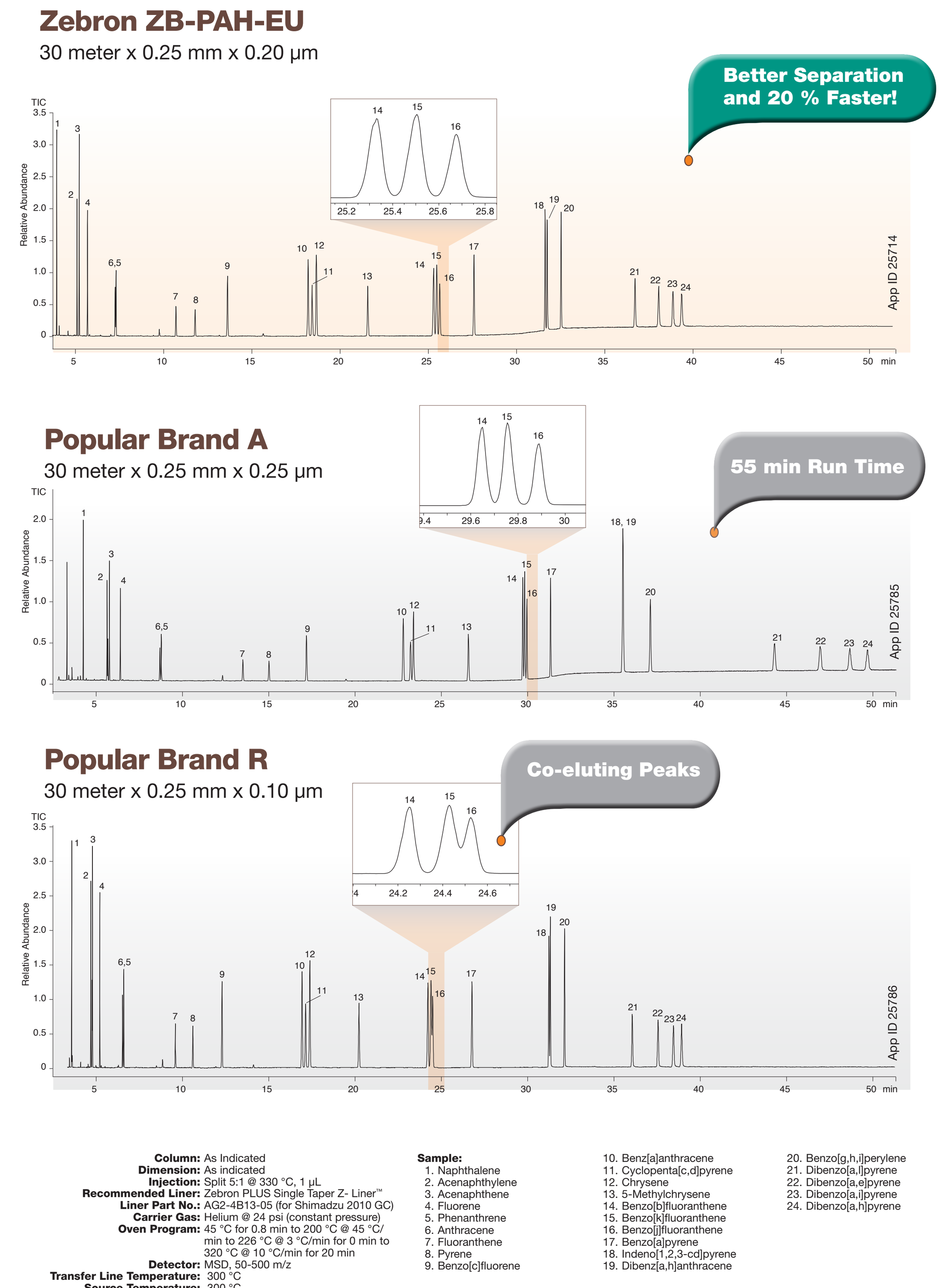


FIGURE 4.
Comparison of Resolution and Speed of Analysis on PAH GC Columns



Comparative separations may not be representative of all applications.

FIGURE 5.
Unique Selectivity to Resolve Chrysene and Triphenylene along with other EU(15+1) Isomers

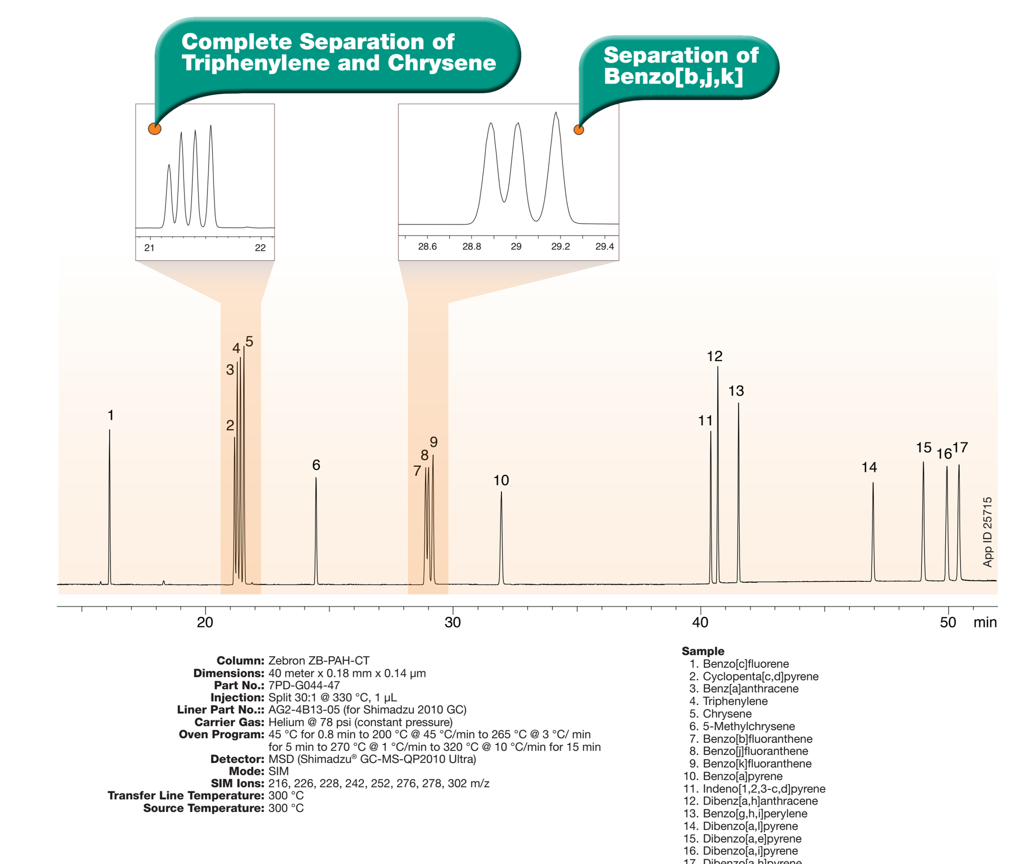
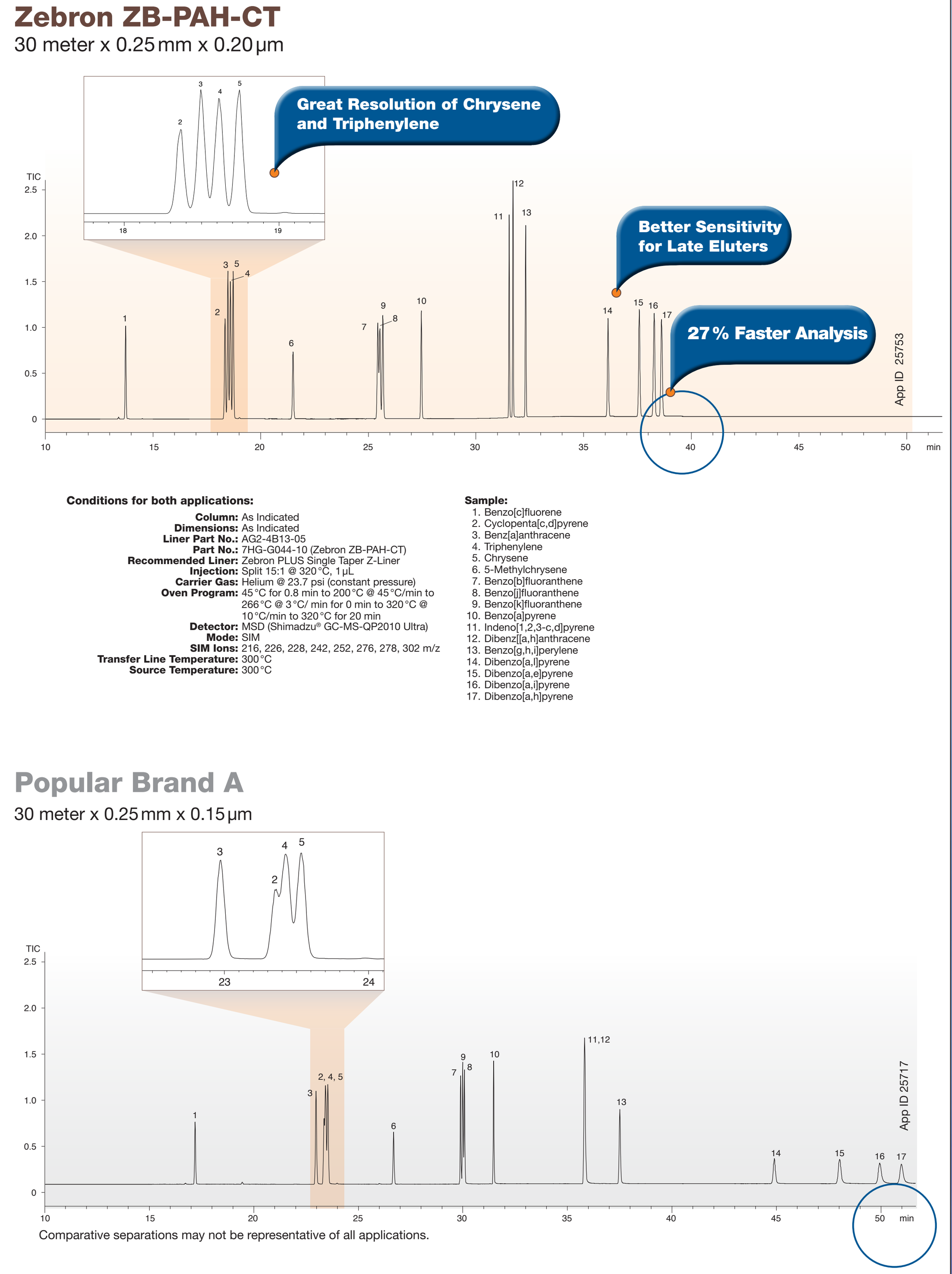


FIGURE 6.
Short Run Time and Better Sensitivity on ZB-PAH-CT



Comparative separations may not be representative of all applications.

Results and Discussion

Analysis of PAH components is challenging considering the structural similarities of the components. In addition, it is a time intensive separation and there is a need for fast GC separation. Presented in this study are two GC solutions for fast separation of PAHs. **Table 1** lists the PAH components mandated in various regulations for environmental and food testing. **Figure 2** represents the complete separation of EU(15+1) isomers on a ZB-PAH-EU GC column.

To further optimize run time 30, 20, and 10 meter ZB-PAH-EU GC columns with comparable phase ratio were evaluated as shown in **Figure 3**. With shorter column length and high efficiency dimension like 10 meter x 0.10 mm x 0.08 µm, a run time as short as 16 min was achieved. Such a short run time is extremely useful for high-throughput analysis. In addition, ZB-PAH-EU 30 meter GC column was compared with other commercially available PAH GC columns in the market under identical conditions. ZB-PAH-EU provided fast analysis as well as good separation of EU 15+1 and EPA Method 610 PAH compounds as illustrated in **Figure 4**.

For alternate selectivity and to resolve critical PAHs, like Chrysene and Tripheylene, a ZB-PAH-CT GC column was employed. The test mix had EU(15+1) PAH and Triphenylene in it. As shown in **Figure 5**, ZB-PAH-CT was able to completely separate Chrysene from Triphenylene in addition to other critical pairs. Due to the unique selectivity, this column can provide a selectivity change. For example, the peak order for Benzofluoranthene are Benzo[b]fluoranthene peak 7, Benzo[j]fluoranthene peak 8 and Benzo[k]fluoranthene peak 9 unlike traditional PAH selectivity. This peak order change helps to eliminate false positives and provides the highest resolution between Benzo[b]fluoranthene and Benzo[k]fluoranthene. **Figure 6** shows short run time and better sensitivity on ZB-PAH-CT compared to a popular GC column.

Conclusion

High efficiency column dimensions are absolutely essential for separation of PAH components. In addition, short run time, higher temperature limits, and unique selectivity are essential for high-throughput applications and challenging matrices. ZB-PAH-EU and ZB-PAH-CT provided fast PAH analysis. While ZB-PAH-EU provided shorter analysis time, it also offers a higher temperature limit of 340/360 °C to elute higher PAH and to bake out matrix contaminants. ZB-PAH-CT offered alternate selectivity that can resolve Chrysene from Triphenylene completely, thereby avoiding false positives.

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