

Exploring GC Column Selectivity for Analysis of Aromatic Compounds



Presented By

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Global Product Manager -GC

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Phenomenex

Overview

Polycyclic Aromatic Hydrocarbons

- PAH Terminologies & Regulations
- Challenges with PAH Analysis

ZB-PAH-EU

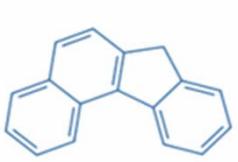
- Complete resolution of EU15+1 and EPA 610 PAHs
- Speed up your PAH Analysis
- PAH in Food
- One Column for PAH & PCB

ZB-PAH-CT

- Unique Selectivity for Chrysene & Triphenylene
- Short Run Time & better Sensitivity
- Phthalates in Food
- EPA-8270 Method Resolution Improvement

Aromatic Contaminants

PAH



Benzo[c]fluorene

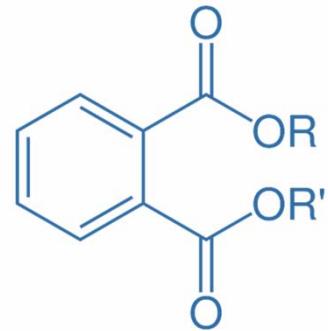


Benz[a]
anthracene



Cyclopenta[cd]pyrene

Phthalates



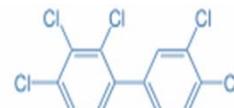
PCB



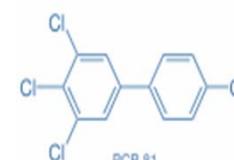
PCB 77



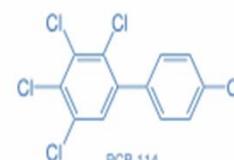
PCB 105



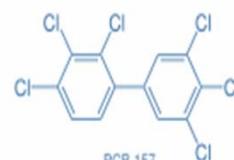
PCB 156



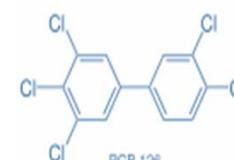
PCB 81



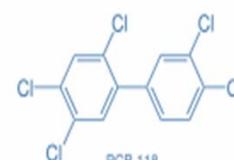
PCB 114



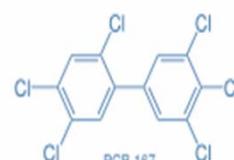
PCB 157



PCB 126



PCB 118



PCB 167

PAH Terminologies

- Polycyclic Aromatic Hydrocarbons
- Polynuclear Aromatic Hydrocarbons
- Poly Aromatic Hydrocarbons
- Polycyclic Organic Hydrocarbons
- Priority Pollutant
- Persistent Organic Pollutant(POPs)
- Poly Chlorinated Biphenyls (PCBs)
- Terphenyl

Regulations

- Environmental Protection Agency: **EPA**
- European Food Safety Authority: **EFSA**
- International Electrotechnical Commission: **IEC**
- AfPS GS 2014:01 pak regulation
- European Chemical Agency **ECHA**
- Health Canada
- EU 15+1

Common Challenges with PAH Testing



Time Intensive Analysis

- Generally 50-60 min run time
- Heavy PAHs have lower sensitivity
- Heavy PAH has inlet discrimination



Resolution of Critical pairs

- PAH isomers are very close in structure & difficult to resolve
- Different regulations require different pair separation



Matrix

- Matrix can have adverse effect on analysis quality
- Food matrix needs higher temperature bakeout
- Ghost peaks can appear from high boiling matrix contamination



Sensitivity

- Heavier PAH may lose sensitivity because of inlet discrimination and low column max temperature



Unleash the power of Fast GC PAH Analysis



Selectivity1: ZB-PAH-EU

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

Selectivity2: ZB-PAH-CT

- Enhanced Resolution for Chrysene and Triphenylene
- Increased Benzo[b,k]fluoranthene Separation

Factors Influencing Resolution

Master Resolution Equation

$$R_s = \frac{\sqrt{N}}{4} \cdot \frac{a - 1}{a} \cdot \frac{k}{1 + k}$$

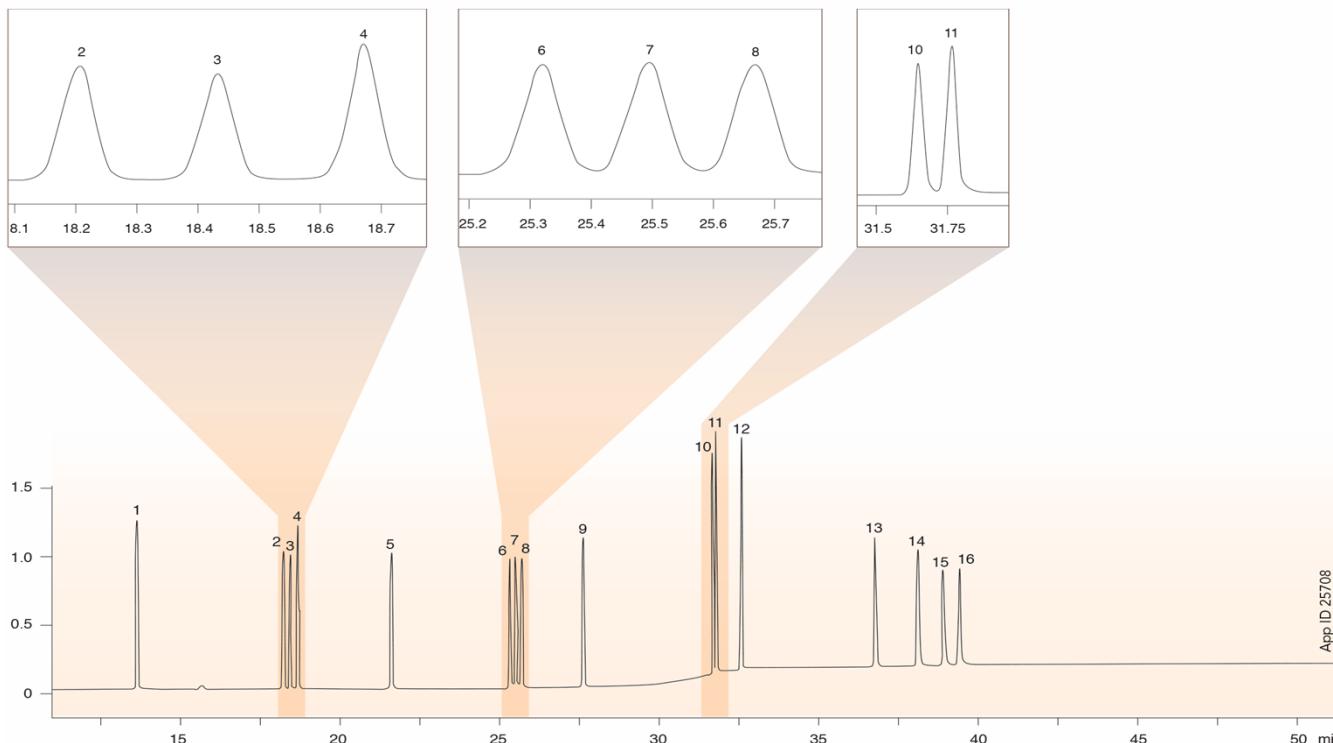
Efficiency **Selectivity** **Retention**

N = theoretical plates

$\alpha = k_2/k_1$

$k = (t_R - t_o)/t_o$

Complete Resolution of EU 15+1 PAH



GC-MS Method Conditions

Column: Zebron ZB-PAH-EU

Dimensions: 30 meter x 0.25 mm x 0.20 µm

Part No.: 7HG-G043-10

Injection: Split 5:1 @ 330 °C, 1 µL

Recommended Liner: Zebron 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

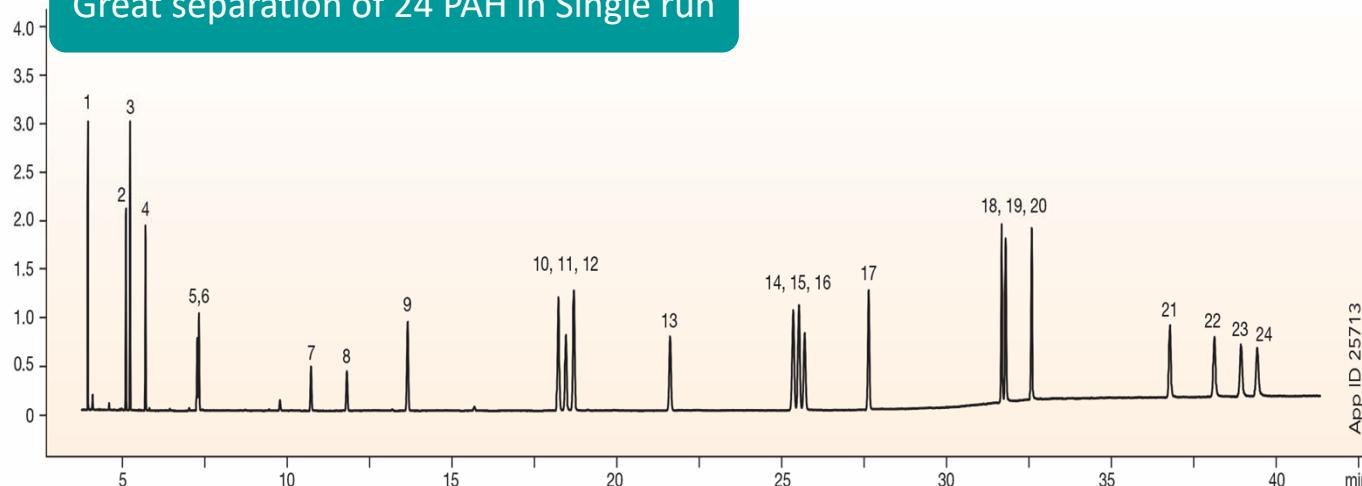
Source Temperature: 300 °C

Sample:

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

Complete Resolution of EU (15+1) and EPA 610 PAHs

Great separation of 24 PAH in Single run



GC-MS Method Conditions

Column: Zebron ZB-PAH-EU

Dimensions: 30 meter x 0.25 mm x 0.20 µm

Part No.: 710G-G043-10

Injection: Split 5:1 @ 330 °C, 1 µL

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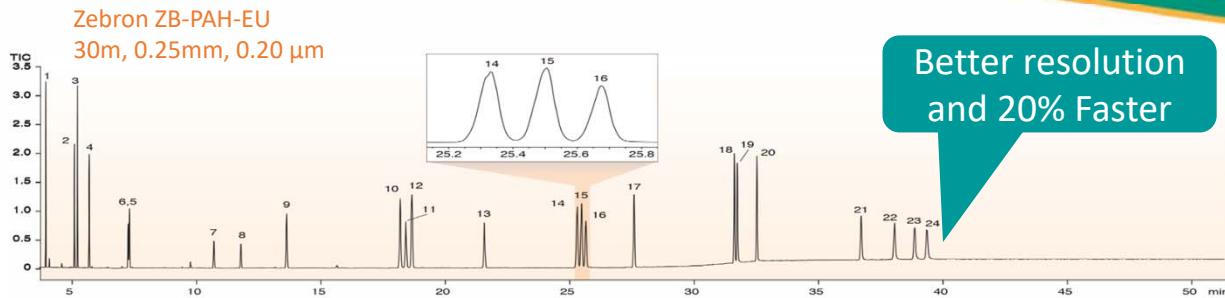
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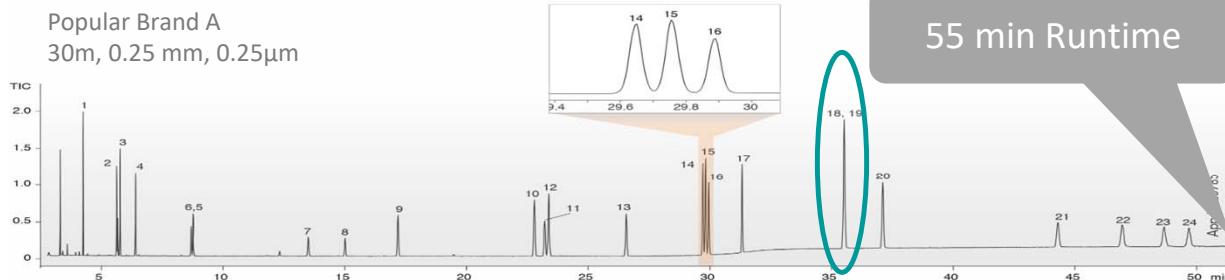
Transfer line Temperature: 300 °C

Source Temperature: 300 °C

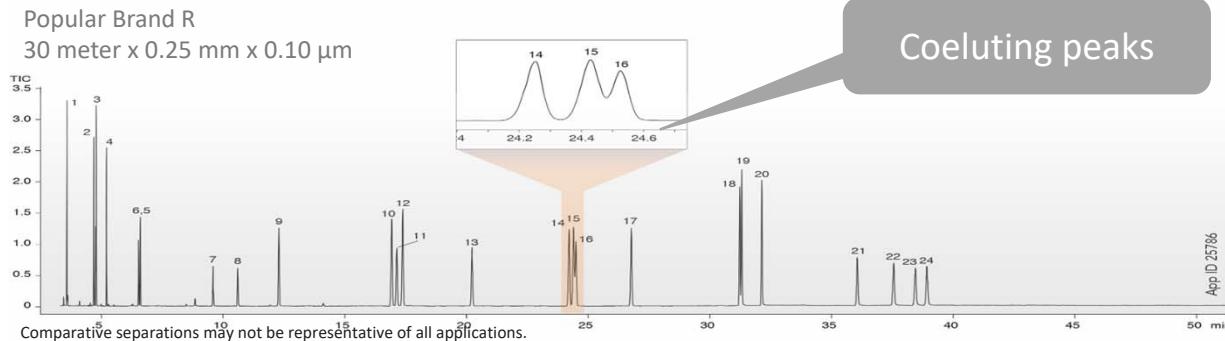
- | | | | | |
|-------------------|-----------------------|---------------------------|----------------------------|------------------------|
| 1. Naphthalene | 6. Anthracene | 11. Cyclopenta[c,d]pyrene | 16. Benzo[j]fluoranthene | 21. Dibenzo[a,l]pyrene |
| 2. Acenaphthylene | 7. Fluoranthene | 12.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



Better resolution
and 20% Faster



55 min Runtime



Coeluting peaks

Comparative separations may not be representative of all applications.

Better Combination of Resolution and Speed

GC-MS Conditions

Column: As Indicated

Dimension: As indicated

Part No.: 7HG-G043-10 (Zebron ZB-PAH-EU column)

Injection: Split 5:1 @ 330 °C, 1 μ L

Recommended Liner: Zebron PLUS Single Taper Z- Liner™

Liner Part No.: AG2-4B13-05 (for Shimadzu 2010 GC)

Carrier Gas: Helium @ 24 psi (constant pressure)

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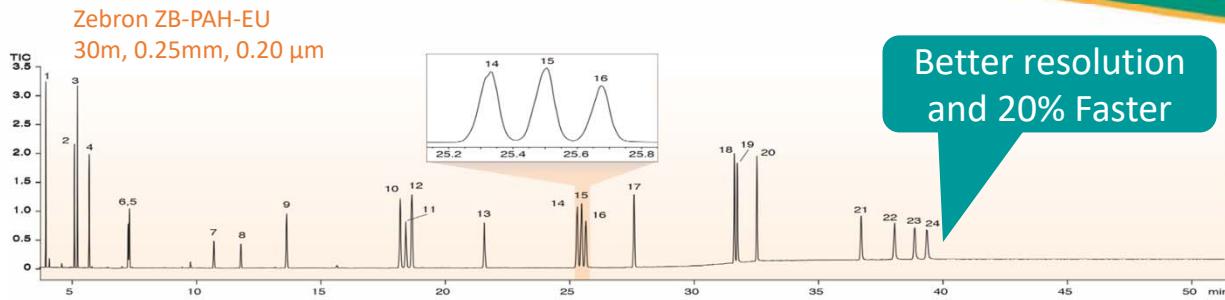
Detector: MSD, 50-500 m/z

Transfer line Temperature: 300 °C

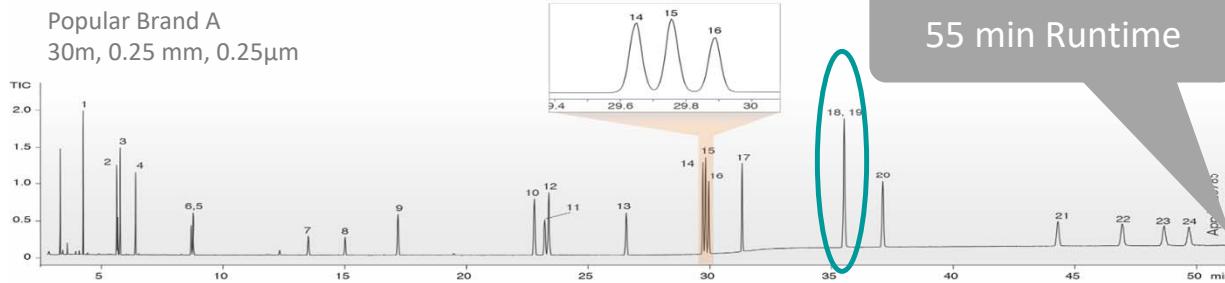
Source Temperature: 300 °C

Sample:

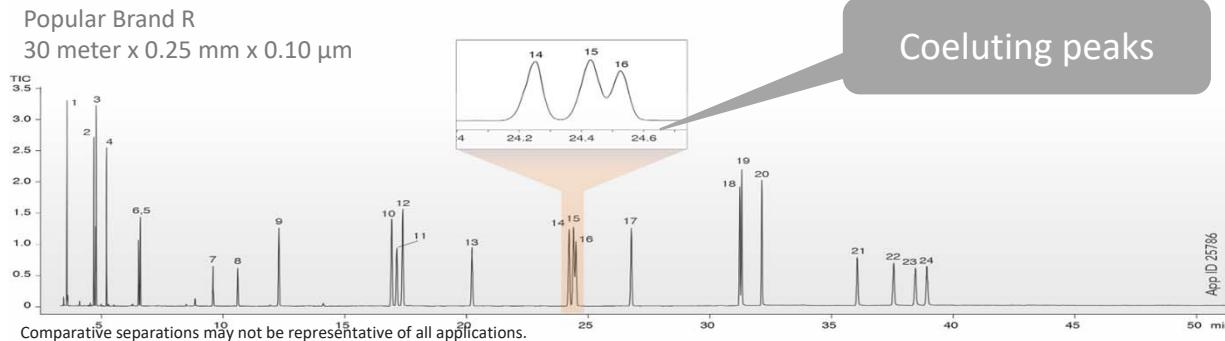
- | | |
|---------------------------|----------------------------|
| 1. Naphthalene | 13. 5-Methylchrysene |
| 2. Acenaphthylene | 14. Benzo[b]fluoranthene |
| 3. Acenaphthene | 15. Benzo[k]fluoranthene |
| 4. Fluorene | 16. Benzo[j]fluoranthene |
| 5. Phenanthrene | 17. Benzo[a]pyrene |
| 6. Anthracene | 18. Indeno[1,2,3-cd]pyrene |
| 7. Flouranthene | 19. Dibenz[a,h]anthracene |
| 8. Pyrene | 20. Benzo[g,h,i]perylene |
| 9. Benzo[c]fluorene | 21. Dibenzo[a,l]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 |



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55 min Runtime



Coeluting peaks

Comparative separations may not be representative of all applications.

Better Combination of Resolution and Speed

GC-MS Conditions

Column: As Indicated

Dimension: As indicated

Part No.: 7HG-G043-10 (Zebron ZB-PAH-EU column)

Injection: Split 5:1 @ 330 °C, 1 μ L

Recommended Liner: Zebron 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

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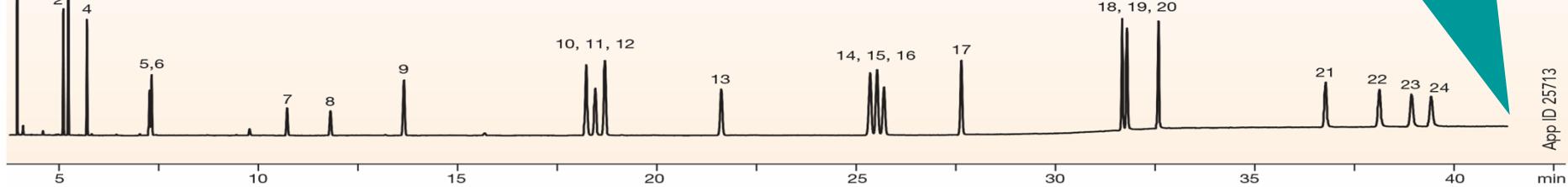
Source Temperature: 300 °C

Sample:

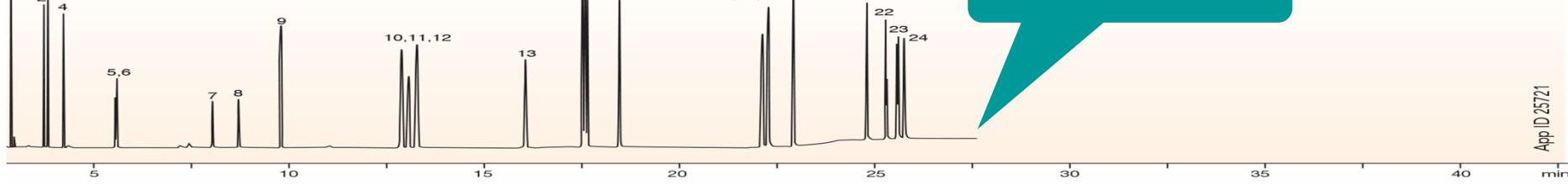
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| 5. Phenanthrene | 17. Benzo[a]pyrene |
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| 7. Flouranthene | 19. Dibenz[a,h]anthracene |
| 8. Pyrene | 20. Benzo[g,h,i]perylene |
| 9. Benzo[c]fluorene | 21. Dibenzo[a,l]pyrene |
| 10. Benz[a]anthracene | 22. Dibenzo[a,e]pyrene |
| 11. Cyclopenta[c,d]pyrene | 23. Dibenzo[a,i]pyrene |
| 12. Chrysene | 24. Dibenzo[a,h]pyrene |

Up to 70% Faster PAH Analysis

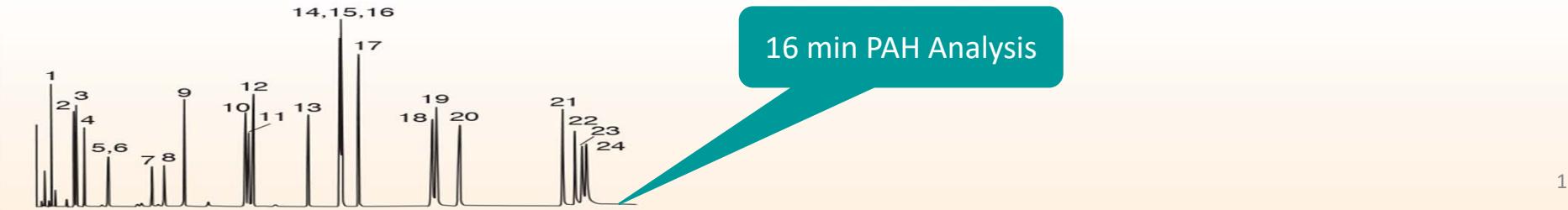
ZB-PAH-EU 30m, 0.25mm, 0.20 µm



ZB-PAH-EU 20m, 0.18mm, 0.14 µm



ZB-PAH-EU 10m, 0.10mm, 0.08µm

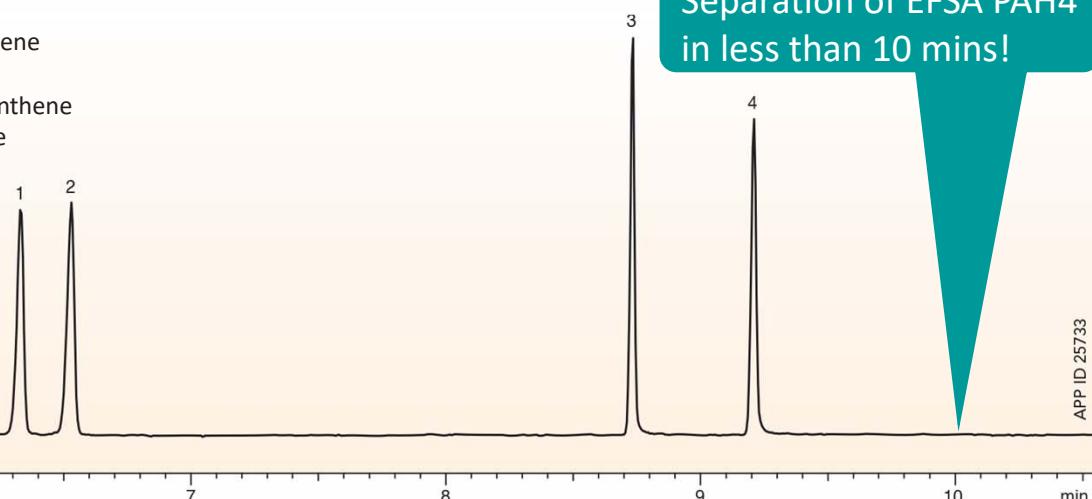




Fast & Accurate PAH Analysis in Food

Sample:

1. Benz[a]anthracene
 2. Chrysene
 3. Benzo[b]fluoranthene
 4. Benzo[a]pyrene



GC-MS Conditions

Column: Zebron ZB-PAH-EU

Dimensions: 10 meter x 0.10 mm x 0.08 µm

Part No.: 7CB-G043-59

Recommended Liner: Zebron PLUS Single Taper Z-Liner™

Liner Part No.: AG2-4B13-05 (for Shimadzu® 2010 GC)

System)

Injection: Split (15:1) @ 320 °C, 1 μL

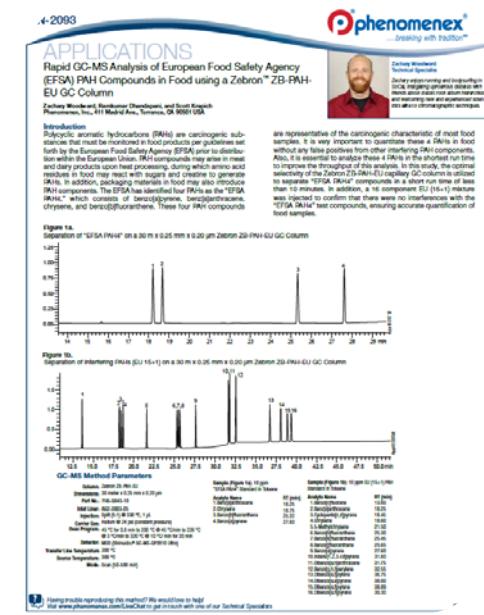
Carrier Gas: Helium @ 0.88 mL/min (constant Flow)

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 330 °C @ 40 °C/min for 5 min

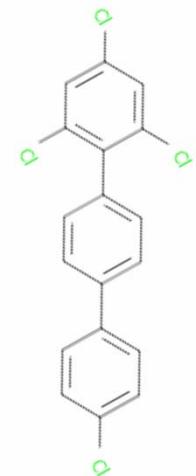
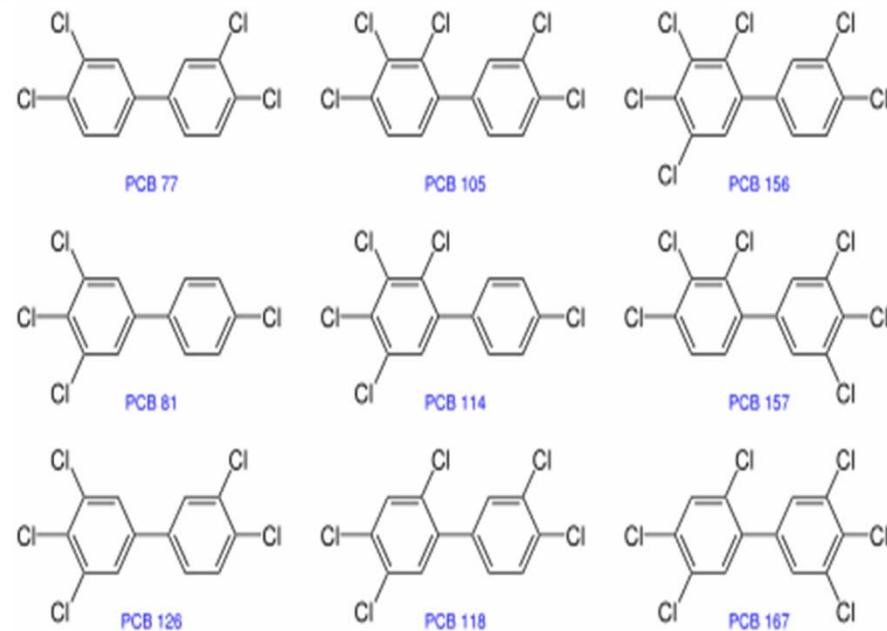
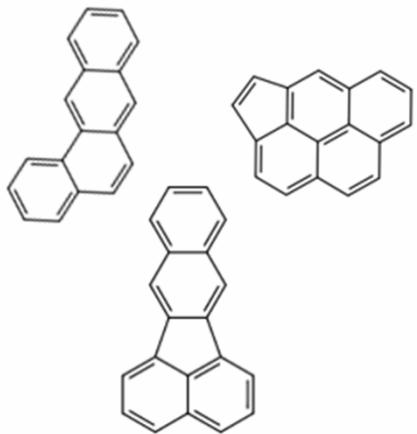
Detector: MSD, 100-500 m/z

Transfer line Temperature: 300 °C

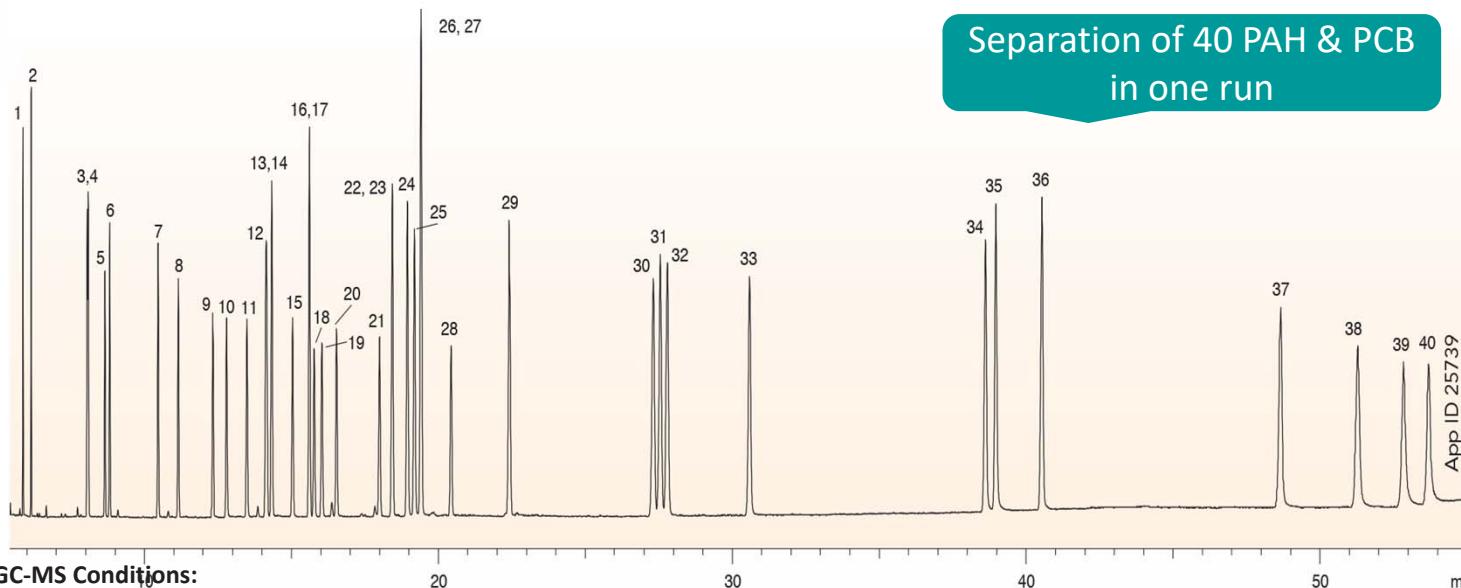
Source Temperature: 300 °C



POPs Compound Classes



PAH & PCB in One Method



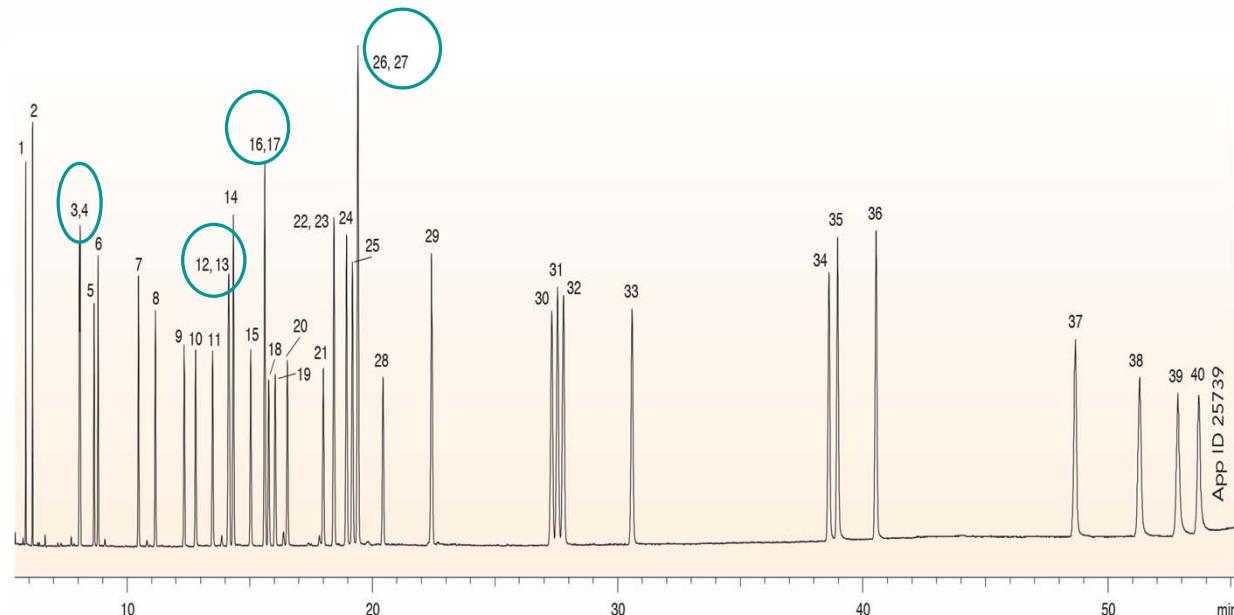
Separation of 40 PAH & PCB
in one run

Sample:

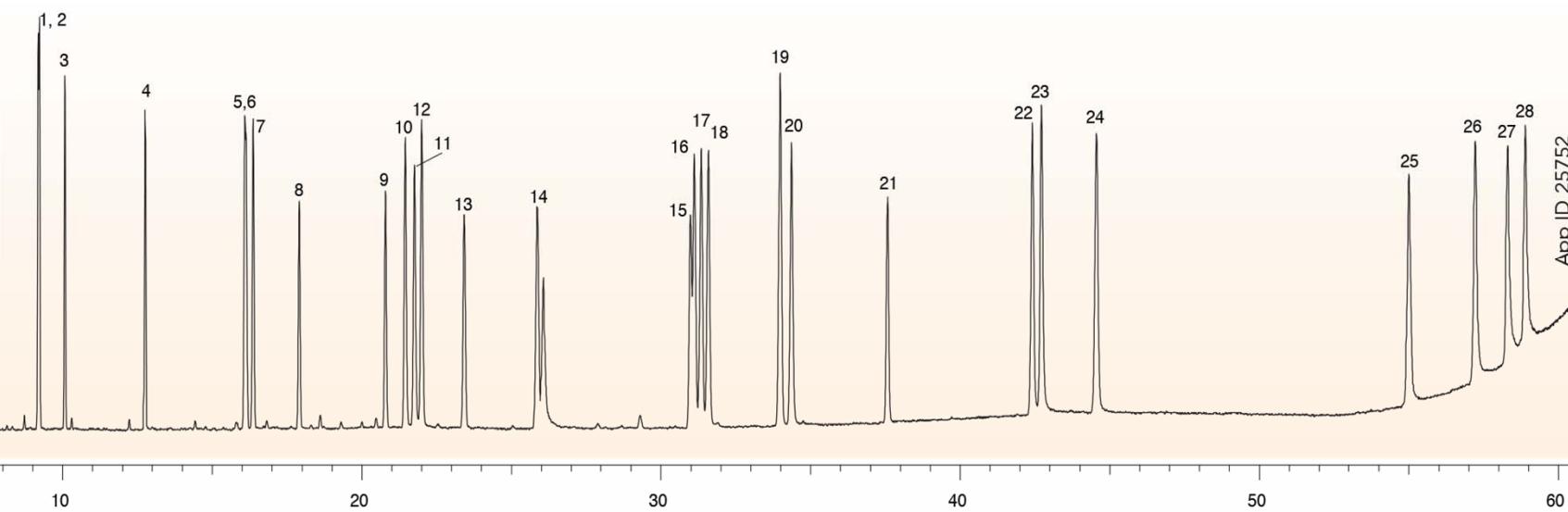
- 1. TCMX
- 2. DCB 22
- 3. PCB 31
- 4. PCB 28
- 5. PCB 69
- 6. PCB 52
- 7. PCB 70
- 8. PCB101
- 9. PCB 81
- 10. PCB 77
- 11. PCB 123
- 12. PCB 153
- 13. PCB 118
- 14. Benzo[c]fluorene
- 15. PCB 105
- 16. PCB 164
- 17. PCB 163
- 18. PCB 138
- 19. PCB 126
- 20. PCB 167
- 21. PCB 156
- 22. PCB 180
- 23. PCB 157
- 24. Benz[a]anthracene
- 25. Cyclopenta[c,d]pyrene
- 26. PCB 169
- 27. Chrysene
- 28. PCB 170
- 29. 5-Methylchrysene
- 30. Benzo[b]fluoranthene
- 31. Benzo[k]fluoranthene
- 32. Benzo[j]fluoranthene
- 33. Benzo[a]pyrene
- 34. Indeno[1,2,3-cd]pyrene
- 35. Dibenz[a,h]anthracene
- 36. Benzo[g,h,i]perylene
- 37. Dibenz[a,l]pyrene
- 38. Dibenz[a,e]pyrene
- 39. Dibenz[a,i]pyrene
- 40. Dibenz[a,h]pyrene

PAH & PCB in One Method

Peak No	Analyte	EI Ion (m/z)	EI Ion (m/z)
3	PCB31	256	186
4	PCB28	256	186
12	PCB118	326	254
13	PCB153	360	290
16	PCB163	360	290
17	PCB164	360	290
22	PCB180	394	359
23	PCB 157	360	290
26	PCB169	360	290
27	Chrysene	228	202

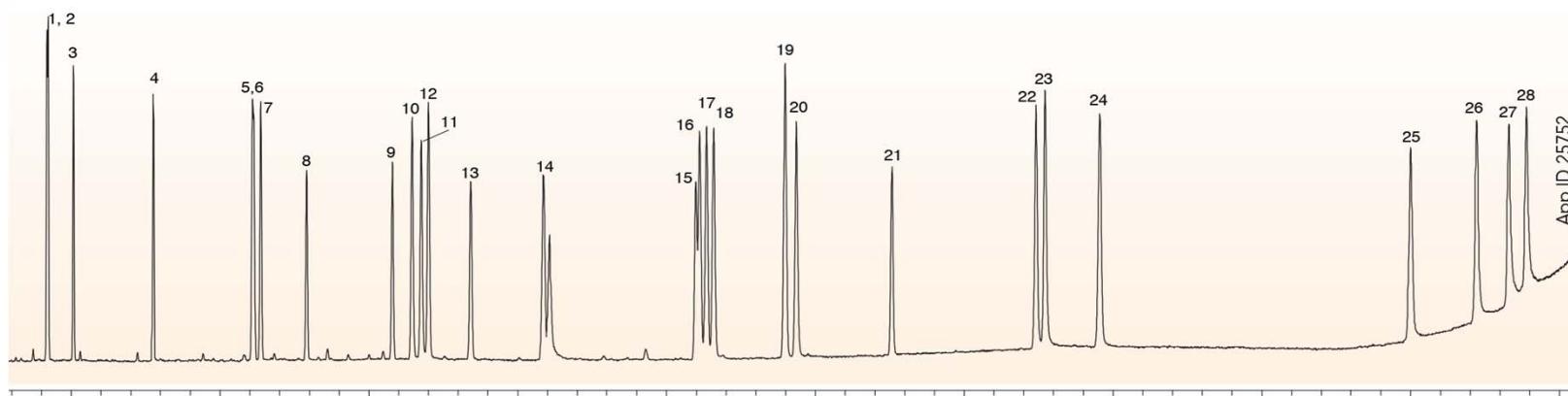


PAH, PCB and Terphenyl in One Method



- Sample:**
1. PCB 31
 2. PCB 28
 3. PCB-52
 4. PCB 101
 5. PCB 153
 6. PCB118
 7. Benzo[c]fluorene
 8. PCB138
 9. PCB180
 10. Benz[a]anthracene
 11. Cyclopenta[c,d]pyrene
 12. Chrysene
 13. Trichlorotriphenyl
 14. 5-Methylchrysene
 15. Tetrachloroterphenyl
 16. Benzo[b]fluoranthene
 17. Benzo[k]fluoranthene
 18. Benzo[j]fluoranthene
 19. Benzo[e]pyrene
 20. Benzo[a]pyrene
 21. Pentachloroterphenyl
 22. Indeno[1,2,3-cd]pyrene
 23. Dibenzo[a,h]anthracene
 24. Benzo[g,h,i]perylene
 25. Dibenzo[a,l]pyrene
 26. Dibenzo[a,e]pyrene
 27. Dibenzo[a,i]pyrene
 28. Dibenzo[a,h]pyrene

PAH, PCB and Terphenyl in One Method



Peak	Analyte	EI Ion (m/z)	EI Ion (m/z)
1	PCB31	256	186
2	PCB28	256	186
5	PCB118	326	254
6	PCB153	360	290
15	Tetrachloroterpheynyl	368	296
16	Benzo[b]fluoranthene	252	224

Sample:

1. PCB 31
2. PCB 28
3. PCB-52
4. PCB 101
5. PCB 153
6. PCB118
7. Benzo[c]fluorene
8. PCB138
9. PCB180
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21. Pentachloroterpheynyl
22. Indeno[1,2,3-cd]pyrene
23. Dibenzo[a,h]anthracene
24. Benzo[g,h,i]perylene
25. Dibenzo[a,l]pyrene
26. Dibenzo[a,e]pyrene
27. Dibenzo[a,i]pyrene
28. Dibenzo[a,h]¹⁸pyrene



Unleash the Power of Fast GC PAH Analysis

ZB-PAH-EU

- Up to 70% Faster PAH Analysis
- Elevated Temperature Stability
- **ZB-PAH-CT**
- Enhanced Resolution for **Chrysene** and **Triphenylene**
- Increased Benzo[b,k]fluoranthene Separation

Factors Influencing Resolution

Master Resolution Equation

$$R_s = \frac{\sqrt{N}}{4} \cdot \frac{a - 1}{a} \cdot \frac{k}{1 + k}$$

Efficiency **Selectivity** **Retention**

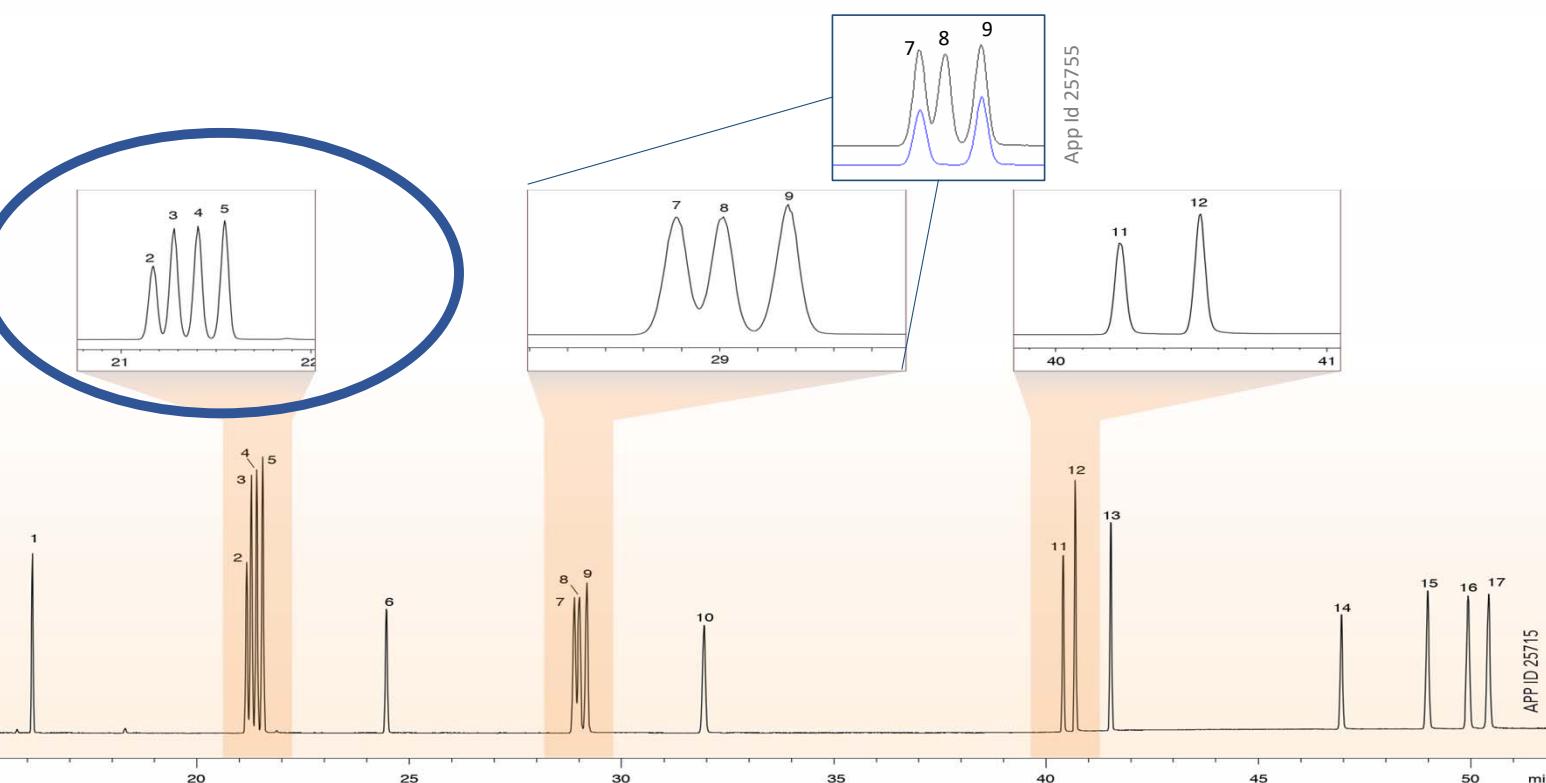
N = theoretical plates

$\alpha = k_2/k_1$

$k = (t_R - t_o)/t_o$

ZB-PAH-CT

Excellent Resolution of Chrysene and Triphenylene



Column: Zebron ZB-PAH-CT

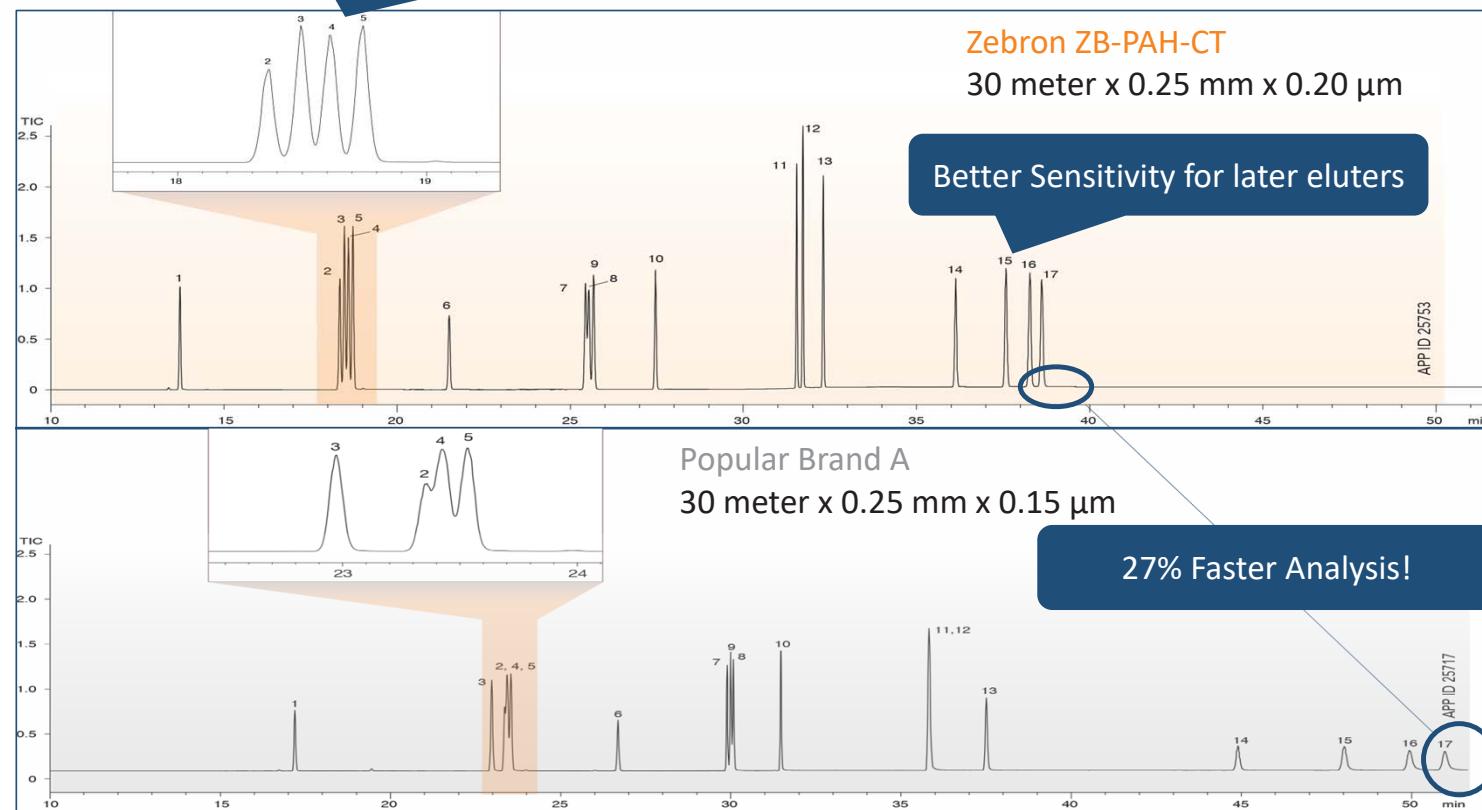
40 meter x 0.18 mm x 0.14 μm

Sample:

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

Great resolution of Chrysene and Triphenylene

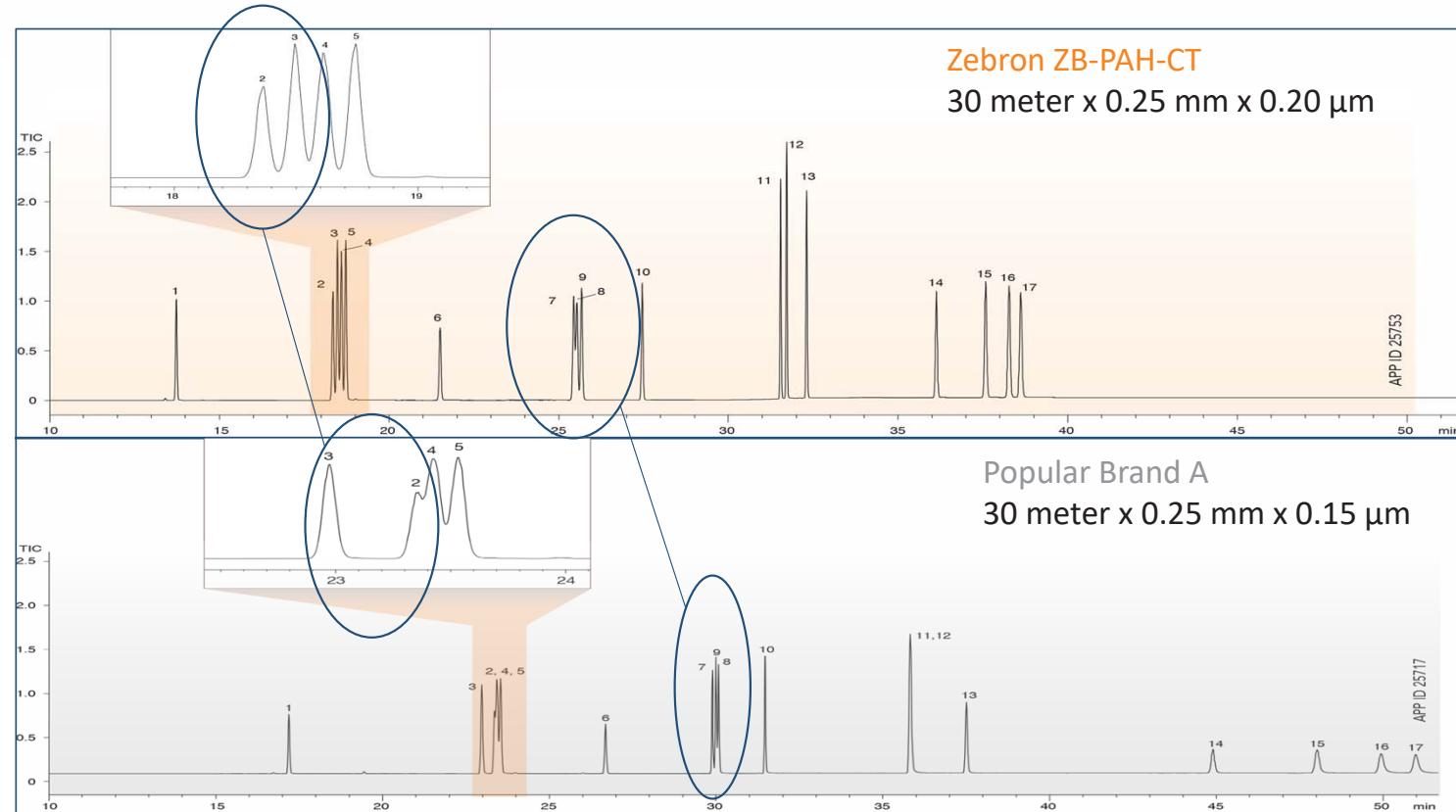
ZB-PAH CT: Shorter Run Time & Better Sensitivity



Sample:

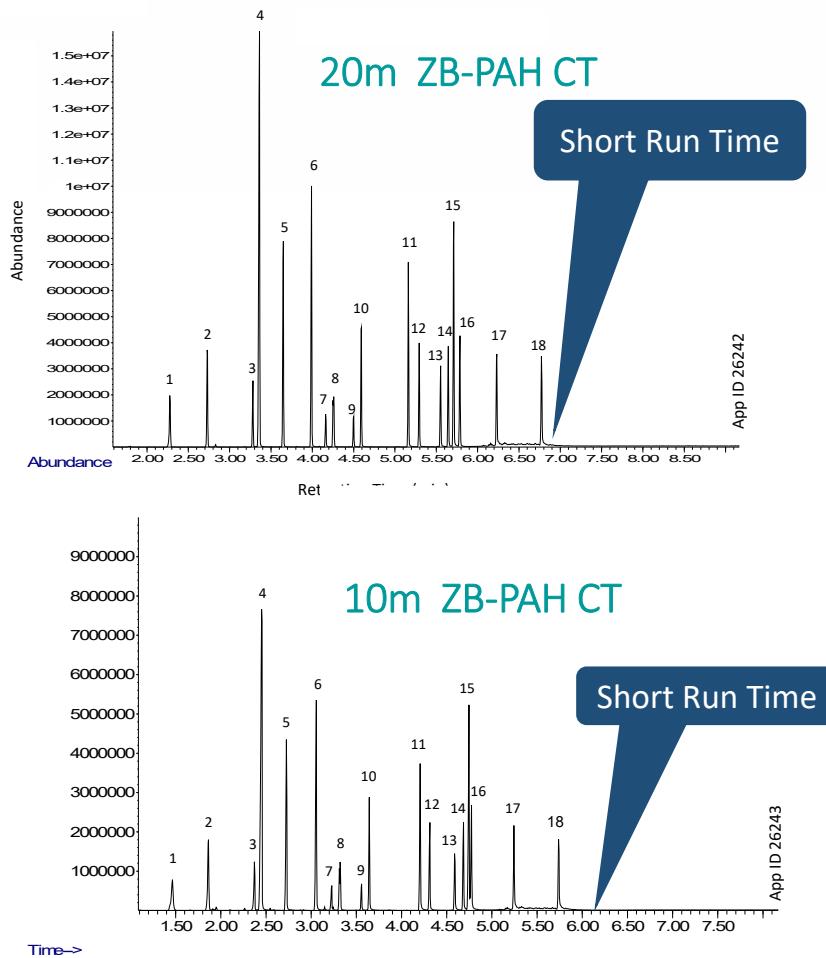
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10. Benzo[a]pyrene
11. Indeno[1,2,3-c,d]pyrene
12. Dibenzo[a,h]anthracene
13. Benzo[g,h,i]perylene
14. Dibenzo[a,l]perene
15. Dibenzo[a,e]perene
16. Dibenzo[a,i]perene
17. Dibenzo[a,h]perene

ZB-PAH CT: Notice Selectivity Change



Sample:

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



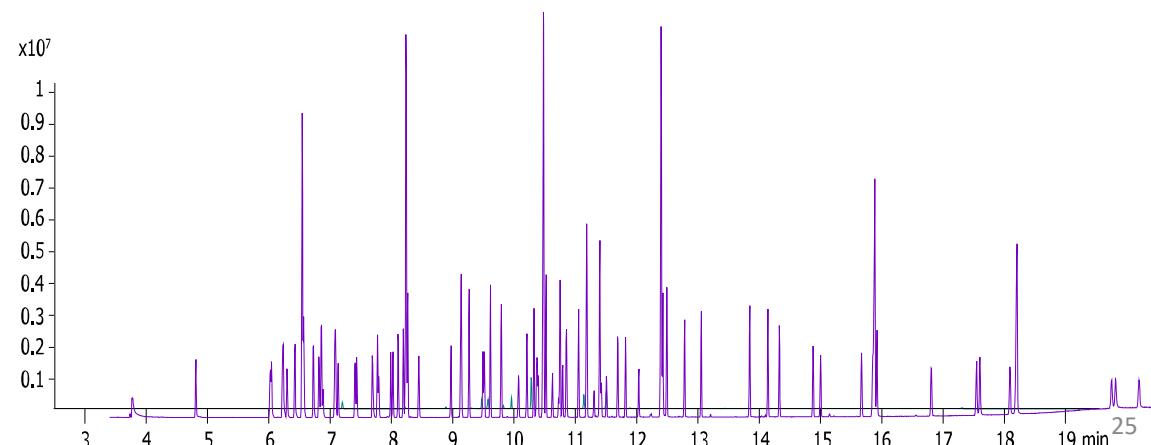
Separation of Phthalate on ZB-PAH CT

#	Analyte	Average RT(min)		%RSD 20m column		Average RT (min)		%RSD 10m Column	
		20m Column	RT	Peak Area	10m column	RT	Peak Area	Peak Area	Area
1	Dimethyl phthalate	2.279	0.02	1.31	1.466	0.02	1.51		
2	Diethyl phthalate	2.731	0.02	1.08	1.864	0.02	1.23		
3	Diallyl phthalate	3.282	0.02	1.21	2.375	0.02	0.83		
4	Dipropyl phthalate	3.361	0.02	9.57	2.454	0.02	0.72		
5	Diisobutyl phthalate	3.649	0	0.7	2.729	0.02	1.51		
6	Dibutyl phthalate	3.991	0	0.69	3.058	0.02	1.23		
7	bis(2-Methoxyethyl)phthalate	4.164	0.01	0.53	3.229	0.02	8.41		
8	bis(4-Methyl-2-pentyl)phthalate	4.261	0.01	0.98	3.325	0.02	0.72		
9	bis(2-Ethoxyethyl)phthalate	4.501	0.01	1.28	3.559	0.01	3.46		
10	Dipentyl phthalate	4.594	0.01	0.67	3.646	0.02	1.22		
11	Dihexyl phthalate	5.164	0.01	0.72	4.208	0.01	0.97		
12	Butylbenzyl phthalate	5.295	0.01	0.48	4.313	0.01	2.42		
13	bis(2-Butoxyethyl)phthalate	5.554	0.02	1.29	4.592	0.02	3.41		
14	Di(ethylhexyl) phthalate	5.646	0.03	0.5	4.687	0.01	0.94		
15	Diheptyl phthalate	5.711	0.04	0.19	4.747	0.01	1.1		
16	Dicyclohexyl phthalate	5.787	0.05	0.47	4.776	0.01	0.6		
17	Di-n-octyl phthalate	6.232	0.09	0.57	5.246	0.02	0.84		
18	Dinonyl phthalate	6.775	0.11	1.26	5.737	0.04	0.73		

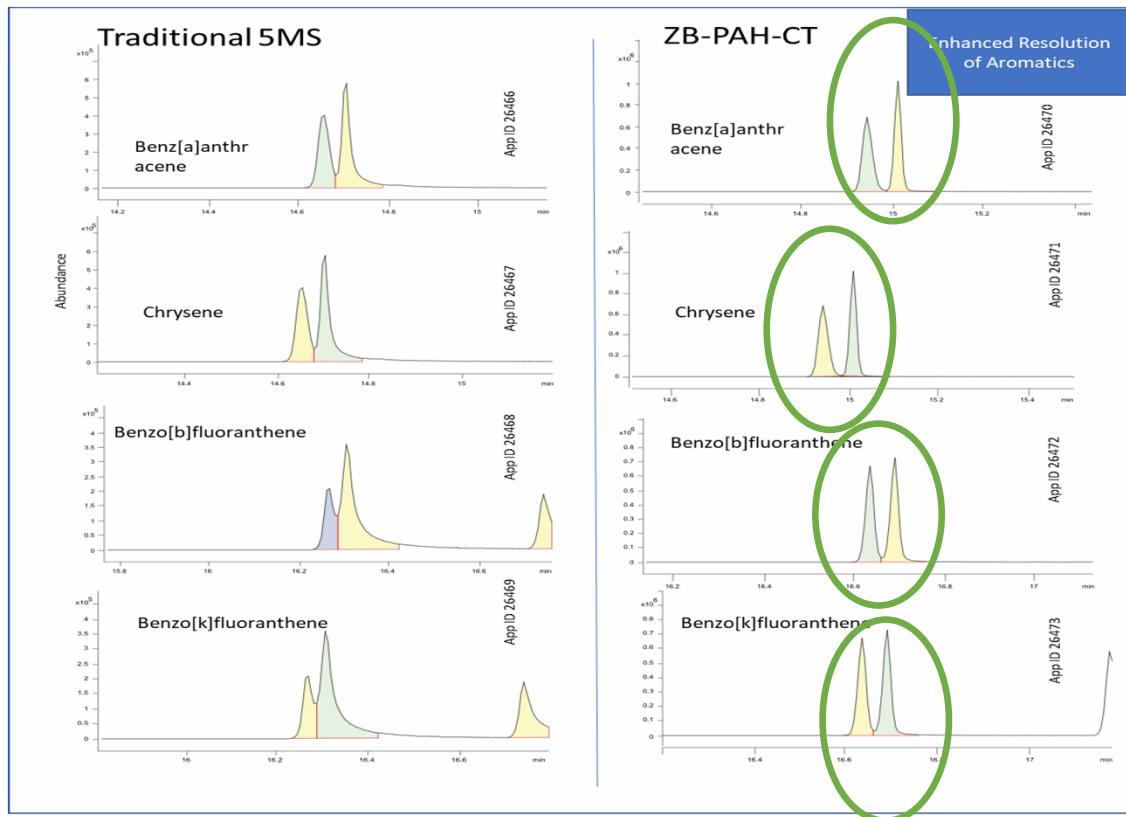
EPA-8270 on ZB-PAH CT

Peak	Analyte Name	RT (min)	Sim Ions (m/z)	Concentration (mg/L)
1	N-Nitrosodimethylamine(NDMA)	2.858	74, 43	8.5558
2	Pyridine	3.896	79,52	10.4750
3	2-Fluorophenol (SSTD)	3.896	111,92, 64	15.1345
4	Phenol-d5 (SSTD)	5.113	99, 71, 42	18.4146
5	Phenol (CC)	5.132	94,66	18.3206
6	Aniline	5.311	93,66	22.6076
7	2-Chlorophenol	5.323	128,64, 130	15.4539
8	bis (2-chloroethyl) ether	5.385	92,63,95	14.3546
9	1,3-Dichlorobenzene	5.515	146, 148, 110	18.6464
10	1,4-Dichlorobenzene (CC)	5.657	146, 148, 110	18.9806
11	1,2-Dichlorobenzene	5.818	146, 148, 111	17.9633
12	Benzyl alcohol	5.904	108, 79, 51	8.6580
13	2-Methylphenol	5.941	107, 108, 79	12.7840
14	Bis (2-chloroisopropyl) ether	5.947	45, 121	9.9582
15	3 and 4-Methylphenols	6.164	107, 108	15.3823
16	Hexachloroethane	6.176	200, 116, 165	6.8428
17	N-Nitro-di-n-propylamine (NDPA) (SP)	6.219	70, 43,130	11.2333
18	Nitrobenzene-d5 (SSTD)	6.497	82, 54, 128	16.0045
19	Nitrobenzene	6.522	77,51, 123	16.0799
20	Isophorone	6.775	82, 138, 54	28.7534
21	2,4-Dimethylphenol	6.862	122, 107, 121	12.9483
22	Benzoic acid	6.862	122, 105,77	ND
23	2-Nitrophenol (CC)	6.88	139, 81, 109	7.8679
24	bis (2-Chloroethoxy) methane	7.078	93, 95, 123	16.9629
25	2,4-Dichlorophenol (CC)	7.115	162, 163, 97	12.7863
26	1,2,4-Trichlorobenzene	7.195	179, 181, 144	15.5531
27	Hexachlorobutadiene (CC)	7.282	224,226, 222	3.7682
28	Naphthalene	7.356	128,129,126	19.4055
29	4-Chloroaniline	7.535	127,129, 65	7.2401
30	4-Chloro-3-methylphenol (CC)	8.06	107,142,144	4.9281

Peak	Analyte Name	RT (min)	Sim Ions (m/z)	Concentration (mg/L)
31	Hexachlorocyclopentadiene (SP)	8.215	236,234,271	3.6914
32	2-Methylnaphthalene	8.227	142,141,115	13.5397
33	1-Methylnaphthalene	8.357	142,141,115	12.7486
34	2,4,6-Trichlorophenol (CC)	8.579	195,197,199	3.3466
35	2,4,5-Trichlorophenol	8.604	195,198,97	3.5454
36	2-Fluorobiphenyl (SSTD)	8.703	172,171,170	14.7471
37	2-Chloronaphthalene	8.882	162,127,164	12.1502
38	o-Nitroaniline	9.16	65,138,92	2.7961
39	Dimethyl phthalate	9.302	163,77,194	13.3250
40	Acenaphthylene	9.413	152,151,153	17.7443
41	2,6-Dinitrotoluene	9.469	165,63,89	2.8922



EPA-8270 on ZB-PAH CT



Enhanced Resolution of Aromatic Compounds

Summary

Polycyclic Aromatic Hydrocarbons

- PAH Terminologies & Regulations
- Challenges with PAH Analysis

ZB-PAH-EU

- Complete resolution of EU15+1 and EPA 610 PAHs
- Speed up your PAH Analysis
- PAH in Food
- One Column for PAH & PCB

ZB-PAH-CT

- Unique Selectivity for Chrysene & Triphenylene
- Short Run Time & better Sensitivity
- Phthalates in Food
- EPA-8270 Method Resolution Improvement

Thank You



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