Resolving 18 PAHs, Including Benzofluoranthenes, Using the Unique Selectivity of a 50% Phenyl Methylpolysiloxane-Phased GC Column

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Key Words
PAH, GC-MS, selectivity, fluoranthenes

Abstract
One of the major difficulties in analyzing polycyclic aromatic hydrocarbons (PAHs) by GC-MS is achieving baseline resolution for isobaric compounds. In demonstrating the separation of 18 PAHs using a Thermo Scientific™ TraceGOLD™ TG-17SilMS GC column, the unique selectivity of this stationary phase is shown to fully resolve benzo[j]fluoranthene from benzo[b] fluoranthene and benzo[k]fluoranthene.

Introduction
The GC column most commonly used for PAH analysis is a low polarity 5% diphenyl/95% dimethyl polysiloxane (equiv to 5SilMS) stationary phase. This can separate a 16 priority PAH standard mix according to US EPA Method 610. However, when two EU PAHs, benzo[e] pyrene and benzo[j]fluoranthene are added to the mix, benzo[j]fluoranthene co-elutes with two other isobaric components benzofluoranthenes b and k. These three challenging isobaric compounds are difficult to resolve by GC-MS.

The stationary phase in the TraceGOLD TG-17SilMS GC column, unlike any other 50% phenyl methylpolysiloxane (17MS) phase, is optimized to give unique selectivity to separate isomeric pairs as well as isobaric PAHs using GC-MS. Thus, the column provides adequate resolution for these critical pairs.

The resolution of benzo[j]fluoranthenes from benzofluoranthenes b and k, and the separation of 18 PAHs on a TraceGOLD TG-17SilMS GC column is demonstrated. This low bleed column offers more resolving power for the separation of critical pairs and isobaric compounds compared to a 5% diphenyl/95% dimethyl polysiloxane stationary phase GC column.

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Experimental Details

Consumables		Part Number
Columns:	TraceGOLD TG-17SilMS, 30 m × 0.25 mm × 0.25 µm	26072-1420
	5% diphenyl/95% dimethylpolysiloxane (equiv to 5SilMS), 30 m × 0.25 mm × 0.25 µm	
Septum:	Thermo Scientific BTO, 17 mm	31303211
Liner:	Thermo Scientific™ Splitless FocusLiner™ for 50 mm needle, 5 × 8 × 105 mm	453T2999
Column ferrules:	100% graphite ferrules for Thermo Scientific™ TRACE™ injector 0.1–0.25 mm i.d.	29053488
Column ferrules:	Graphite/Vespel® for transfer line 0.1–0.25 mm i.d.	29033496
Injection syringe:	10 µL fixed needle syringe for Thermo Scientific™ TriPlus™ RSH Autosampler	365D0291
Vials and closures:	Thermo Scientific™ National™ Target™ DP (9 mm) wide opening screw thread vial	C4000-1W
	Thermo Scientific National 9 mm screw caps and septa, red PTFE/white silicone/red PTFE	C5000-53B

Sample Preparation

Benzo[j]fluoranthene and benzo[e]pyrene were added to a 16 PAH mix according to US EPA Method 610 at a concentration of 10 µg/mL. The standard was prepared in hexane for GC-MS analysis.

GC-MS Conditions

Instrumentation:	
GC:	Thermo Scientific™ TRACE GC Ultra™
Autosampler:	Thermo Scientific™ TriPlus RSH™
Detector type:	Thermo Scientific™ ISQ™ mass spectrometer
Carrier gas:	Helium
Column flow:	1.2 mL/min, constant flow
Oven temperature:	
TG-17SilMS:	90 °C (1.0 min), 30 °C/min, 250 °C, 4 °C/min, 330 °C (5 min)
Equivalent to 5SilMS:	90 °C (1.0 min), 25 °C/min, 280 °C, 4 °C/min, 320 °C (2 min)
Injector type:	Split/Splitless
Injector mode:	Split 25:1, 30 mL/min split flow
Injector temperature:	250 °C
Transfer line temperature:	300 °C
Source temperature:	250 °C
Ionization conditions:	EI
Electron energy:	70 eV
Emission current:	50 µA
Solvent delay time:	2.8 min
Scan range:	40–450 amu full scan
Injection volume:	1 µL

Data Processing

Data was acquired and processed using Thermo Scientific™ Xcalibur™ software

Results

Figure 1 shows the separation of 18 PAHs on a TraceGOLD TG-17SilMS GC column, and Figure 2 shows the comparison with an equivalent phase to a 5SilMS phased GC column. Due to their isobaric status, the compounds benzofluoranthenes b, k, and j require chromatographic separation. To do this, the unique selectivity provided by the TG-17SilMS capillary column is required as shown in Figure 1. When compared to a 5SilMS GC column (Figure 2), the TG-17SilMS column fully separates the three isobaric compounds.

Comparing the selectivity for peaks 16 and 17, there is co-elution with the TG-17SilMS GC column; however, they can be resolved by using the mass spectrometer based on their difference in molecular weights. The equivalent 5SilMS in Figure 2 shows isomeric separation of compounds 16 and 17.

The stationary phase in the TG-17SilMS column is designed to give low bleed at high temperatures as shown in Figure 1. This is ideal for analyzing heavy, late-eluting PAHs at high boiling points.

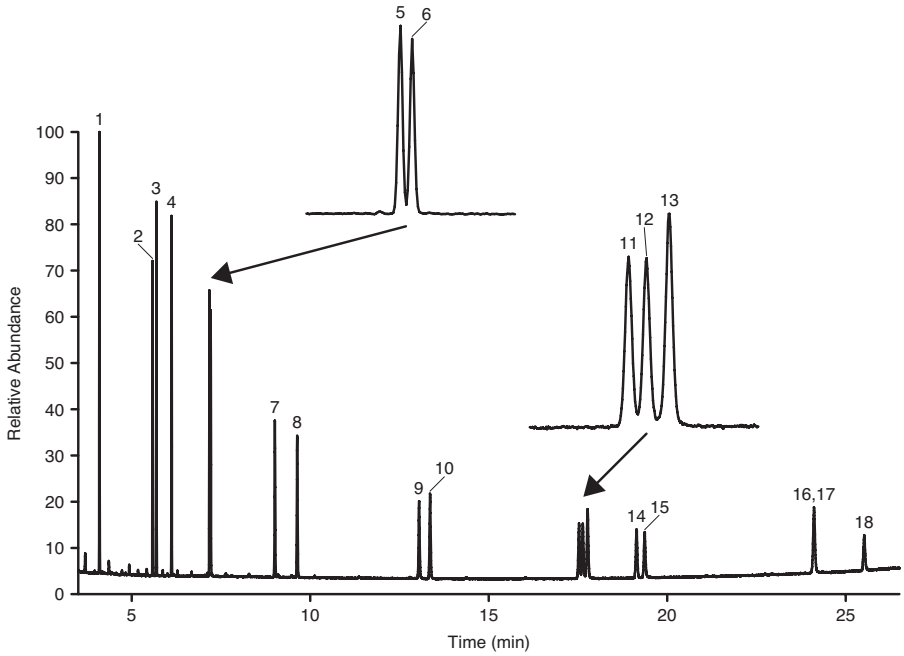

Figure 1: TIC chromatogram for 10 µg/mL 18 component PAH mixture on a TG-17SilMS 30 m × 0.25 mm × 0.25 µm GC column

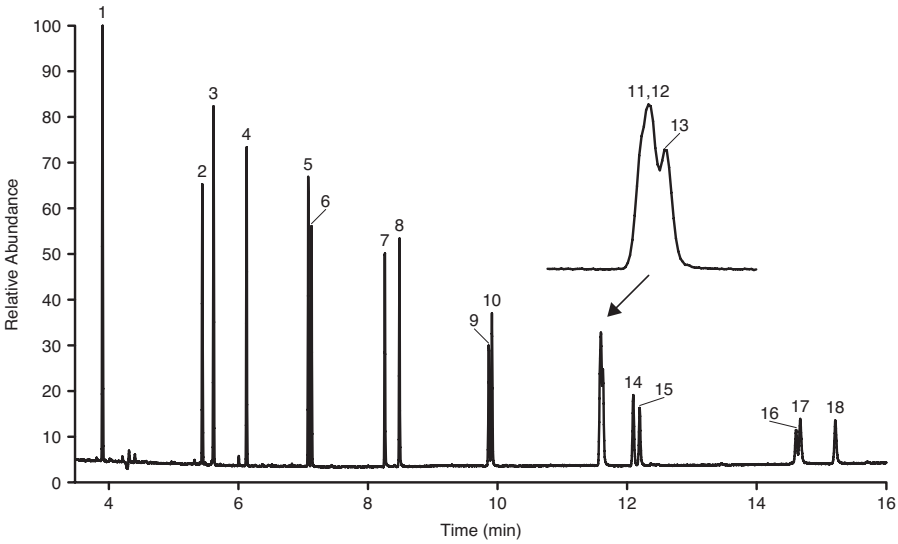

Figure 2: TIC chromatogram for 10 µg/mL 18 component PAH mixture on an equiv 5SilMS phased 30 m × 0.25 mm × 0.25 µm GC column

Peak	Compound	TG-17SilMS t_R (min)	Equiv to 5SilMS t_R (min)
1	Napthalene	4.10	3.91
2	Acenaphthylene	5.59	5.44
3	Acenapthene	5.69	5.62
4	Fluorene	6.12	6.13
5	Phenanthrene	7.18	7.08
6	Anthracene	7.21	7.13
7	Fluoranthene	9.01	8.26
8	Pyrene	9.63	8.49
9	Benzo[a]anthracene	13.05	9.86
10	Chrysene	13.36	9.91
11	Benzo[b]fluoranthene	17.53	11.59
12	Benzo[j]fluoranthene	17.64	11.59
13	Benzo[k]fluoranthene	17.77	11.62
14	Benzo[a]pyrene	19.14	12.09
15	Benzo[e]pyrene	19.37	12.20
16	Indeno[1,2,3-cd]pyrene	24.11	14.61
17	Dibenzo[a,h]anthracene	24.11	14.67
18	Benzo[g,h,i]perylene	25.52	15.21

Table 1: Peak identification according to retention times for 10 µg/mL 18 component PAHs

Conclusion

The TraceGOLD TG-17SilMS GC column can resolve the three challenging isobaric PAHs, the benzofluoranthenes b,k and j. This column can also resolve other isobaric pairs such as phenanthrene and anthracene. Since the critical pair indeno[1,2,3-cd]pyrene (peak 16) and dibenzo[a,h]anthracene (peak 17) are not isobaric, they are easily resolved by a mass spectrometer.

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