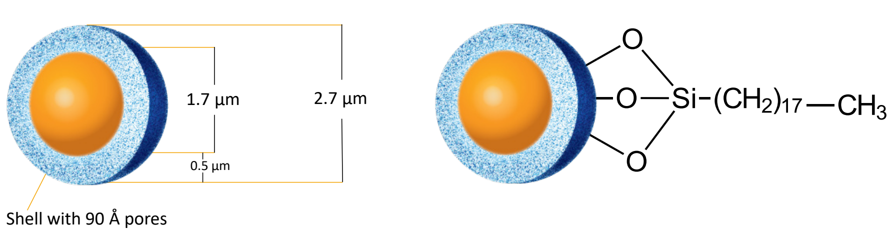
Introducing HALO® PAH

INTRODUCTION

Polycyclic aromatic hydrocarbons (PAHs) are a class of organic compounds that consist of multiple rings containing only carbon and hydrogen. These compounds are produced naturally (e.g. petroleum seeps, forest fires, volcanoes) or anthropogenically by incomplete combustion or high-pressure processes, such as burning coal, oil, gasoline, trash, tobacco, and wood. Grilling meats can also form PAHs. These compounds are ubiquitous and human exposure can cause irritation, mutation, and cancer. The most common way for non-smokers to be exposed to PAHs is through eating PAH contaminated food while smokers are exposed through smoking. Due to the negative health effects, government agencies have established methods for detection and reporting. PAHs are often screened in panels of 18 compounds or more by environmental laboratories using regulated HPLC methods. The 16 compounds that are specified in EPA 610 and EPA 8310 are shown in Table 1 (next page).

HALO® PAH columns with trifunctional C18 bonded phase have been specifically designed to provide fast, efficient, selective separations of PAH compounds. This un-endcapped phase is built on the 2.7 µm Fused-Core® particle with a 1.7 µm solid silica core, 0.5 µm shell and 90 Å pores as shown in Figure 1.

Figure 1. HALO® PAH 2.7 µm particle design and trifunctional C18 ligand



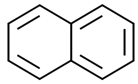
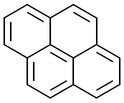
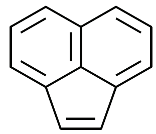
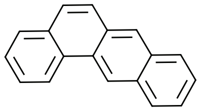
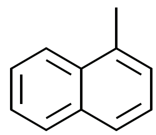
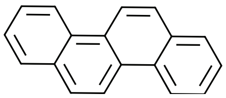
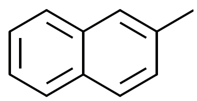
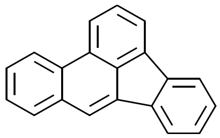
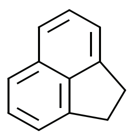
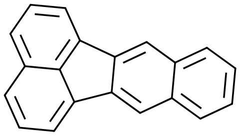
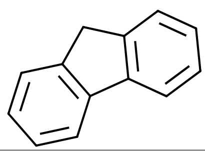
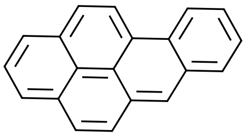
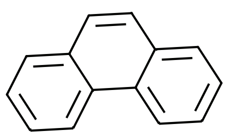
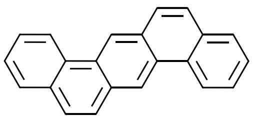
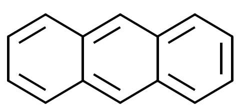
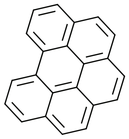
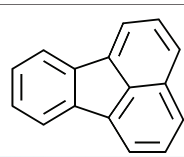
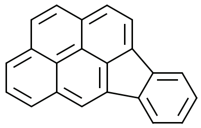
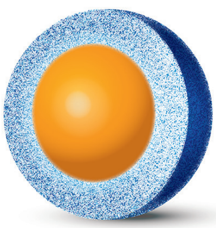
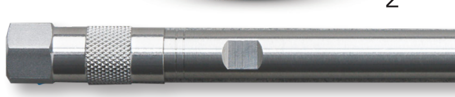
Peak #	Name	Structure	Peak #	Name	Structure
1	Naphthalene		10	Pyrene	
2	Acenaphthylene		11	Benzo[a] anthracene	
3	1-methylnaphtha-lene		12	Chrysene	
4	2-methylnaphtha-lene		13	Benzo[b]flu-oranthene	
5	Acenaphthene		14	Benzo[k] fluoranthene	
6	Fluorene		15	Benzo[a] pyrene	
7	Phenanthrene		16	Dibenzo[a,h] anthracene	
8	Anthracene		17	Benzo[g,h,i] perylene	
9	Fluoranthene		18	Indeno [1,2,3-cd] pyrene	

Table 1. Structures of the 16 PAH compounds specified by EPA 610 and EPA 8310 and two extra compounds that are commonly added (1-methylnaphthalene and 2-methylnaphthalene) listed in elution order.

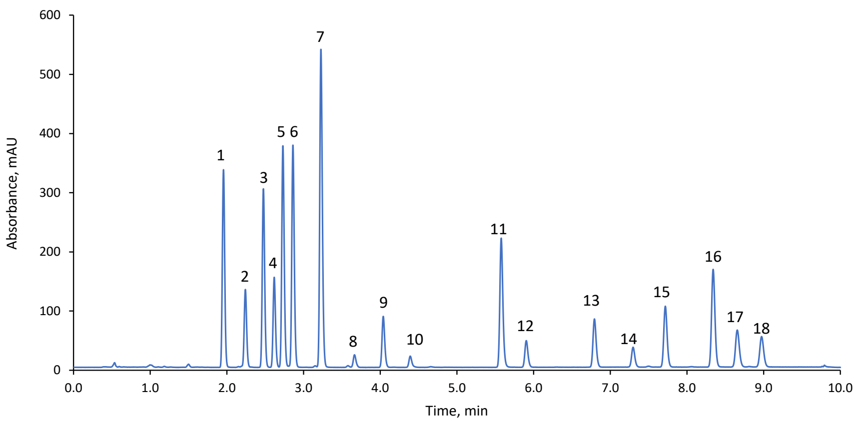




APPLICATIONS

A typical separation of 18 PAH compounds with high resolution is shown in Figure 2.

Figure 2. High resolution separation of 18 PAH compounds on HALO® PAH. Elution order as listed in Table 1.



TEST CONDITIONS:

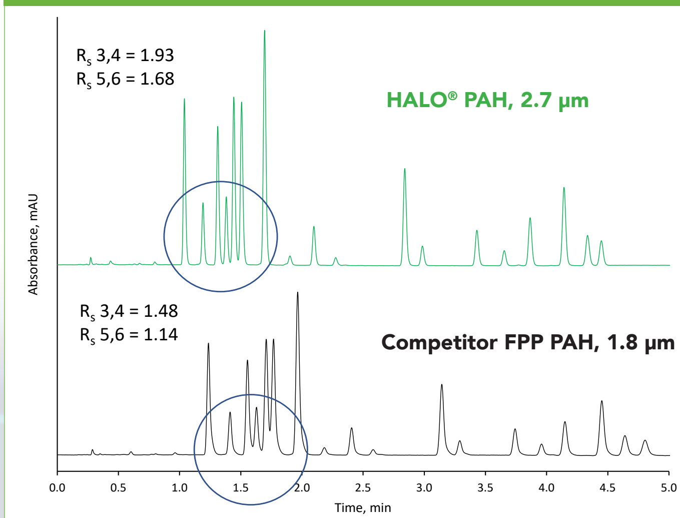
Column: HALO 90 Å PAH, 2.7 µm 3.0 x 100 mm
Part Number: 92843-612
Mobile Phase A: Water
B: Acetonitrile

Gradient:	Time	%B
	0.0	50
	8.0	100
	10.0	100

Flow Rate: 0.77 mL/min
Back Pressure: 263 bar
Temperature: 30 °C
Detection: 280 nm
Injection Volume: 2 µL
Sample Solvent: Methanol
Data Rate: 100 Hz
Response Time: 0.025 sec.
Flow Cell: 1 µL
LC System: Shimadzu Nexera

This 10 minute gradient is 3 times faster than the method conditions suggested in EPA 610, which means higher throughput for laboratories that have many samples to analyze. HALO® PAH outperforms a fully porous particle (FPP) 1.8 µm, 95 Å column for a fast 5 min separation using 4.6 x 50 mm columns as shown in Figure 3.

Figure 3. Comparison of HALO® PAH to FPP PAH for a separation of 18 PAHs using a 5 minute gradient. Elution order as listed in Table 1.



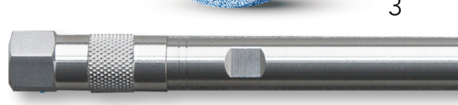
TEST CONDITIONS:

Column: HALO 90 Å PAH, 2.7 µm 4.6 x 50 mm
Part Number: 92844-412
Competitor Column: FPP PAH, 1.8 µm 4.6 x 50 mm
Mobile Phase A: Water
B: Acetonitrile

Gradient:	Time	%B
	0.0	50
	4.0	100
	5.0	100
	5.01	50

Flow Rate: 1.8 mL/min
HALO® Pressure: 256 bar
Competitor Back Pressure: 344 bar
Temperature: 30 °C
Detection: 280 nm
Injection Volume: 2 µL
Sample Solvent: Methanol
Data Rate: 100 Hz
Response Time: 0.025 sec.
Flow Cell: 1 µL
LC System: Shimadzu Nexera

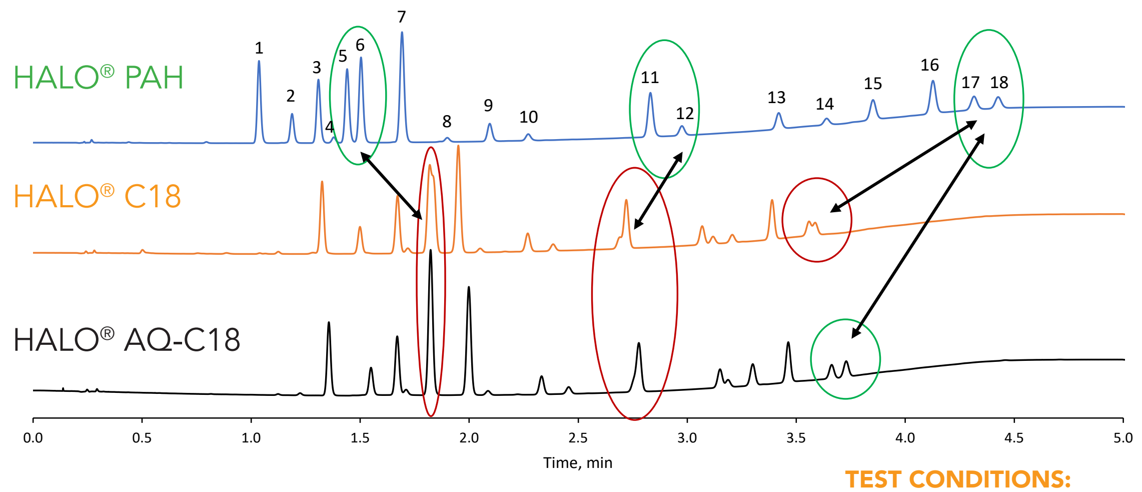
The resolution between peaks 3 and 4 is 1.9 for the HALO® PAH column compared to 1.48 for the FPP column while the resolution between peaks 5 and 6 is 1.68 for the HALO® PAH column compared to 1.14 for the FPP column.



PHASE COMPARISON

Some people may wonder what is so unique about the HALO® PAH. Will a HALO® C18 or HALO® AQ-C18 work just as well for PAH separations? C18 phases are often the first tried during method development. In Figure 4, it is clear that the selectivity of HALO® PAH is different from both HALO® C18 and HALO® AQ-C18.

Figure 4. Comparison of the selectivity of HALO® PAH to HALO® C18 and HALO® AQ-C18. Elution order as listed in Table 1.



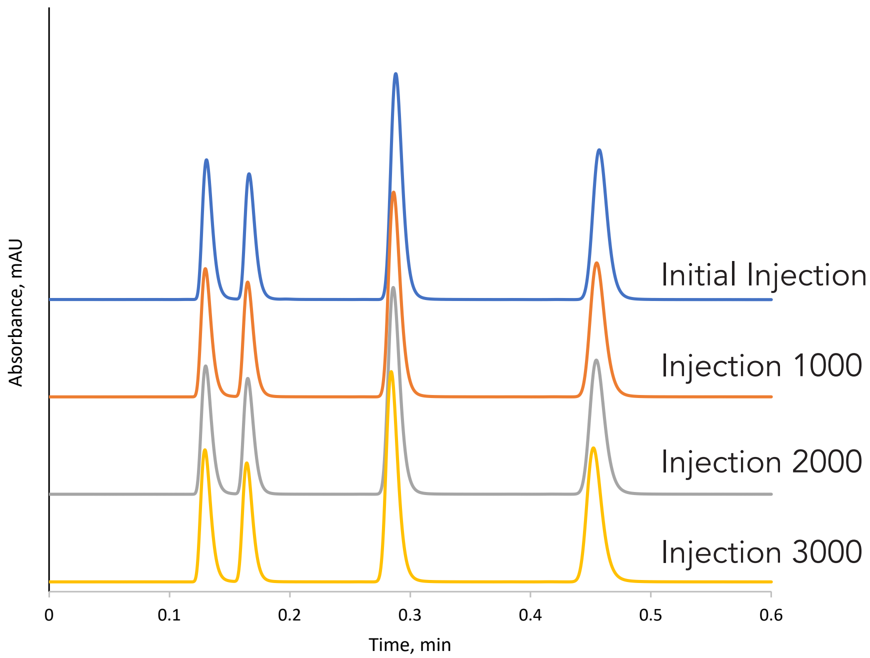
TEST CONDITIONS:
CONDITIONS SAME AS FIGURE 3

The unique shape selectivity of HALO® PAH is needed for the separations of the compounds acenaphthene and fluorene (peaks 5 and 6) as well as benzo[a]anthracene and chrysene (peaks 11 and 12). Furthermore, benzo[g,h,i]perylene and indeno[1,2,3-cd]pyrene (peaks 17 and 18) are coeluted on HALO® C18, but resolved on both HALO® PAH and HALO® AQ-C18. It is interesting to note that HALO® PAH is less retentive for the lower molecular weight compounds and more retentive for the higher molecular weight compounds compared to HALO® C18 and HALO® AQ-C18. As the HALO® C18 is a trifunctional phase containing more C18 on the surface, this is one explanation for the observed retention difference.

PERFORMANCE YOU CAN RELY ON

While the conditions for PAH analysis are normally unbuffered gradients using acetonitrile and water at ambient temperature, the HALO® PAH column was stability tested at pH 2 and 60 °C to demonstrate its ruggedness. See Figure 5.

Figure 5. Stability test results of 3000 Injections on HALO® PAH at pH 2 and 60 °C.

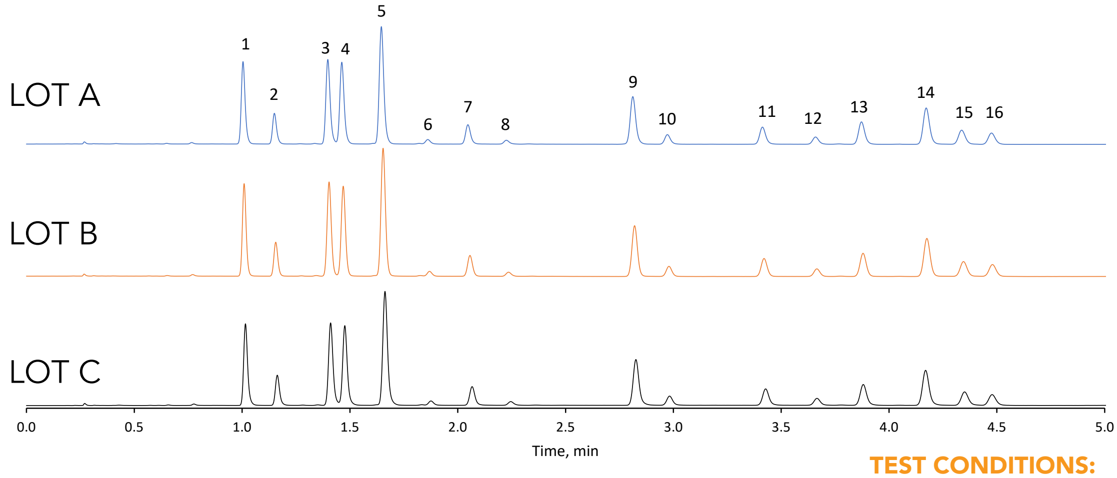


TEST CONDITIONS:
Column: HALO 90 Å PAH, 2.7 µm 2.1 x 50 mm
Part Number: 92842-412
Mobile Phase A: Water/0.1% TFA
B: Acetonitrile/0.1% TFA
Isocratic: 50% B
Flow Rate: 0.7 mL/min
Back Pressure: 169 bar
Temperature: 60 °C
Detection: 254 nm
Injection Volume: 0.5 µL
Peak Identities: uracil, phenol, 1-chloro-4-nitro-benzene, naphthalene (in elution order)
Sample Solvent: 60/40 ACN/ Water
Data Rate: 100 Hz
Response Time: 0.025 sec.
Flow Cell: 1 µL
LC System: Shimadzu Nexera

After 3000 injections, the column did not experience any significant loss of plates nor did the retention change. Additionally, there was no increase in back pressure. This shows that the column will have a long lifetime using the usual conditions for PAH analysis. In delivering an applications specific product to the environmental market, this is an especially important design criteria as extended column lifetime and reliable performance with operation at standard LC back pressures are often required.

HALO® products are manufactured according to tightly controlled procedures and HALO® PAH is no exception. See Figure 6 for an example of the excellent lot to lot reproducibility of HALO® PAH.

Figure 6. HALO® PAH comparison of three different lots showing excellent reproducibility. Peak elution order is as listed in Table 1, but mix does not contain 1-methylnaphthalene and 2-methylnaphthalene.



TEST CONDITIONS:
CONDITIONS SAME AS FIGURE 3

CONCLUSIONS

With its highly selective stationary phase, excellent peak shapes, and baseline resolution, the benefits of a Fused-Core® particle design are now available for PAH analysis. The HALO® PAH column is an excellent choice for laboratories analyzing food and water samples for PAH compounds.

ORDERING INFORMATION

HALO® PAH SPECIFICATIONS	ANALYTICAL COLUMN PART NUMBERS		GUARD COLUMN PART NUMBERS (3-PACK)	
Particle Size: 2.7 µm	ID x Length (in mm)	PN	ID x Length (in mm)	PN
Pore Size: 90 Å	2.1 x 50	92842-412	2.1 x 5	92842-112
Surface Area: 135 m²/g	2.1 x 100	92842-612	3.0 x 5	92843-112
Carbon Load: 9.9 %	2.1 x 150	92842-712	4.6 x 5	92844-112
Endcapped: No	3.0 x 50	92843-412	Guard Column Holder	94900-001
Low pH Limit /Max T: 2/60°C	3.0 x 100	92843-612		
High pH Limit/Max T: 9/40°C	3.0 x 150	92843-712		
	4.6 x 50	92844-412		
	4.6 x 100	92844-612		
	4.6 x 150	92844-712		

shop.fused-core.com