

Rapid Screening Method for Polycyclic Aromatic Hydrocarbons Using an Advanced Solid Core UHPLC Column and System Combination

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Key Words

Vanquish, Accucore, PAH, polycyclic aromatic hydrocarbons

Goal

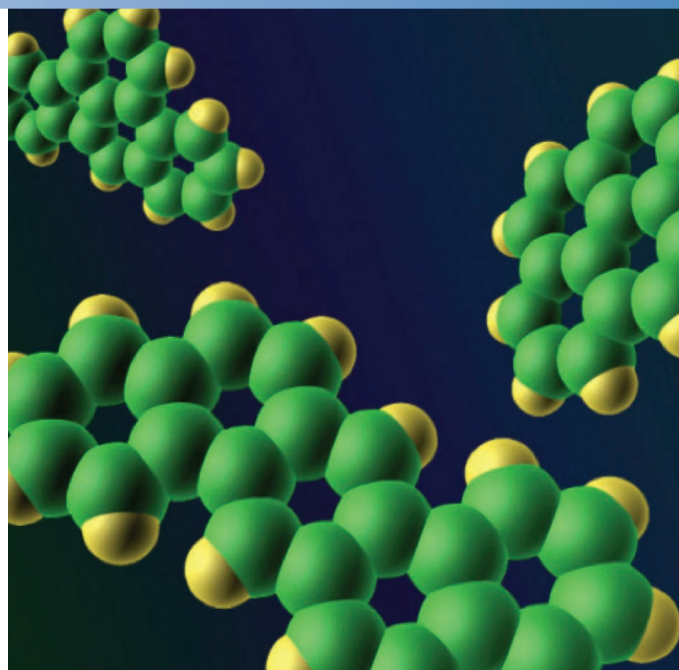
To demonstrate the advantages of using the Thermo Scientific™ Accucore™ Vanquish™ C18+, 1.5 µm column and Thermo Scientific Vanquish UHPLC system for the fast analysis of polycyclic aromatic hydrocarbons (PAHs). The advanced capabilities of the Vanquish UHPLC system allow the Accucore Vanquish columns to be operated at high flow rates that enable development of rapid analytical methods while maintaining high performance.

Introduction

Sixteen polycyclic aromatic hydrocarbons are targeted by the United States Environmental Protection Agency (EPA Method 610) as priority pollutants. These PAHs are often found in soil and sediment and are known to be carcinogenic, mutagenic, and teratogenic. Natural sources include volcanic activity and forest fires, and man-made sources include incomplete combustion of fossil fuels, waste incineration, tobacco smoke, and barbecued meat.

Accucore Vanquish C18+ UHPLC columns use Core Enhanced Technology™ to facilitate fast and highly efficient separations. This next-generation column features 1.5 µm solid core particles that are not totally porous, but instead have a solid core and a porous outer layer. The optimized phase bonding creates a high-coverage, robust phase. This coverage results in a significant reduction in secondary interactions and delivers highly efficient peaks. The tightly controlled 1.5 µm diameter of Accucore Vanquish particles, in combination with controlled manufacturing processes, results in a column that delivers the increased chromatographic performance required for rapid screening methods.

The Accucore Vanquish UHPLC column and Vanquish UHPLC systems were designed in combination to achieve the best possible chromatographic performance. The system is optimized to reduce extra column band dispersion and allow users to significantly improve the separation power in their analytical assays. By exploiting the 1500 bar high pressure capability of the Vanquish UHPLC system, the flow rates can be increased while maintaining peak capacity, resulting in shorter method



times and increased assay throughput. The separation of 16 PAHs using the Accucore Vanquish UHPLC column and Vanquish UHPLC system is demonstrated in this application.

Experimental

Consumables and Apparatus

- Accucore Vanquish C18+, 1.5 μm UHPLC column, 100 \times 2.1 mm (P/N 27101-102130)
- Thermo Scientific™ Virtuoso™ Vial Identification System (P/N 60180-VT-100)
- Thermo Scientific™ Virtuoso™ 9 mm wide opening screw thread vial, 2 mL, clear glass vial with V-patch and red PTFE/white silicone/red PTFE septum (P/N 60180-VT400)
- LC-MS grade 18 M Ω water from Thermo Scientific™ Smart2Pure™ system (P/N 50129845)
- Fisher Scientific™ HPLC grade methanol (P/N M/4056/17)
- Fisher Scientific HPLC grade acetonitrile (P/N A/0626/17)

Sample Preparation

PAH calibration mix (Sigma-Aldrich®) contained 10 $\mu\text{g}/\text{mL}$ of each of the following components in acetonitrile: naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benzo[*a*]anthracene, chrysene, benzo[*b*]fluoranthene, benzo[*k*]fluoranthene, benzo[*a*]pyrene, dibenzo[*a,b*]anthracene, benzo[*g,b,i*]perylene, and indeno[1,2,3-*cd*]pyrene.

Vial labeling was supported by the Virtuoso Vial Identification System.

Instrumentation

Analyses were performed using a Vanquish UHPLC System consisting of:

- System base (P/N VH-S01-A)
- Binary pump H (P/N VH-P10-A)
- Split sampler HT (P/N VH-A10-A)
- Column compartment H (P/N VH-C10-A)
- Diode array detector HL (P/N VH-D10-A)

UHPLC Conditions

UHPLC column Accucore Vanquish C18+, 1.5 μm , 100 \times 2.1 mm

Mobile phase A Methanol/water (50:50 v/v)

Mobile phase B Acetonitrile

Flow rate 500 $\mu\text{L}/\text{min}$

Column temp. 40 $^{\circ}\text{C}$, still air with eluent pre-heating

Injection details 2 μL standard needle in loop

UV detection 254 nm

Table 1. LC gradient conditions.

Time (min)	% B
0.00	20
0.03	50
4.50	100
8.00	20
10.00	20

Software

The Thermo Scientific™ Chromeleon™ 7.2 SR2 Chromatography Data System was used for data acquisition and analysis.

Results and Discussion

By exploiting the high pressure capabilities of the Vanquish UHPLC system, in conjunction with the Accucore Vanquish UHPLC column and a simple binary gradient, it was demonstrated that a screening method for 16 compounds within a 7.5 minute detection window (and a full method cycle time of 10 minutes) can be achieved (Figure 1 and Table 2). Baseline resolution for the critical pairs chrysene/benzo[*a*]anthracene and indeno[1,2,3-*cd*]pyrene/benzo[*g,b,i*]perylene has not been achieved; however, as a screening method, sufficient resolution to allow identification prior to conformation analysis is demonstrated.

Using a 500 $\mu\text{L}/\text{min}$ flow rate, the maximum system pressure was 1197 bar. The Vanquish UHPLC system and Accucore Vanquish UHPLC column are able to routinely operate at these pressure conditions.

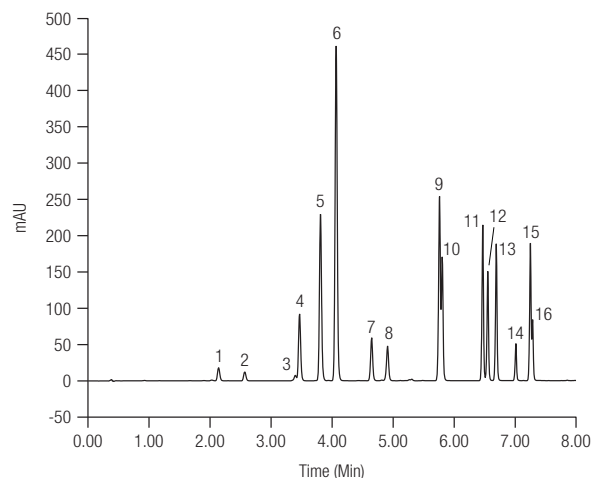


Figure 1. Chromatogram showing separation of 16 PAHs within a 7.5 minute detection window.

Table 2. Peak identification for 16 PAHs.

Peak Number	Analyte Name	Retention Time (min)	% RSD of Retention Time (n=6)
1	Naphthalene	2.14	0.18
2	Acenaphthylene	2.57	0.13
3	Acenaphthene	3.39	0.060
4	Fluorene	3.46	0.060
5	Phenanthrene	3.81	0.043
6	Anthracene	4.06	0.045
7	Fluoranthene	4.65	0.036
8	Pyrene	4.91	0.054
9	Chrysene	5.76	0.054
10	Benzo[a]anthracene	5.80	0.046
11	Benzo[b]fluoranthene	6.47	0.035
12	Benzo[k]fluoranthene	6.55	0.036
13	Benzo[a]pyrene	6.69	0.038
14	Dibenzo[a,h]anthracene	7.01	0.033
15	Indeno[1,2,3-cd]pyrene	7.25	0.035
16	Benzo[g,h,i]perylene	7.28	0.035

Conclusion

The performance of the Accucore Vanquish UHPLC column coupled with the low internal volume and advanced capabilities of the Vanquish UHPLC system deliver a rapid screening UHPLC method for 16 PAHs with a method time of 10 minutes.

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