NUCLEODUR® C_{18} PAH special octadecyl phase for PAH analysis · USP L1

Technical data

- Base material NUCLEODUR® silica, particle sizes 1.8 and 3 µm, pore size 110 Å; polymeric coating

Recommended application

- Allows efficient gradient separation of the 16 PAHs according to EPA

Analysis of 16 EPA PAHs with or without acetonitrile

MN Appl. Nos. 123820 / 123830

Separation with acetonitrile

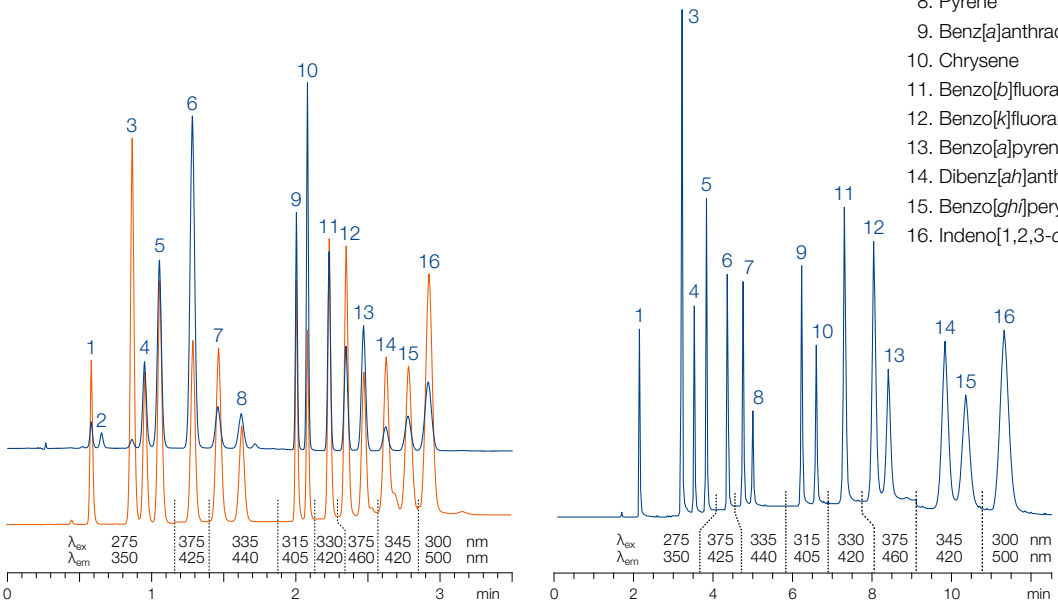
Column:	100 x 4 mm NUCLEODUR® C18 PAH, 3 µm
Eluent:	A) methanol – water (80:20, v/v) B) acetonitrile 2–20 % B in 1.2 min, 20–100 % B in 0.5 min, 100 % B for 2.5 min, 100–2 % B in 0.4 min
Flow rate:	2.5 mL/min, temperature 35 °C
Detection:	UV, 254 nm fluorescence (see chromatogram)

Separation without acetonitrile

Column:	125 x 4 mm NUCLEODUR® C18 PAH, 3 µm
Eluent:	A) water B) methanol 65–97 % B in 6 min, 97 % B for 5 min, 97–65 % B in 0.5 min
Flow rate:	2 mL/min, temperature 35 °C
Detection:	fluorescence (see chromatogram)

Peaks:

1. Naphthalene
2. Acenaphthylene (not detectable by fluorescence)
3. Acenaphthene
4. Fluorene
5. Phenantrene
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benz[*a*]anthracene
10. Chrysene
11. Benzo[*b*]fluoranthene
12. Benzo[*k*]fluoranthene
13. Benzo[*a*]pyrene
14. Dibenz[*ah*]anthracene
15. Benzo[*ghi*]perylene
16. Indeno[1,2,3-*cd*]pyrene



Detection of separated PAHs with UV (250–280 nm), diode array or fluorescence detection at different wavelengths for excitation and emission (acenaphthylene cannot be analyzed with fluorescence detection).

Ordering information

Eluent in column acetonitrile – water (70:30, v/v)

ID	Length → 100 mm	125 mm	150 mm	250 mm	EC guard columns*
NUCLEODUR® C_{18} PAH, 1.8 µm particle size 1.8 µm · UHPLC					
Analytical EC columns					
2 mm	760773.20				761970.20
3 mm	760773.30				761970.30
4 mm	760773.40				761970.30
NUCLEODUR® C_{18} PAH, 3 µm particle size 3 µm					
Analytical EC columns					
3 mm	760783.30	760784.30	760785.30	760786.30	761971.30
4 mm	760783.40	760784.40	760785.40	760786.40	761971.30

Guard column system

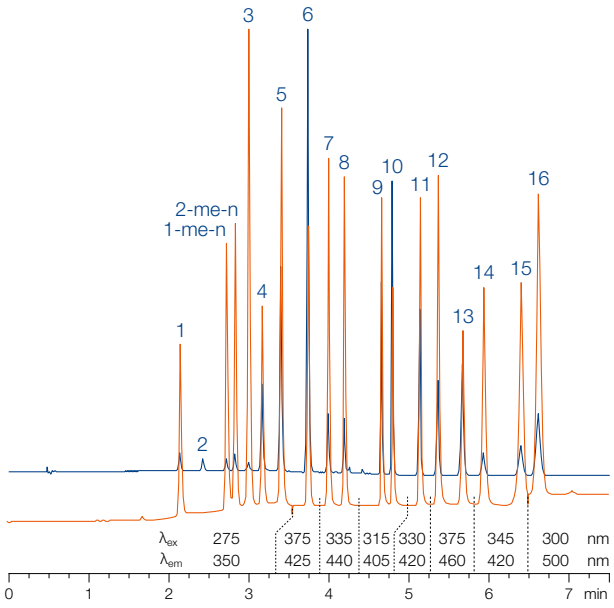
Guard columns for EC columns with ID		2 mm	3 mm	4 mm	4.6 mm	Guard column holder
* Column Protection System (pack of)	EC	4/2 (3)	4/3 (3)	4/3 (3)	4/3 (3)	718966

Separation of 18 PAHs on NUCLEODUR® C_{18} PAH

MN Appl. No. 123840

Column:	125 x 4 mm NUCLEODUR® C_{18} PAH, 3 µm
Eluent:	A) methanol – water (70:30, v/v); B) acetonitrile 0–20 % B in 1.5 min, 20–50 % B in 1.5 min, 50–100 % B in 1.0 min, 100 % B for 3 min, 100–0 % B in 0.5 min
Flow rate:	1.5 mL/min
Temperature:	35 °C
Injection:	UV: 1 µL,
Fluorescence:	0.5 µL
Detection:	UV, 254 nm fluorescence (see chromatogram)

Peaks:
(concentrations 10 ng/µL per compound)
1.–16. see page 227
1-me-n: 1-methylnaphthalene
2-me-n: 2-methylnaphthalene

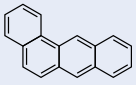


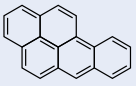
Analysis of polycyclic aromatic hydrocarbons (PAHs) by HPLC

Polycyclic aromatic hydrocarbons (PAHs) are chemical compounds that consist of fused aromatic rings and do not contain heteroatoms or carry substituents. As a pollutant, they are of concern because some compounds have been identified as carcinogenic, mutagenic, and teratogenic. PAHs are natural components of coal or gas. They are delivered to our environment by pyrolysis (incomplete burning) of organic materials like coal, oil, fuel, wood, tobacco, … and hence can be found globally. Today most PAHs accrue from anthropogenic processes – but also natural origins (forest fire) are possible. Regarding to past pollutions an important impact had production of coke and gas from black coal. Waste products (e.g., tar) from coking or gas plants are often origin of serious ground water pollutions.

Since a number of PAHs (e.g., benzo[a]pyrene, 3-methylcholanthrene and benzanthracene) have been proven to be carcinogenic, control of the PAH content of food, water and soil is an important task for routine analysis. For choice and limiting values of the polycyclics we refer to the governmental regulations, which exist in many countries (e.g., EPA method 610 of the United States Environmental Protection Agency).

PAHs can be determined by different chromatographic techniques (TLC, GC, HPLC). Thus the 6 PAHs according to German drinking water specification (TVO) can, e.g., be analyzed by TLC (see German Standard DIN 38 409), while a much larger number of polycyclic aromatics can be determined by GC or HPLC.


Benzo[a]anthracen


Benzo[a]pyren

HPLC columns for PAH analysis

For PAH analyses we have developed specially modified C_{18} phases based on NUCLEODUR® and NUCLEOSIL® which allow efficient gradient separation of 16 PAHs according to EPA. Detection of the separated PAHs can be achieved by UV (250–280 nm), with diode array or with fluorescence detection at different wavelengths for excitation and emission. Acenaphthylene cannot be analyzed with fluorescence detection. For cost-effective routine PAH analysis we recommend applications using methanol instead of acetonitrile as eluent. For rapid analysis NUCLEODUR® C_{18} PAH (3 µm) in short columns (100 mm) provides excellent results at high flow rates. Hereby separation of 16 PAHs according to EPA can be achieved in less than 3 min.

Tightened regulations require determination of 2 additional PAHs (1- and 2-methylnaphthalene) – so we developed highly efficient methods for 18 PAHs on the NUCLEODUR® C_{18} PAH.

NUCLEOSIL® 100-5 C_{18} PAH special octadecyl phase for PAH analysis · USP L1

Technical data

- Base material NUCLEOSIL® silica, particle size 5 µm, pore size 100 Å; polymeric coating
- Detection of the separated PAH with UV (250–280 nm), diode array or fluorescence detection at different wavelengths for excitation and emission (acenaphthylene cannot be analyzed with fluorescence detection)

Recommended application

- Efficient gradient separation of the 16 PAHs according to EPA

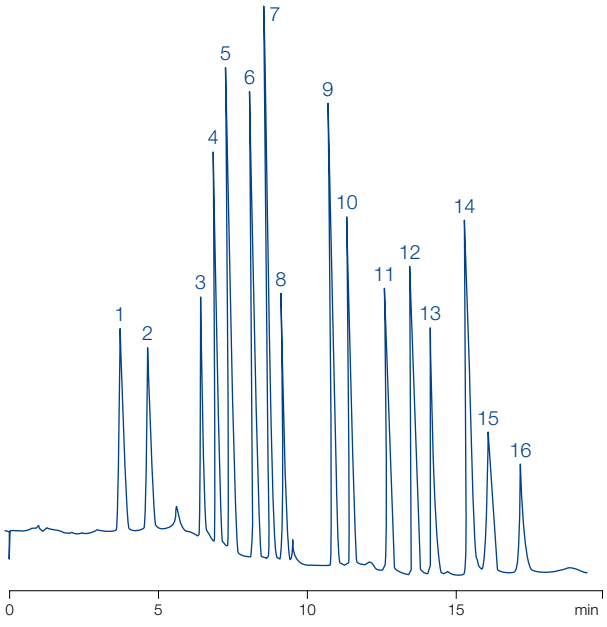
Separation of the PAH standard according to EPA (REF 722393)

MN Appl. No. 115040

Column:	150 x 4 mm NUCLEOSIL® 100-5 C_{18} PAH
Eluent:	A) methanol – water (80:20)
	B) acetonitrile – tetrahydrofuran (93:7)
	0–100 % B in 10 min, 5 min 100 % B
Flow rate:	1 mL/min
Pressure:	140 bar
Temperature:	20 °C
Detection:	UV, 260 nm

Peaks: (10 µg/mL each in acetonitrile)

1. Naphthalene
2. Acenaphthylene
3. Acenaphthene
4. Fluorene
5. Phenanthrene
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benz[*a*]anthracene
10. Chrysene
11. Benzo[*b*]fluoranthene
12. Benzo[*k*]fluoranthene
13. Benzo[*a*]pyrene
14. Dibenz[*ah*]anthracene
15. Benzo[*ghi*]perylene
16. Indeno[1,2,3-*cd*]pyrene



Ordering information

Eluent in column acetonitrile – water 70:30

ID	Length → 150 mm	250 mm	EC guard columns*
NUCLEOSIL® 100-5 C_{18} PAH particle size 5 µm, pore size 100 Å			
Analytical EC columns			
2 mm		720117.20	721168.20
3 mm	720923.30	720117.30	721168.30
4 mm	720923.40	720117.40	721168.30
4.6 mm		720117.46	721168.30
PAH standard according to EPA for HPLC			
Analytical EC columns			
PAH standard for HPLC	16 PAH according to EPA method 610 in acetonitrile (1 mL) for composition see chromatogram above		722393

Guard column system

Guard columns for EC columns with ID		2 mm	3 mm	4 mm	4.6 mm	Guard column holder
* Column Protection System (pack of)	EC	4/2 (3)	4/3 (3)	4/3 (3)	4/3 (3)	718966

EC columns in packs of 1, guard columns in packs of 3. For details of our column systems see page 250.

This product contains harmful substances which must be specially labeled as hazardous. For detailed information please see SDS.

HPLC columns for environmental analyses

Anion columns for analysis of inorganic anions

NUCLEOGEL® Anion I

Technical data

- Strongly basic polymer-based anion exchanger, particle size 10 µm; pH stability 1–14
- Eluent in column 4 mmol/L salicylate buffer pH 7.8
- Contrary to the silica-based phase also suited for fluoride analysis

NUCLEOSIL® Anion II

Technical data

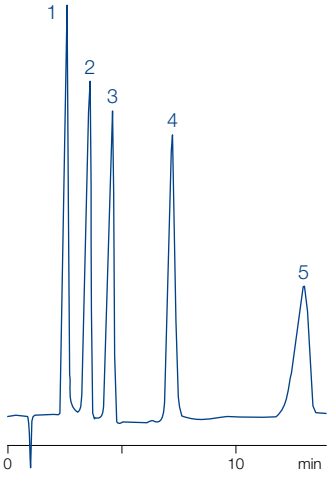
- Base material NUCLEOSIL® silica, particle size 10 µm, pore size 300 Å strongly basic anion exchanger, exchange capacity 50 µeq/g, pH stability 2–7.5
- Eluent in column 0.15 mol/L $(NH_4)_2HPO_4$ buffer pH 5.2 recommended buffer concentration for separation of inorganic anions: 2 mmol/L phthalate
- Preferred method of detection: conductivity or negative UV detection

Separation of an anion standard

MN Appl. No. 106440

Column: 250 x 4 mm NUCLEOSIL® Anion II
Eluent: 2 mmol/L potassium hydrogen phthalate, pH 5.7
Flow rate: 2 mL/min
Detection: UV, 280 nm

Peaks:
1. H_2PO^{4-}
2. Cl^-
3. NO^{2-}
4. NO^{3-}
5. SO_4^{2-}

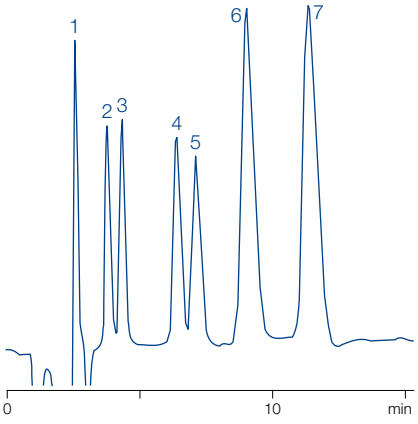


Separation of inorganic anions

MN Appl. No. 115050

Column: 120 x 4.6 mm NUCLEOGEL® Anion I
Eluent: 4 mmol/L salicylic acid – Tris pH 7.8
Flow rate: 1 mL/min
Detection: UV, 254 nm

Peaks:
1. F^-
2. Cl^-
3. NO^{2-}
4. Br^-
5. NO_3^-
6. PO_4^{3-}
7. SO_4^{2-}



Ordering information

ID	Length → 120 mm	250 mm	Guard columns*
NUCLEOGEL® Anion I eluent 4 mmol/L salicylate buffer pH 7.8			
Analytical Valco type columns			
4.6 mm	719533		719543
NUCLEOSIL® Anion II eluent 0.15 mol/L $(NH_4)_2HPO_4$ buffer pH 5.2			
Analytical EC columns			
4 mm		720094.40	721169.30

* NUCLEOGEL® Anion I Valco type guard columns cartridges are 21 x 4 mm, require guard column holder C, REF 719538, see page 250 (columns in packs of 1, guard columns in packs of 2)
NUCLEOSIL® Anion II guard columns are used with the Column Protection System (REF 718966, see page 251).