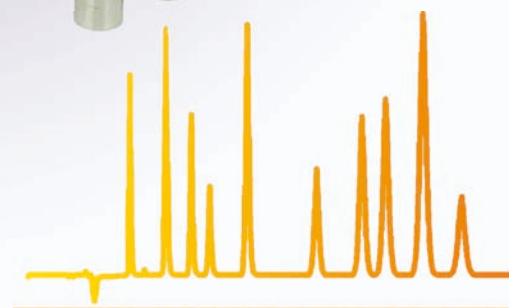


Chromatography

# NUCLEODUR® C<sub>18</sub> HTec

A New Dimension  
in Preparative HPLC



... we Meet your Needs

MACHERY-NAGEL

[www.mn-net.com](http://www.mn-net.com)



# NUCLEODUR® C<sub>18</sub> HTec

## key features:

- reliable and durable standard RP phase for up-scaling to preparative scale, suited for **LC/MS**
- high loadability and excellent stability
- outstanding base deactivation

## technical characteristics:

high density octadecyl modification (C<sub>18</sub>)  
 pore size 110 Å, particle sizes 5 µm, 7 µm and 10 µm for analytical and preparative separations,  
 carbon content 18 %, pH stability 1-11,  
 high reproducibility from batch to batch

## recommended application:

sophisticated analytical and preparative separations of basic, neutral and acidic pharmaceuticals, derivatised amino acids, pesticides, fat-soluble vitamins, aldehydes, ketones and phenolic compounds

**USP L1**

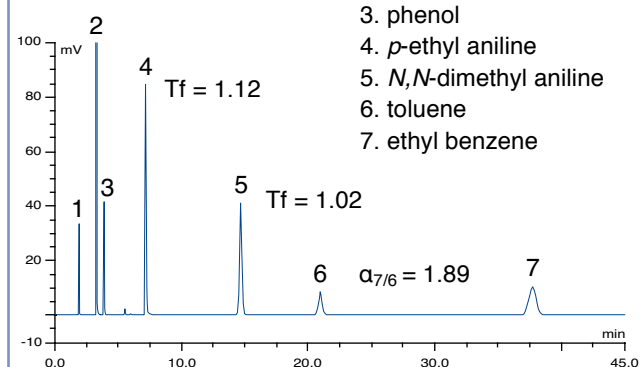
## Engelhardt-Test

excellent symmetry and peak shape over the entire polarity range

Column: NUCLEODUR® 100-5 C<sub>18</sub> HTec, 250 x 4 mm  
 Eluent: methanol / water 49:51 (w/w)  
 Flow rate: 1.0 ml/min  
 Temperature: 40 °C  
 Inj. volume: 5 µl  
 Pressure: 130 bar  
 Detection: UV, 254 nm

### Peaks:

- uracil
- anilin
- phenol
- p*-ethyl aniline
- N,N*-dimethyl aniline
- toluene
- ethyl benzene



MN Appl. No. 123580

## MS bleeding test

low bleeding, good MS efficiency

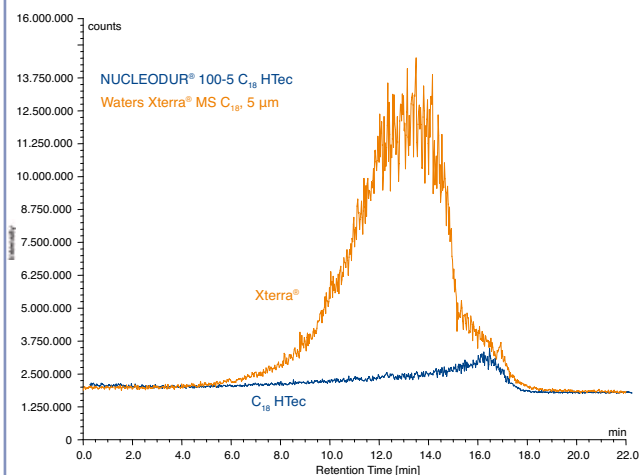
Columns: NUCLEODUR® 100-5 C<sub>18</sub> HTec,  
 Waters Xterra® C<sub>18</sub>, 5 µm  
 (150 x 4.6 mm each)

### HPLC conditions:

Eluent A: water  
 Eluent B: acetonitrile  
 Gradient:  
 Start: 10 % B → 100 % B in 15 min  
 100 % B 5 min  
 100 % B → 10 % B in 5 min  
 Flow rate: 0.26 ml/min  
 Temperature: 22 °C

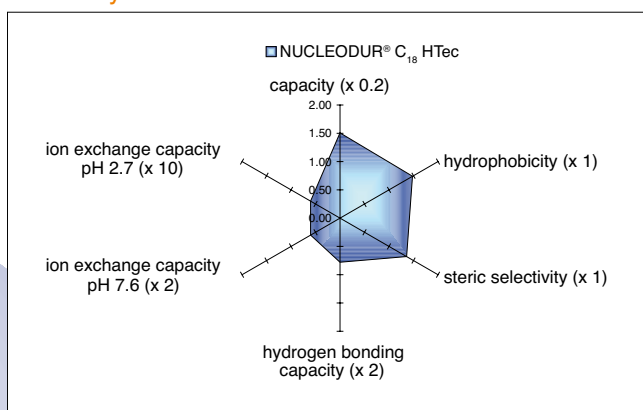
### MS conditions:

sample temperature: 450 °C  
 Needle: 3 kV  
 Cone voltage: + 50 V  
 Detection: ESI+



## Tanaka diagram

outstanding base deactivation and hydrophobic selectivity



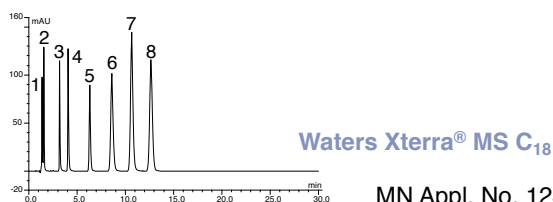
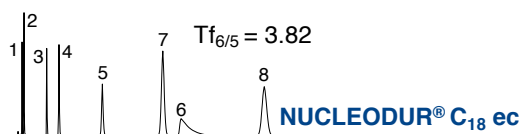
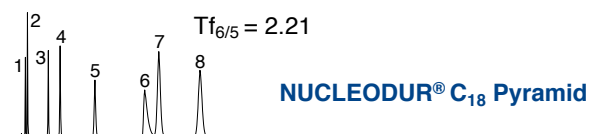
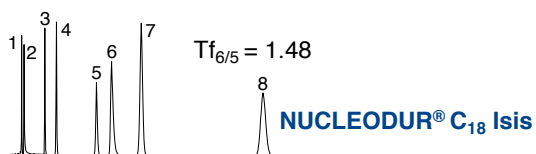
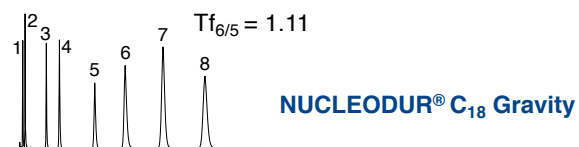
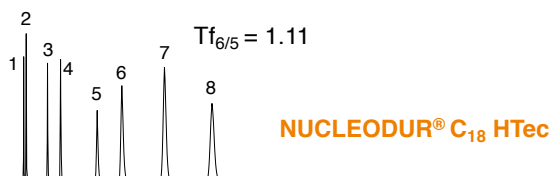
## NUCLEODUR® C<sub>18</sub> HTec: excellent base deactivation

### Phase comparison

Columns: 150 x 4.6 mm each; particle size 5 µm  
 Eluent: methanol / 25 mM KH<sub>2</sub>PO<sub>4</sub>, pH 7 (75:25, v/v)  
 Flow rate: 1.3 ml/min  
 Temperature: 30 °C  
 Detection: UV, 254 nm

#### Peaks:

1. 2,7-dihydroxynaphthalene (metal ion content)
2. 2,3-dihydroxynaphthalene (metal ion content)
3. toluene (hydrophobicity, CH<sub>2</sub> group selectivity)
4. ethylbenzene (hydrophobicity, CH<sub>2</sub> group selectivity)
5. acenaphthene (silanol activity)
6. amitriptyline (silanol activity)
7. o-terphenyl (steric selectivity)
8. triphenylene (steric selectivity)



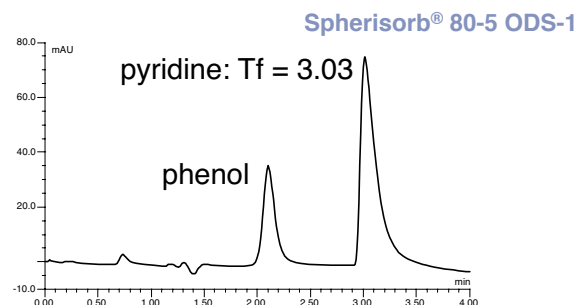
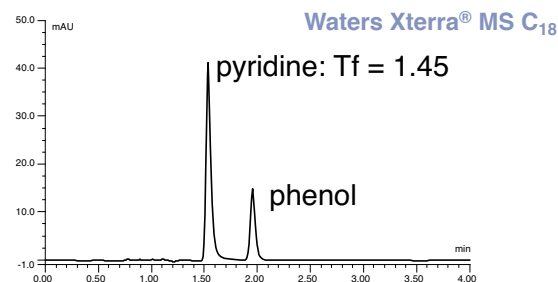
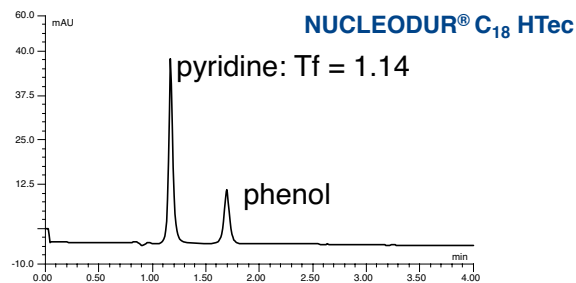
MN Appl. No. 123770

### Base deactivation test

Columns: NUCLEODUR® 100-5 C<sub>18</sub> HTec (125 x 4.6 mm)  
 Waters Xterra® MS C<sub>18</sub>, 5 µm (150 x 4.6 mm)  
 Spherisorb® 80-5 ODS-1 (125 x 4.6 mm)  
 Eluent: acetonitrile / water (50:50, v/v)  
 Flow rate: 1.3 ml/min  
 Temperature: 25 °C  
 Detection: UV, 254 nm

**Important note:** critical test without buffer, acetonitrile instead of methanol (no masking of pyridine by MeOH protons)

**Quality criteria:** pyridine elutes prior to phenol, maximal resolution, tailing (Tf) about 1.0



more applications at  
[www.mn-net.com/apps](http://www.mn-net.com/apps)

# pH Stability & Transferability

## NUCLEODUR® C<sub>18</sub> HTec

- excellent transferability from analytical to preparative particle sizes
- good peak symmetry and minimal peak broadening
- available in 5, 7 and 10 µm

### pH 10 stability test

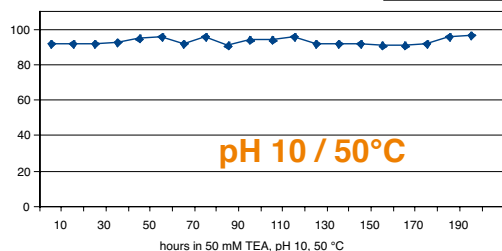
High pH-values can destroy the silica support resulting in dead volume and loss of theoretical plates.

Column: NUCLEODUR® 100-5 C<sub>18</sub> HTec, 150 x 4 mm  
 Eluent: methanol / 50 mM triethylamine, pH 10 (25:80)  
 Flow rate: 1 ml/min  
 Temperature: 50 °C  
 Detection: UV, 254 nm  
 Inj. volume: 5.0 µl  
 Injections: 1034

#### Peak:

theophylline

% Initial N



### pH 1 stability test

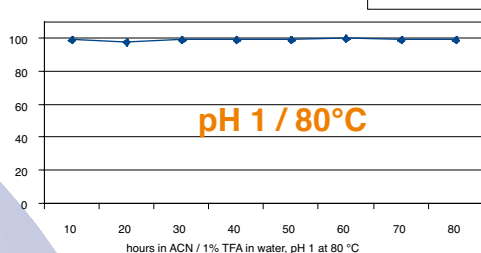
Low pH-values can attack the C<sub>18</sub>-modification resulting in loss of retention.

Column: NUCLEODUR® 100-5 C<sub>18</sub> HTec, 150 x 4 mm  
 Eluent: acetonitrile / 1 % TFA in water, pH 1 (50:50, v/v)  
 Flow rate: 1 ml/min  
 Temperature: 80 °C  
 Detection: UV, 254 nm  
 Inj. volume: 5.0 µl  
 Injections: 693

#### Peaks:

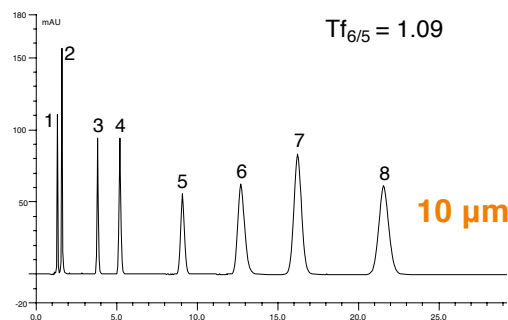
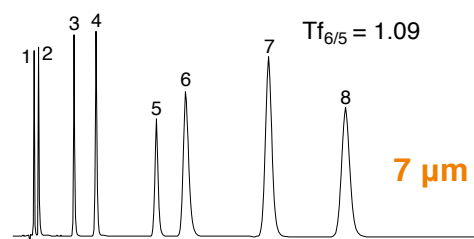
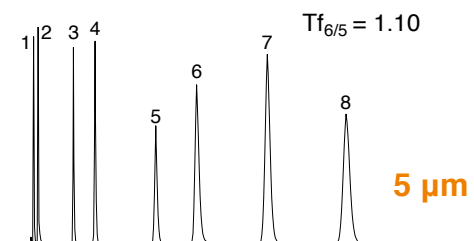
1. pyridine
2. toluol
3. ethylbenzene

% Initial Retention



### Transferability

Column: NUCLEODUR® 100-5 C<sub>18</sub> HTec, 150 x 4.6 mm each  
 Eluent: methanol / 25 mM KH<sub>2</sub>PO<sub>4</sub>, pH 7 (75:25)  
 Flow rate: 1.3 ml/min  
 Temperature: 30 °C  
 Detection: UV, 254 nm



#### Peaks:

1. 2,7-dihydroxynaphthalene (metal ion content)
2. 2,3-dihydroxynaphthalene (metal ion content)
3. toluene (hydrophobicity, CH<sub>2</sub> group selectivity)
4. ethylbenzene (hydrophobicity, CH<sub>2</sub> group selectivity)
5. acenaphthene (silanol activity)
6. amitriptyline (silanol activity)
7. o-terphenyl (steric selectivity)
8. triphenylene (steric selectivity)

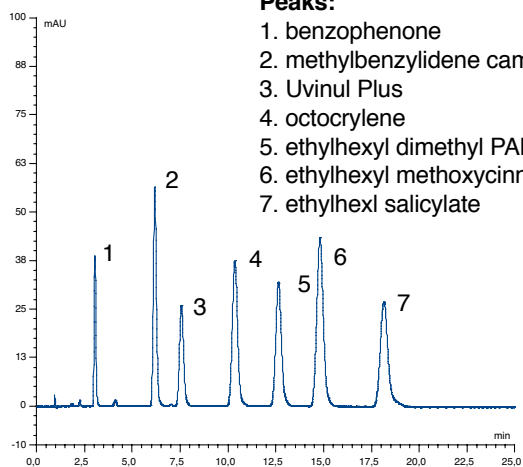
## Analytical application examples for NUCLEODUR® C<sub>18</sub> HTec

### Sunscreen ingredients

Column: NUCLEODUR® 100-5 C<sub>18</sub> HTec, 125 x 4 mm  
 Eluent: methanol / 100 mM ammonium acetate, pH 4.5 (80:20, v/v)  
 Flow rate: 0.9 ml/min  
 Temperature: 35 °C  
 Inj. volume: 12 µl  
 Detection: UV, 275 nm

**Peaks:**

1. benzophenone
2. methylbenzylidene camphor
3. Uvinul Plus
4. octocrylene
5. ethylhexyl dimethyl PABA
6. ethylhexyl methoxycinnamate
7. ethylhexyl salicylate



MN Appl. No. 123640

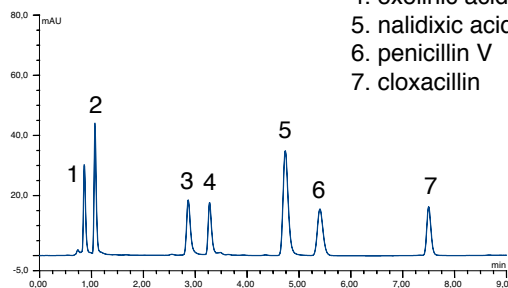
### Antibiotics

Column: NUCLEODUR® 100-5 C<sub>18</sub> HTec, 125 x 4 mm  
 Eluents: A: acetonitrile B: 0.05 % TFA  
 Gradient: 70 % B for 1 min  
 70 % B → 60 % B in 0.5 min  
 60 % B for 3.5 min  
 60 % B → 50 % B in 1 min  
 50 % B → 37.5 % B in 4 min

Flow rate: 0.9 ml/min  
 Temperature: 25 °C  
 Inj. volume: 10 µl  
 Detection: UV, 254 nm  
 Concentration: 300 µg/ml

**Peaks:**

1. amoxicillin
2. enrofloxacin
3. cinoxacin
4. oxolinic acid
5. nalidixic acid
6. penicillin V
7. cloxacillin



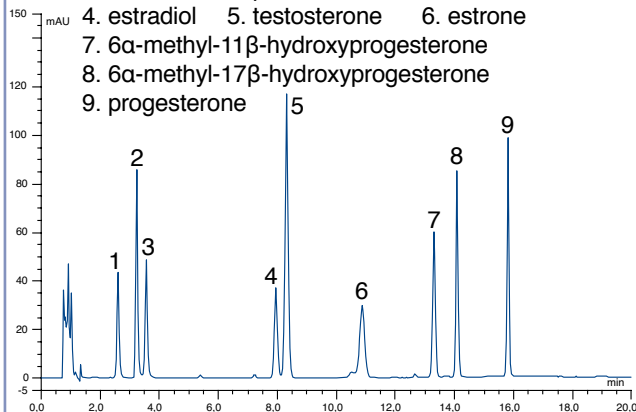
MN Appl. No. 123760

### Steroids

Column: NUCLEODUR® 100-5 C<sub>18</sub> HTec, 125 x 4 mm  
 Eluents A: acetonitrile B: water  
 Gradient: 70 % B → 60 % B in 5 min  
 60 % B for 5 min  
 60 % B → 35 % B in 5 min  
 35 % B for 5 min  
 Flow rate: 1.0 ml/min  
 Temperature: 35 °C  
 Inj. volume: 10 µl  
 Detection: UV, 254 nm

**Peaks:**

1. estriol
2. prednisolone
3. cortisone
4. estradiol
5. testosterone
6. estrone
7. 6α-methyl-11β-hydroxyprogesterone
8. 6α-methyl-17β-hydroxyprogesterone
9. progesterone



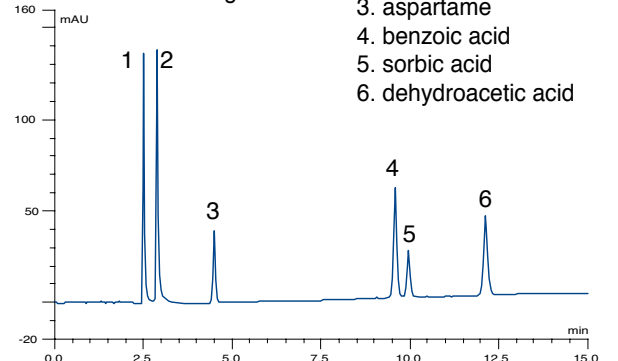
MN Appl. No. 123710

### Sweeteners

Column: NUCLEODUR® 100-5 C<sub>18</sub> HTec, 125 x 4.6 mm  
 Eluents: A: acetonitrile B: 25 mM KH<sub>2</sub>PO<sub>4</sub>, pH 3.5  
 Gradient: 15 % A for 2.5 min  
 15 % A → 25 % A in 9.5 min  
 25 % A for 3 min  
 Flow rate: 1.3 ml/min  
 Temperature: 40 °C  
 Inj. volume: 5 µl  
 Detection: UV, 220 nm  
 Concentration: 0.1 mg/ml each

**Peaks:**

1. acesulfame
2. saccharin
3. aspartame
4. benzoic acid
5. sorbic acid
6. dehydroacetic acid



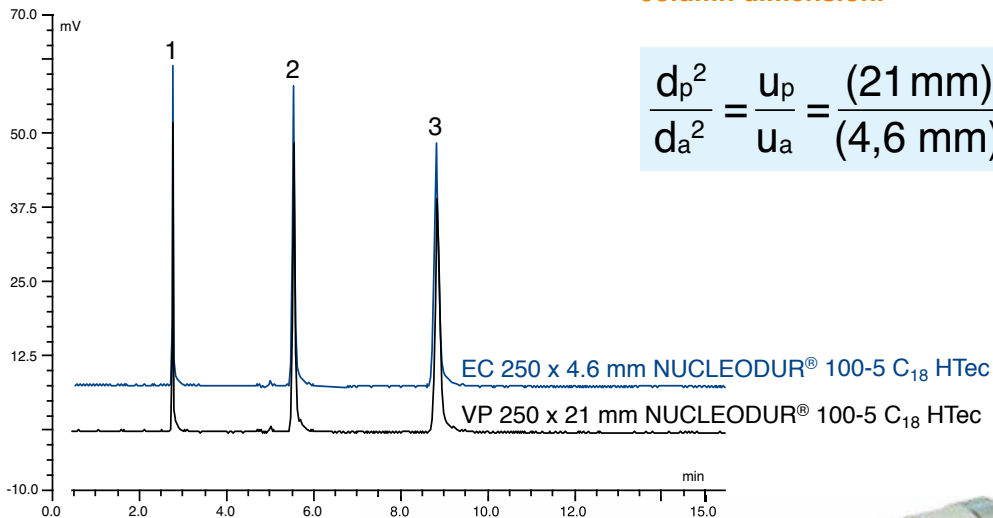
MN Appl. No. 123750

more applications at  
[www.mn-net.com/apps](http://www.mn-net.com/apps)

# Up-Scaling

## NUCLEODUR® C<sub>18</sub> HTec . . . ideal for up-scaling

Identical peak shapes and retention while up-scaling from analytical to preparative column dimension.



$$\frac{d_p^2}{d_a^2} = \frac{u_p}{u_a} = \frac{(21 \text{ mm})^2}{(4,6 \text{ mm})^2} \approx \frac{27 \text{ ml / min}}{1,3 \text{ ml / min}}$$



Columns: EC 250 x 4.6 mm NUCLEODUR® 100-5 C<sub>18</sub> HTec  
VP 250 x 21 mm NUCLEODUR® 100-5 C<sub>18</sub> HTec

Eluent: acetonitrile / water (80:20, v/v)

Flow rate: 1.3 ml/min  
27.0 ml/min

Temperature: 22 °C

Detection: UV, 254 nm

Pressure: 84 bar  
109 bar

### Peaks:

1. phenol
2. naphthalene
3. anthracene  
(1 mg/ml each)

Inj. volume: 3 µl  
60 µl

MN Appl. No. 123780

## Scale up factors and parameters for common MN column dimensions

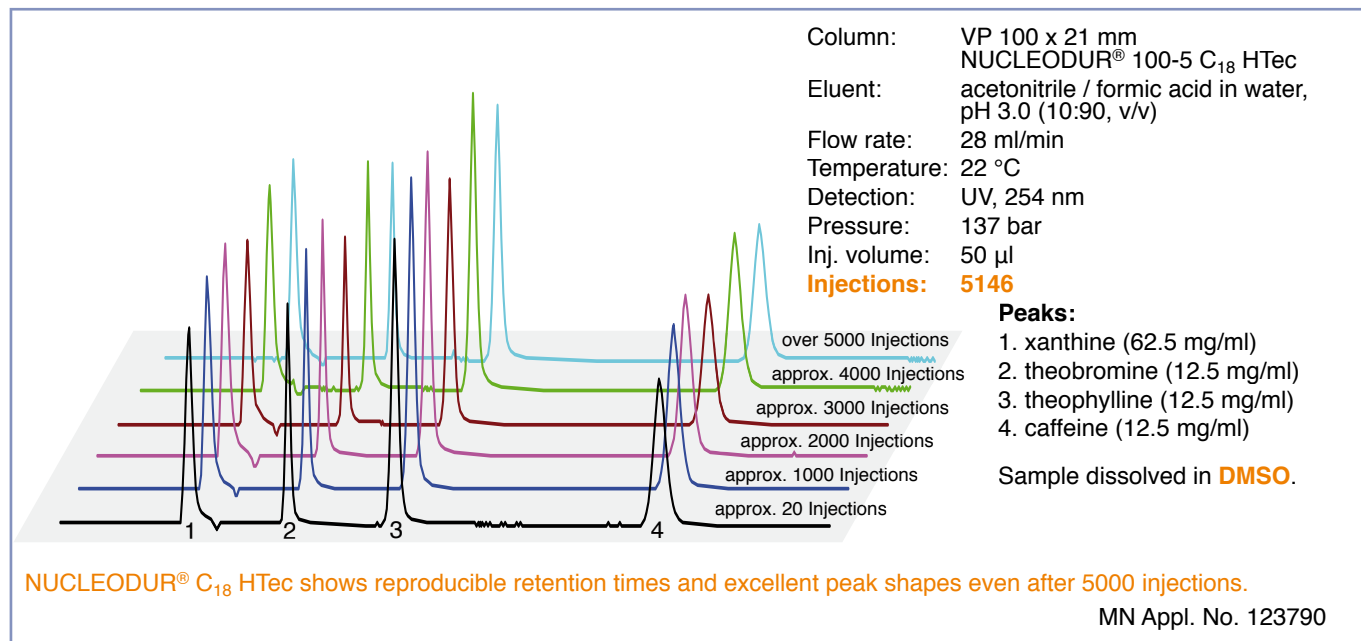
	4 x 250	8 x 250	10 x 250	16 x 250	21 x 250	32 x 250	40 x 250	50 x 250	80 x 250
ID x length [mm]	4 x 250	8 x 250	10 x 250	16 x 250	21 x 250	32 x 250	40 x 250	50 x 250	80 x 250
Linear scale-up factor	1	4	6,25	16	27,6	64	100	161,3	400
Typical sample mass* [mg]	0,02 – 2	0,08 – 8	0,13 – 13	0,3 – 35	0,6 – 60	1,3 – 130	2 – 210	3 – 350	10 – 850
Typical flow rate [ml/min]	0,5 – 1,5	2 – 6	3 – 9	8 – 24	14 – 40	32 – 96	50 – 150	80 – 250	200 – 600

\* For RP material; the maximum amounts given here always depend on the separation problem and on the sample composition. In some cases half of the amount given can cause drastic overload, in other cases the maximum amounts can be even higher still giving acceptable separations.

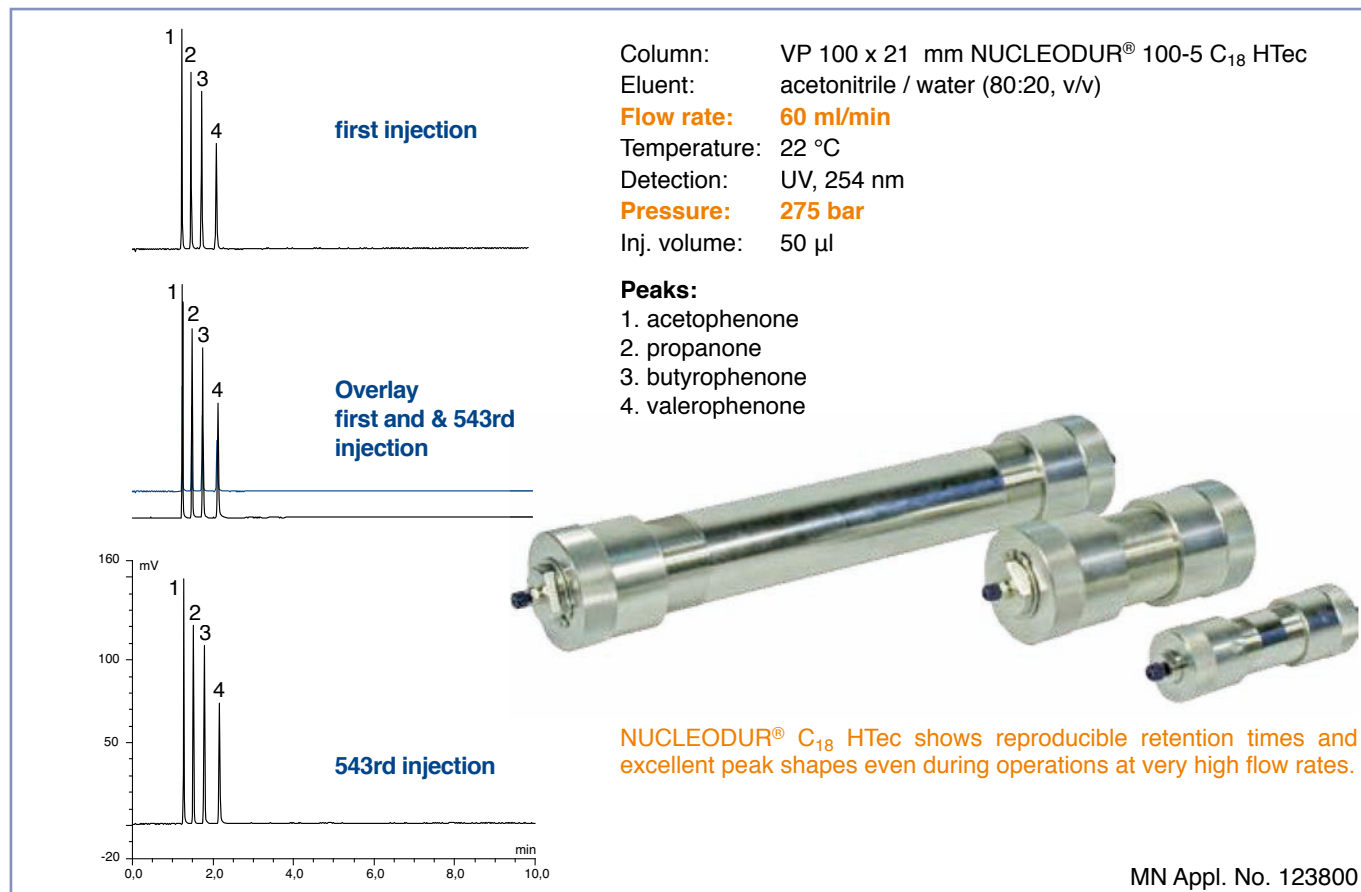
# Column Life Time

## NUCLEODUR® C<sub>18</sub> HTec . . . exceptional column life time

### under critical conditions



### at high flow rates

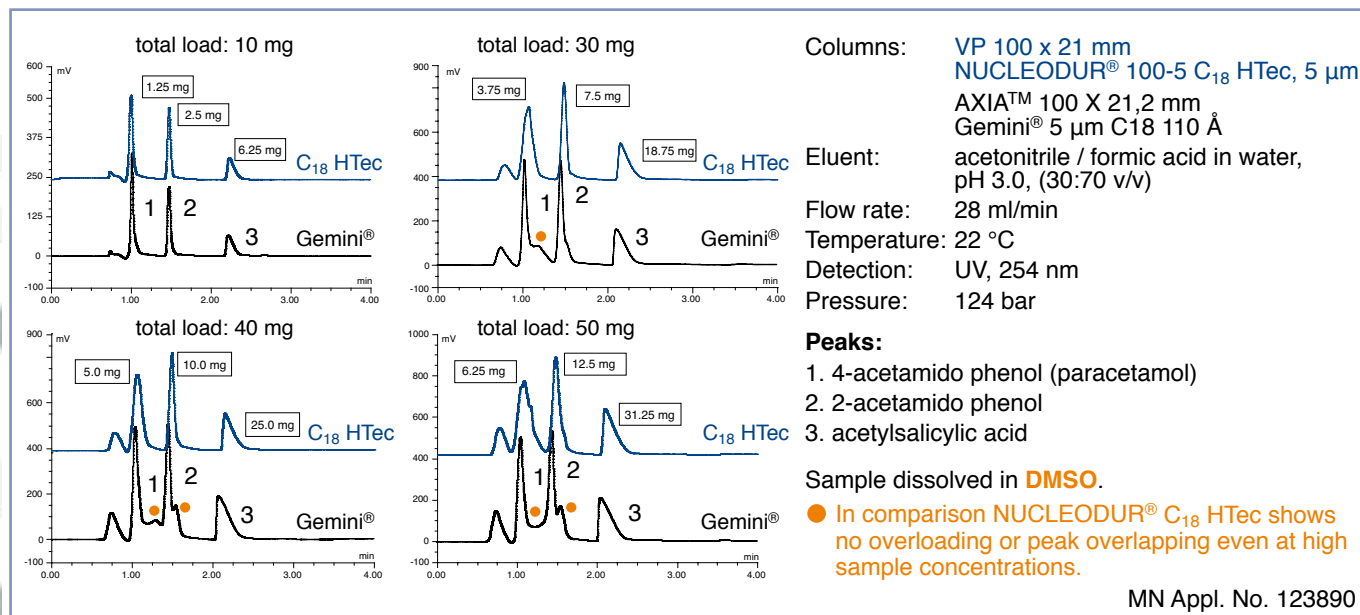


# Loadability 1

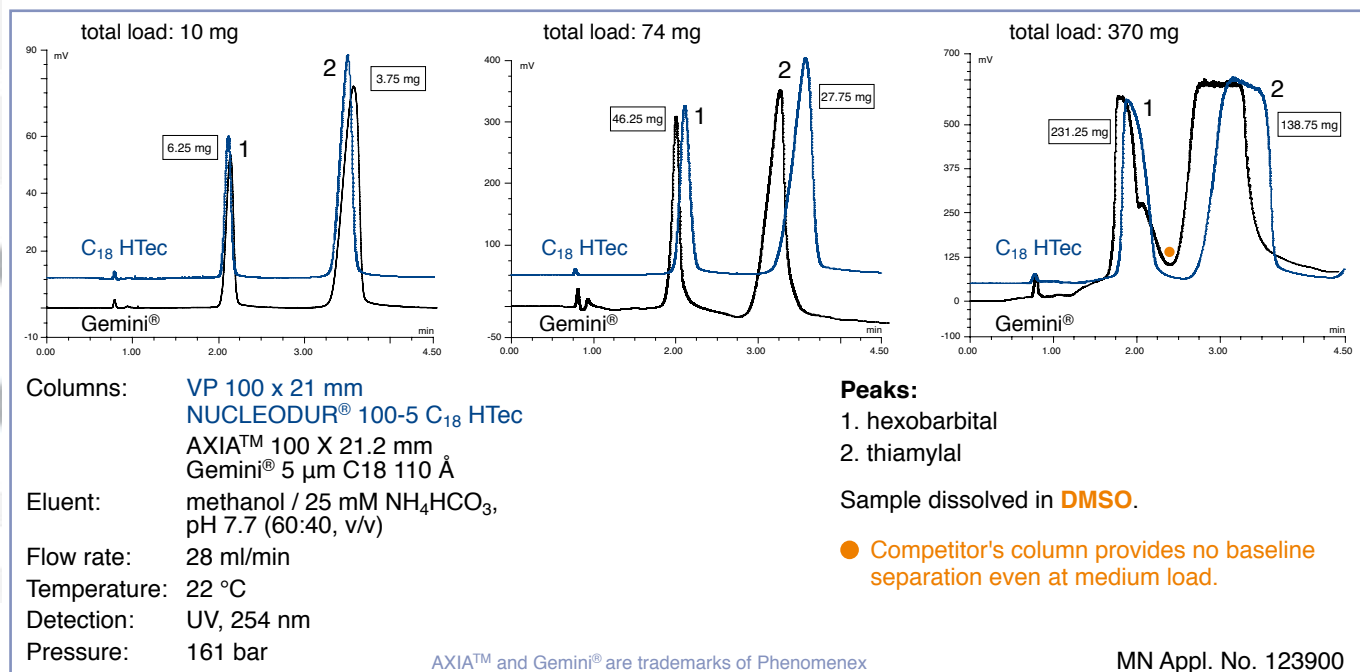
## NUCLEODUR® C<sub>18</sub> HTec

### highest loadability under acidic and basic conditions

#### acidic conditions pH 3.0



#### basic conditions pH 7.7



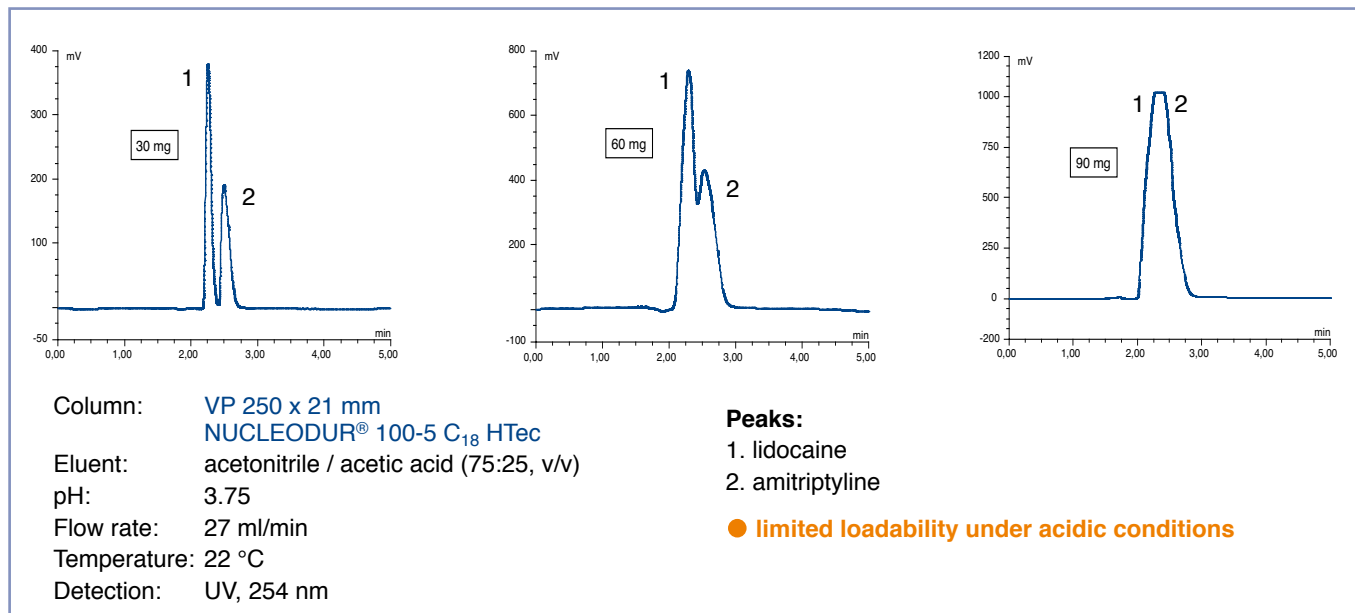
- no overloading effects even at maximum sample concentration
- no overlapping effects or intermixture of compounds
- high yields and efficiency



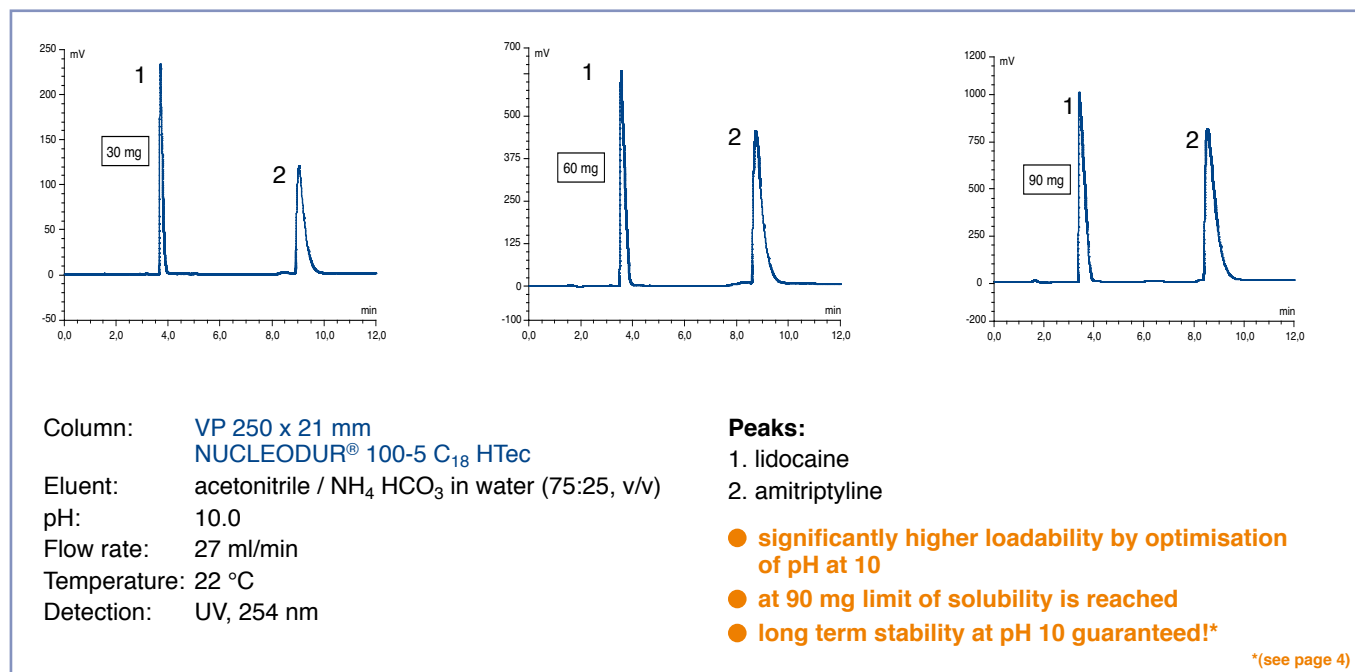
## NUCLEODUR® C<sub>18</sub> HTec

### optimal loadability by variation of pH

#### acidic conditions pH 3.75



#### basic conditions pH 10.0



- limited loadability at acidic conditions (pH 3.75)
- at pH 10 even 90 mg sample don't result in overloading effects
- highest yields and efficiency

# The VarioPrep System

## VarioPrep columns

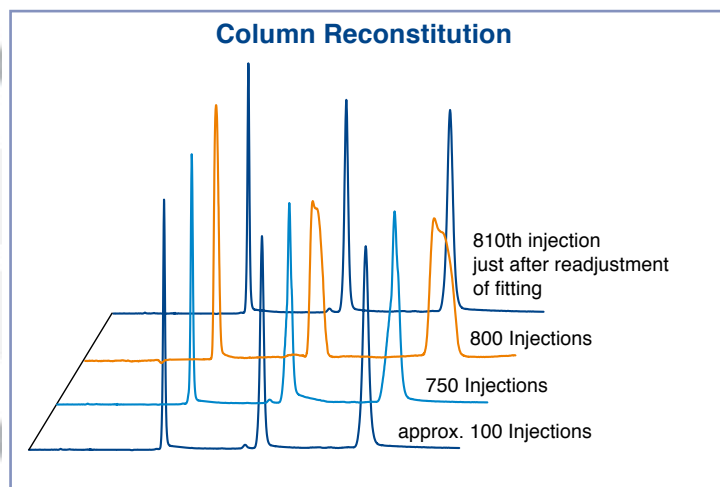
- column system for preparative HPLC manufactured from stainless steel with adjustable end fittings (suitable for frequent use of back-flushing techniques)
- allows compensation of a dead volume, which could result at the column inlet after some time of operation at demanding conditions, without need for opening the column
- supplied with all NUCLEODUR® and NUCLEOSIL® spherical silicas



## The VarioPrep principle



Based on our special packing procedure VarioPrep columns are produced with highest packing quality and bed density (A). Due to intensively chemical and/or mechanical exposure of the column adsorbent, shrinking of the column bed can occur (B; orange gap). In this even unlikely case readjustment of the VarioPrep column fitting (C; turning the nut at column inlet clockwise) will eliminate the emerged dead volume (D). The performance of the VarioPrep column is completely reconstituted and column lifetime is significantly extended.



## Reconstitution of VarioPrep column performance:

- slight peak broadening and deformation after 800 injections under strongly demanding conditions (pH 11; 50°C; sample in DMSO)
- readjustment of the column fitting restores the column performance and prolongs column lifetime noticeably

## Available standard dimensions of VarioPrep columns with axially adjustable end fittings

ID [mm]	Length [mm]					end fitting design					
	10*	15*	20*	30*	50	100	125	150	250	500	
8	x				x	x	x	x	x		
10					x	x	x	x	x		
16			x		x	x	x	x	x		
21					x	x	x	x	x		
32		x				x		x	x		
40					x	x	x	x	x	x	
50				x		x		x	x		
80									x	x	

\* 10 x 8, 20 x 16, 15 x 32 and 30 x 50 mm ID columns are used as guard columns and require the adequate holders. Details see on page 13.

# VarioPrep Guard Columns



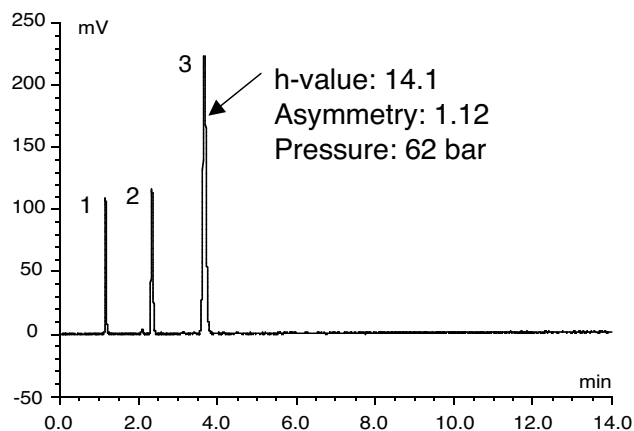
**VP 8/10** for 8 and 10 mm ID columns  
**VP 20/16** for 16 and 21 mm ID columns  
**VP 15/32** for 32 and 40 mm ID columns  
**VP 30/50** for  $\geq 50$  mm ID columns

## The new guard column system for (semi-) preparative HPLC

- easy handling and cartridge exchange
- robust hardware
- cost-efficient cartridges
- minimally invasive / no disturbance of the separation efficiency of main column
- low back pressure

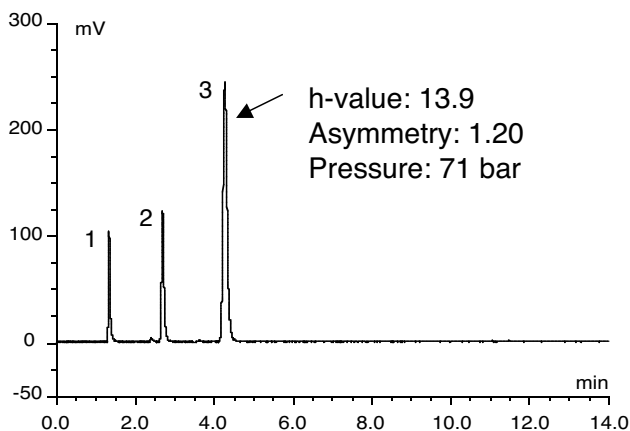
## Performance of preparative column when used with VarioPrep guard column

VP 125/16 NUCLEODUR® 100-5 C<sub>18</sub> HTec



Eluent: acetonitrile / water (80:20, v/v)  
 Flow rate: 16 ml/min  
 Temperature: 22 °C

VP 125/16 NUCLEODUR® C<sub>18</sub> 100-5 HTec  
 VP 20/16 NUCLEODUR® C<sub>18</sub> HTec (guard column)



**Peaks:**  
 1. phenol  
 2. naphthaline  
 3. anthracene

Using VarioPrep guard columns provides ideal protection of your main column – symmetry, pressure and retention stay almost constant.

## Technical data:

- 1/16" thread
- free rotary plunger fittings – low O-ring abrasion
- stainless steel

Guard cartridge	Recommended for column ID	Preferred capillary ID	Typical flow rate
VP 10/8	8 and 10 mm ID	0.17 and 25 mm	1 – 12 ml/min
VP 20/16	16 and 21 mm ID	0.17, 0.25 and 0.5 mm	2 – 32 ml/min
VP 15/32	32 and 40 mm ID	0.25, 0.5 and 1.0 mm	5 – 150 ml/min
VP 30/50	$\geq 50$ mm ID	0.5 and 1.0 mm	20 – 250 ml/min

The cartridge system is designed for pressures up to 400 bar.

# Further NUCLEODUR® Phases

MACHEREY-NAGEL offers all NUCLEODUR® phases as VarioPrep-columns in preparative dimensions.

Phase	Specification	Characteristics*	Stability	Structure
<b>C<sub>18</sub> H Tec</b>	octadecyl phase, with high capacity, high density coating multi-endcapping 18% C · USP L1	A	pH stability 1 - 11, suitable for LC/MS	NUCLEODUR® (Si-O <sub>2</sub> ) <sub>n</sub>
		B		
		C		
<b>C<sub>18</sub> / C<sub>8</sub> Gravity</b>	octadecyl phase, high density coating multi-endcapping C <sub>18</sub> Gravity: 18% C · USP L1 C <sub>8</sub> Gravity: 11% C · USP L7	A C <sub>18</sub>	pH stability 1 - 11, suitable for LC/MS	NUCLEODUR® (Si-O <sub>2</sub> ) <sub>n</sub>
		A C <sub>8</sub>		
		B C <sub>18</sub>		
		B C <sub>8</sub>		
		C C <sub>18</sub>		
		C C <sub>8</sub>		
<b>C<sub>18</sub> Isis</b>	octadecyl phase with specially crosslinked surface modification endcapping 20% C · USP L1	A	pH stability 1 - 10, suitable for LC/MS	NUCLEODUR® (Si-O <sub>2</sub> ) <sub>n</sub>
		B		
		C		
<b>C<sub>18</sub> Pyramid</b>	C <sub>18</sub> modification with polar endcapping 14% C · USP L1	A	stable against 100% aqueous eluents pH stability 1 - 9, suitable for LC/MS	NUCLEODUR® (Si-O <sub>2</sub> ) <sub>n</sub>
		B		
		C		
<b>Sphinx RP</b>	bifunctional RP phase, propylpheny and C <sub>18</sub> ligands; endcapping 15% C; USP L1 and L11	A	pH stability 1 - 10, suitable for LC/MS	NUCLEODUR® (Si-O <sub>2</sub> ) <sub>n</sub>
		B		
		C		
<b>C<sub>18</sub> ec / C<sub>8</sub> ec</b>	octadecyl / octyl phase, medium density coating endcapping C <sub>18</sub> ec: 17.5% C · USP L1 C <sub>8</sub> ec: 10.5% C · USP L7	A C <sub>18</sub>	pH stability 1 - 9	NUCLEODUR® (Si-O <sub>2</sub> ) <sub>n</sub>
		A C <sub>8</sub>		
		B C <sub>18</sub>		
		B C <sub>8</sub>		
		C C <sub>18</sub>		
		C C <sub>8</sub>		
<b>HILIC</b>	zwitterionic ammonium sulfonic acid modification 7% C	A	pH stability 2 - 8.5, suitable for LC/MS	NUCLEODUR® (Si-O <sub>2</sub> ) <sub>n</sub>
		B		
		C -		
<b>CN / CN-RP</b>	cyano (nitrile) phase for NP and RP separations 7% C · USP L10	A	pH stability 1 - 8, stable towards highly aqueous mobile phases	NUCLEODUR® (Si-O <sub>2</sub> ) <sub>n</sub>
		B		
		C -		
<b>NH<sub>2</sub> / NH<sub>2</sub>-RP</b>	amino phase for NP and RP separations 2.5% C · USP L8	A	pH stability 1 - 8, stable towards highly aqueous mobile phases suitable for LC/MS	NUCLEODUR® (Si-O <sub>2</sub> ) <sub>n</sub>
		B		
		C -		
<b>SiOH</b>	unmodified high purity silica USP L3	A -	pH stability 2 - 8	NUCLEODUR® (Si-O <sub>2</sub> ) <sub>n</sub>
		B n.a.		
		C -		

\* A = hydrophobic selectivity, B = polar/ionic selectivity, C = steric selectivity

# Ordering Informations

## NUCLEODUR® C<sub>18</sub> HTec



Lenght →	50 mm	75 mm	100 mm	125 mm	150 mm	250 mm	guard columns#
<b>analytical columns (5 µm)</b>							
2 mm ID	760311.20			760314.20		760316.20	761110.30
3 mm ID	760311.30			760314.30		760316.30	761110.30
4 mm ID	760311.40			760314.40		760316.40	761110.40
4.6 mm ID	760311.46	760312.46	760313.46	760314.46	760315.46	760316.46	761110.40

# requires EC adaptor REF 721359



Lenght →	50 mm	100 mm	125 mm	150 mm	250 mm	guard columns*
<b>preparative columns (5 µm)</b>						
10 mm ID	762551.100		762554.100		762556.100	762594.80
21 mm ID	762551.210	762553.210	762554.210		762556.210	762593.160
32 mm ID		762553.320		762555.320	762556.320	762592.320
40 mm ID				762555.400	762556.400	762592.320
50 mm ID		762553.500		762555.500	762556.500	762595.500



Lenght →	50 mm	100 mm	125 mm	150 mm	250 mm	guard columns*
<b>preparative columns (7 µm)</b>						
10 mm ID	762561.100		762564.100		762566.100	762594.80
21 mm ID	762561.210	762563.210	762564.210		762566.210	762593.160
32 mm ID		762563.320		762565.320	762566.320	762592.320
40 mm ID				762565.400	762566.400	762592.320
50 mm ID		762563.500		762565.500	762566.500	762595.500



Lenght →	50 mm	100 mm	125 mm	150 mm	250 mm	guard columns*
<b>preparative columns (10 µm)</b>						
10 mm ID	762571.100		762574.100		762576.100	762594.80
21 mm ID	762571.210	762573.210	762574.210		762576.210	762593.160
32 mm ID		762573.320		762575.320	762576.320	762592.320
40 mm ID				762575.400	762576.400	762592.320
50 mm ID		762573.500		762575.500	762576.500	762595.500

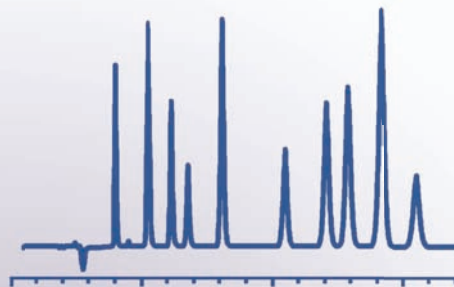


Lenght →	10 mm	15 mm	20 mm	30 mm	Pack of	Holder
<b>*preparative guard columns for NUCLEODUR® C<sub>18</sub> HTec and holders</b>						
8 mm ID	762591.80				1	718251
16 mm ID			762593.160		1	718250
32 mm ID		762592.320			1	718253
50 mm ID				762594.500	1	718254



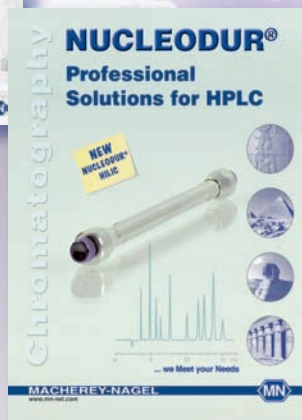
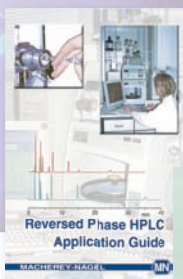
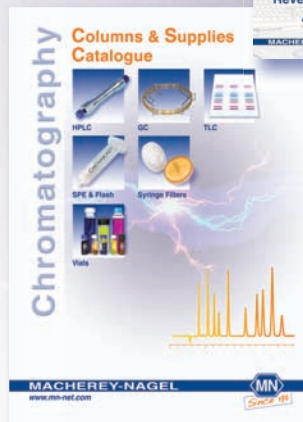
	Pack of 100 g	Pack of 1 kg
<b>NUCLEODUR® C<sub>18</sub> HTec bulk packings</b>		
NUCLEODUR® 100-5 C <sub>18</sub> HTec	713830.0100	713830.1
NUCLEODUR® 100-7 C <sub>18</sub> HTec	713831.0100	713831.1
NUCLEODUR® 100-10 C <sub>18</sub> HTec	713832.0100	713832.1

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