Chromatography

CHROMABOND®
HR-X*pert*

the professional concept of innovative SPE phases

polymer-based RP-phases and mixed-mode ion exchange phases

MACHEREY-NAGEL
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MN Since 1911

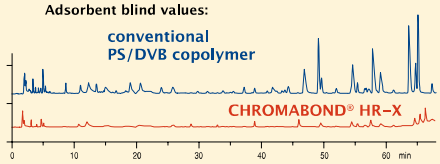
Solid Phase Extraction

CHROMABOND® HR-X*pert*
… the innovative concept of five polymer-based RP- and mixed-mode ion exchange phases for SPE:

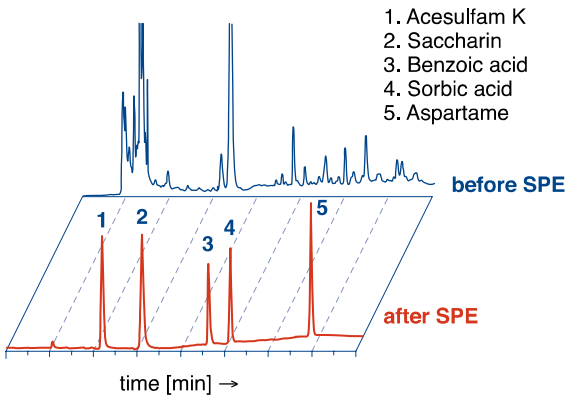
- ➢ CHROMABOND® HR-X — hydrophobic PS/DVB copolymer
- ➢ CHROMABOND® HR-XC — strong mixed-mode cation exchanger
- ➢ CHROMABOND® HR-XA — strong mixed-mode anion exchanger
- ➢ CHROMABOND® HR-XCW — weak mixed-mode cation exchanger
- ➢ CHROMABOND® HR-XAW — weak mixed-mode anion exchanger

. . . these innovative SPE phases offer:

- **state-of-the-art spherical polymer**
  - ⇨ different particle sizes (45 µm and 85 µm) suitable depending on the sample volume and matrix
  - ⇨ broad spectrum of applications with special suitability for enrichment of pharmaceuticals from biological matrices
  - ⇨ ideal flow properties due to low content of particulate matter
- **optimized pore structure and high specific surface**
  - ⇨ high loadability and outstanding elution properties
  - ⇨ low solvent consumption
  - ⇨ rapid, economical analyses
- **high-purity adsorber material**
  - ⇨ allows highest reproducibility with extremely low blind values
  - ⇨ reliable analyses at ultra trace level
  - ⇨ no method adaptation for new batches necessary



Separation of food additives



In modern analyses SPE fulfills 3 important tasks:

- ⇨ **enrichment of analytes**
  up to factor 10.000 – increase of chromatographic sensibility/improved limits of detection
- ⇨ **removal of interfering compounds**
  protection of subsequent analyses like HPLC, GC, TLC, MS, UV or IR spectroscopy, …
- ⇨ **changing of an analyte´s matrix**
  matrix which is simpler or more suitable for subsequent analyses

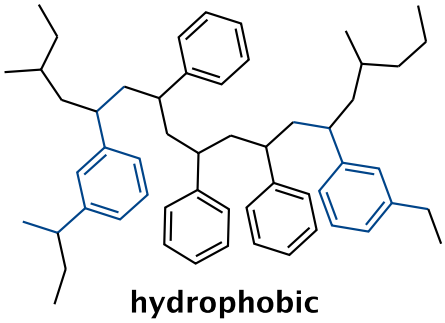
. . . the HR-X*pert* concept guarantees:

- RP- and mixed-mode SPE-phases with distinct ion exchange and reversed-phase properties
  your benefit ⇨ excellent enrichment of neutral, acidic and basic compounds
- modern, spherical support-polymer with optimized pore structure and high surface
  your benefit ⇨ good reproducibility, reliable and cost-efficient analysis
- possibility for more aggressive washing procedures for matrix removal
  your benefit ⇨ cleaner samples and protection of your HPLC and GC-machines
- quantification of analytes also from heavily contaminated samples
  your benefit ⇨ lower limits of detection also for critical matrices

CHROMABOND® HR-X*pert* is the perfect combination for all tasks in sample preparation

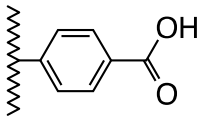
Chemical structures

CHROMABOND® HR-X



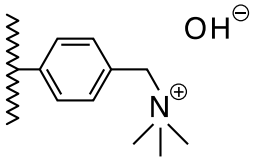
hydrophobic
PS/DVB copolymer

CHROMABOND® HR-XCW



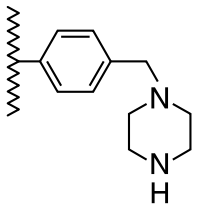
weak acidic
cation exchanger

CHROMABOND® HR-XA



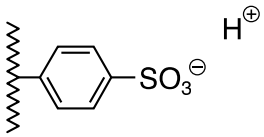
strong basic
anion exchanger

CHROMABOND® HR-XAW

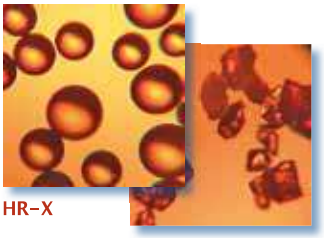


weak basic
anion exchanger

CHROMABOND® HR-XC



strong acidic
cation exchanger



HR-X

conventional
PS/DVB copolymer

HR-X: spherical base material for efficient enrichment and ideal flow behavior

Similary phases:

CHROMABOND® HR-X:	Oasis® HLB, Strata™ X, Nexus, ENVI-Chrom P
CHROMABOND® HR-XC:	Oasis® MCX, Strata™ X-C, StyreScreen® DBX, HyperSep™ Retain™-CX
CHROMABOND® HR-XA:	Oasis® MAX, Strata™ X-A, HyperSep™ Retain™-AX, StyreScreen® QAX
CHROMABOND® HR-XCW:	Oasis® WCX, Strata™-X-CW
CHROMABOND® HR-XAW:	Oasis® WAX, Strata™-X-AW

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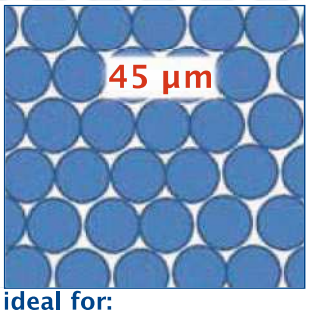
Solid Phase Extraction

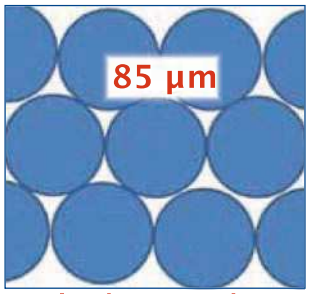
2 particle sizes – 1 goal … with HR-X*pert* to the ideal application

for different applicative requirements the particle sizes complement each other perfectly



ideal for:

- ⊕ smaller sample volumes
- ⊕ smaller amount of sorbent
- ⊕ lower elution volumes



- ⊕ voluminous or viscous samples/ strong matrix load
- ⊕ operation without vacuum (e.g. for volatile analytes)
- ⊕ higher amount of sorbent without pressure rise

Comparison of 45 µm and 85 µm

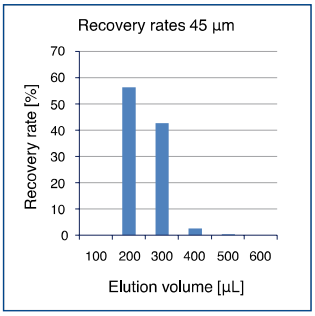
Larger number of particles per volume at 45 µm by about half radius
⇨ eightfold particle number at same amount of sorbent

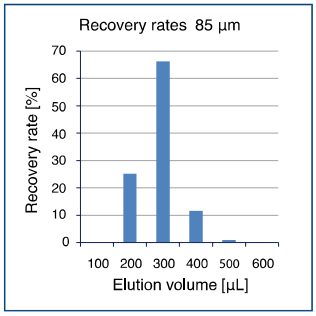
Specific surface is equal for both particle sizes
⇨ freely accessible external surface is at 45 µm particles much larger

More compact sorbent package: Enhanced interaction of the analytes with the sorbent
⇨ improved extraction results

Ideal elution characteristics

Method: 1 mL column with 30 mg CHROMABOND® HR-X, 1 mL standard solution (1 mg/mL hexobarbital); drying; elution in portions of 100 µL with methanol, according to MN Appl. No. 305490





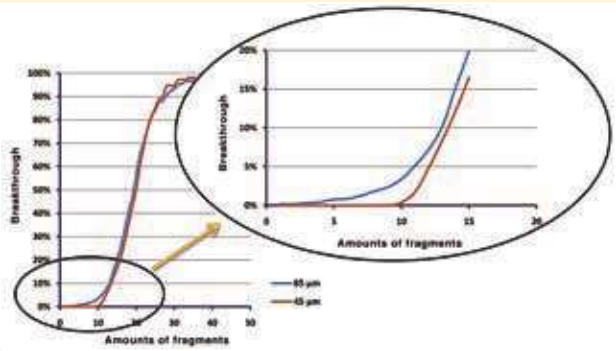
at 45 µm · faster elution · lower elution volumes needed

Breakthrough behavior in enrichment

Method: 1 mL column with 15 mg CHROMABOND® HR-X; apply portions of 1 mL standard solution (250 µg/mL hexobarbital in water); collect the eluate, according to MN Appl. No. 305480

45 µm (red):
Analyte is completely retained up to the fraction 10.

85 µm (blue):
Small amounts even break through at fraction 4.

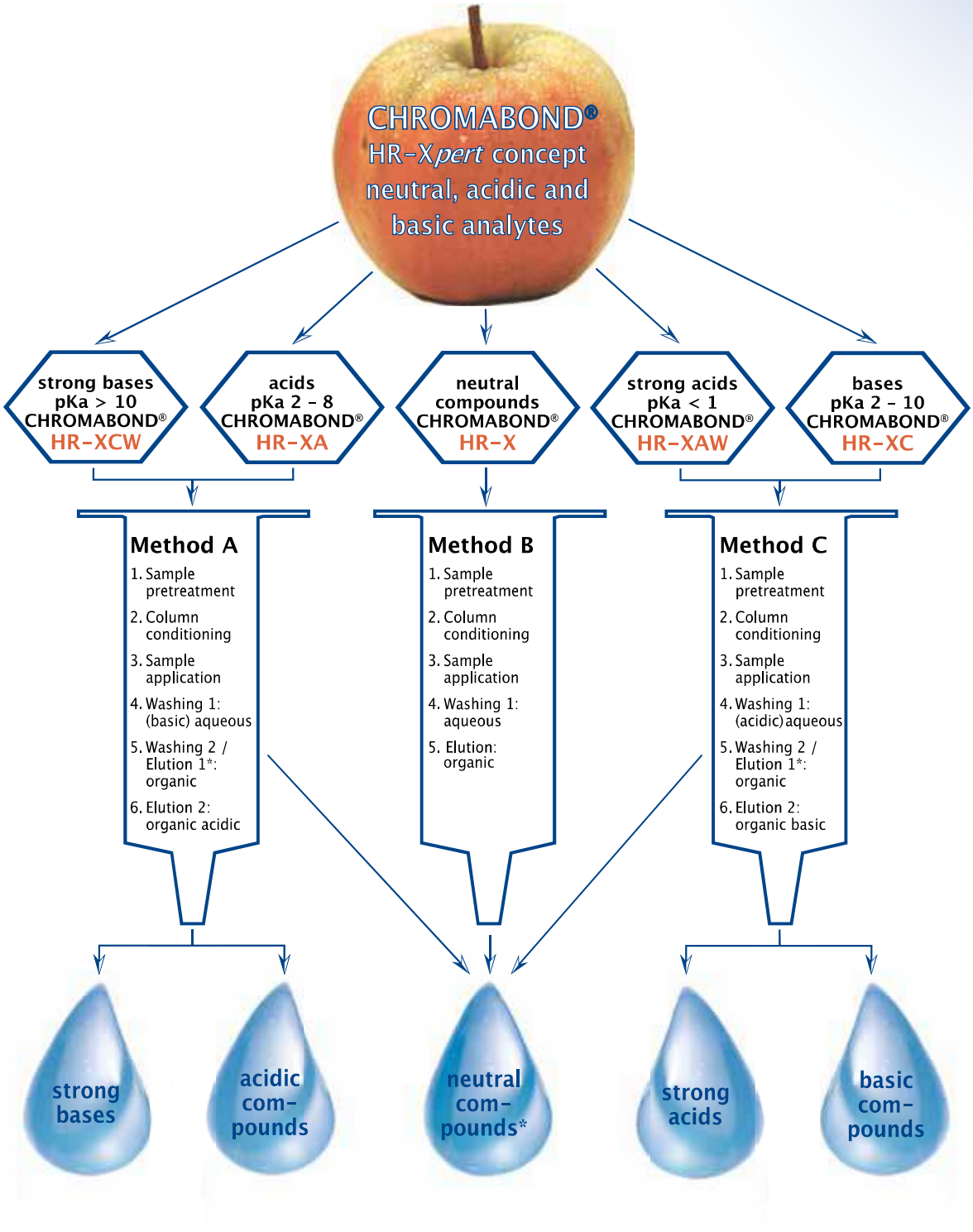


Comparison of 45 µm and 85 µm
⇨ 45 µm are smaller particles and provide better enrichment of sorbent and a superior breakthrough behavior.

When using larger amounts of sorbent this effect is less distinct. Also using 85 µm particles sufficient contact of analyte and sorbent is given. So, 45 µm particles are ideal for small sample and elution volumes, while for large amounts of sample or sorbent 85 µm particles provide advantages due to with better flow properties.

3 paths – 1 goal … cleaner samples by HR-X*pert*:

Depending on the character of the analytes HR-X*pert* offers the suitable adsorbent and optimal method for sample preparation, cleaning and concentration.



*** under organic washing and elution conditions the following compounds will be also eluted:**

HR-X: polar compounds as organic acids and bases

HR-XC/HR- XCW: acidic compounds/impurities

HR-XA/HR- XAW: basic compounds/impurities

HR-X

spherical, hydrophobic polystyrene-divinylbenzene adsorbent resin

- hydrophobic polystyrene-divinylbenzene copolymer pH stability 1–14
- high-purity material with highest reproducibility and lowest blank values due to a novel manufacturing process
- spherical particles, size 45 µm and 85 µm (standard); pore size 55–60 Å, very high surface 1000 m^2/g capacity 390 mg/g (caffeine in water)
- excellent recovery rates especially for the enrichment of pharmaceuticals / active ingredients due to the spherical structure of the particles, very homogeneous surface, and optimized pore structure
- recommended application:
  - pharmaceuticals / active ingredients from tablets, creams and water / waste water
  - drugs and pharmaceuticals from urine, blood, serum and plasma
  - trace analysis of pesticides, herbicides, phenols, PAHs and PCBs from water

Solid Phase Extraction

Standard protocol for CHROMABOND® HR-X

MN Appl. No. 304310

Column type: CHROMABOND® HR-X/3 mL/200 mg, REF 730931

Sample pretreatment: individual sample preparation with reference to analytes and matrix

Conditioning:
5 mL MeOH

Equilibration:
5 mL water

Sample application:
slowly aspirate sample through the column

Washing:
5 mL water
MeOH (95:5, v/v)

Elution:
after drying
3 x 2 mL MeOH

Further analysis: if necessary, evaporate and redissolve in a suitable solvent; HPLC or GC

These conditions are a starting point for SPE method development. Further optimisation may be required to improve results.

Sulfonamides from serum

MN Appl. No. 304220

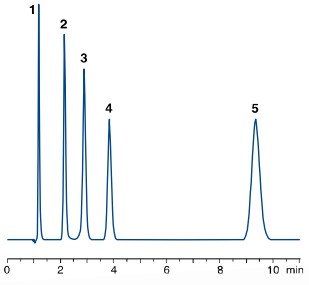
Column type: CHROMABOND® HR-X/3 mL/200 mg REF 730931
Sample: 2 µg/ml each in serum
Column conditioning: 5 mL MeOH, 5 mL dest. water
Sample application: 1 mL spiked serum
Column washing: 5 mL water – MeOH (95:5, v/v)
Elution: after drying 3 x 2 mL MeOH

Recovery rate [%]:

Compound	HR-X	Oasis® HLB	Strata™ X
Sulfanilamide	66	62	63
Sulfadiazine	107	101	108
Sulfamerazine	114	111	111
Sulfadimidine	94	86	89
Succinylsulfathiazole	70	43	48

Further analysis:
HPLC see MN Appl. No. 117880
Column: 125/4 mm NUCLEODUR® C_{18} Gravity, 5 µm
Eluent: MeOH – 0.1 % TFA (20:80, v/v)
Flowrate: 1.0 mL/min
Temperature: 22 °C
Detection: UV, 230 nm

Peaks:
1. Sulfanilamide
2. Sulfadiazine
3. Sulfamerazine
4. Sulfadimidine
5. Succinylsulfathiazole



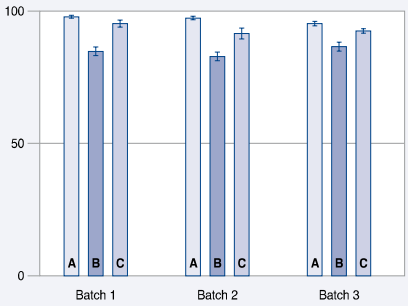
Highest reproducibility

✓ within each batch

✓ from batch to batch

Compounds:

A phenobarbital
B pentobarbital
C hexobarbital



Barbiturates from serum

MN Appl. No. 304290

Column type: CHROMABOND® HR-X/3 mL/200 mg REF 730931

Sample: 100 ng/ml each in serum

Column conditioning: 5 mL MeOH, 5 mL dist. water

Sample application: 1 mL spiked Serum

Column washing: 5 mL water

Elution: after drying 3 x 2 mL MeOH

Further analysis: HPLC on NUCLEODUR® 100-5 C_{18}ec, see MN Appl. No.117820

Pesticides from water

MN Appl. No. 304250 and 304260

GIU GmbH, Teningen

Column type: CHROMABOND® HR-X/3 mL/200 mg REF 730931

Sample: 500 ng/L each in water; pH value adjusted

Column conditioning: 10 mL MeOH, 10 mL water

Sample application: slowly aspirate 1 L sample through the column

Drying: stream of nitrogen

Elution: 5 mL MeOH – THF (1:1, v/v)

Recovery rate [%]:

Compound	pH 7	Compound	pH 2
Desisopropylatrazine	90	Metamitrone	86
2,4-Dichlorbenzamide	95	Quinmerac	90
Desethylatrazine	89	Chloridazone	93
Hexazinone	95	Picloram	83
Bromacil	103	Metribuzine	84
Simazine	91	Cyanazine	83
Desethylterbutylazine	89	Metabenzthiazuron	94
Atrazine	88	Chlortoluron	91
Metalaxyl	97	Isoproturon	89
Metazachlor	93	Diuron	91
Propazine	88	Dimethenamid-P	89
Terbuthylazine	86	Linuron	94
Metolachlor	97	Epoxiconazole	86
		Penconazole	90
		Alachlor	93
		Propiconazole-1	89
		Flufenacet	91

Further analysis:
HPLC e.g. on NUCLEODUR® 100-3 C_8 ec
see MN Appl. No. 118610 or 120485

Drugs from water

MN Appl. No. 304240

Column type: CHROMABOND® HR-X/3 mL/200 mg REF 730931

Sample: each 1 µg/L in water

Column conditioning: 5 mL MeOH, 5 mL water

Sample application: slowly aspirate 500 mL water (pH 3) through the column

Column washing: 5 mL water

Elution: after drying 3 x 2 mL acetonitrile

Recovery rate [%]:

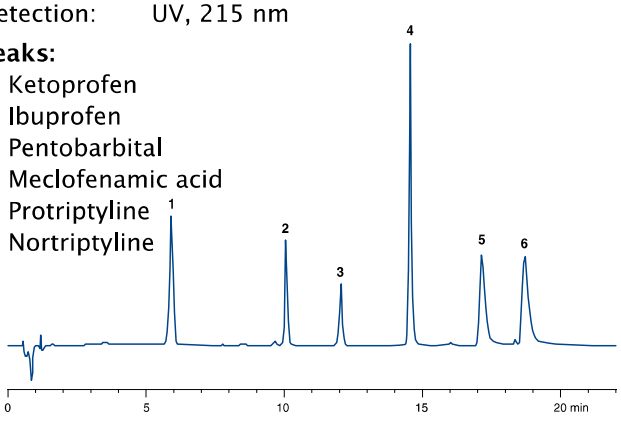
Compound	HR-X	Strata™ X
Ketoprofen	98	92
Ibuprofen	91	93
Pentobarbital	99	95
Meclofenamic acid	92	93
Protriptyline	63	45
Nortriptyline	53	39

Further analysis: HPLC see MN Appl. No. 121690

Column:	125/4 mm NUCLEODUR® C_{18} Gravity, 5 µm
Eluent:	A) 20 mmol KH_2PO_4, pH 7 B) acetonitrile
Gradient:	20 % B → 50 % B in 25 min
Flow rate:	0.5 mL/min
Temperature:	22 °C
Detection:	UV, 215 nm

Peaks:

1. Ketoprofen
2. Ibuprofen
3. Pentobarbital
4. Meclofenamic acid
5. Protriptyline
6. Nortriptyline



Solid Phase Extraction

Solid Phase Extraction

HR-XC

strong cation exchanger

- strong acidic benzenesulphonic acid cation exchanger exchange capacity 1.0 meq/g, pKa <1
- base material polystyrene-divinylbenzene copolymer pH stability 1-14
- high purity material, highest reproducibility and lowest blank values due to an optimized production process
- spherical particles, size 45 µm and 85 µm (standard); pore size 65-75 Å, very large specific surface 800 m^2/g; pore volume 1.4 cm^3/g, RP capacity 300 mg/g (caffeine in water)
- outstanding recovery rates especially for the enrichment of basic analytes
- recommended application:
  basic active ingredients from heavily matrix-contaminated samples like e.g. urine, plasma, serum
  fungicides from food
  melamine from milk products
  basic analytes like e. g. amines
  bases with **pKa 2 - 10**

Standard protocol for CHROMABOND® HR-XC

MN Appl. No. 304790

Column type: CHROMABOND® HR-XC/3 mL, 200 mg, REF 730952

Sample pretreatment: individual sample preparation with reference to analytes and matrix

Conditioning:
5 mL MeOH

Equilibration:
5 mL water

Sample application:
slowly aspirate sample through the column

Washing1:
2 mL 0.1 mol/L HCl in water

Washing 2:/Elution1:
2 mL MeOH
(elution of neutral and acidic compounds)

Elution2:
after drying
5 mL MeOH/5 % NH_3
(elution of basic compounds)

Further analysis: if necessary, evaporate and redissolve in a suitable solvent; HPLC or GC

These conditions are a starting point for SPE method development. Further optimisation may be required to improve results.

Fractionation of acidic, neutral and basic analytes from serum

MN Appl. No. 304780

Column type: CHROMABOND® HR-XC/3 mL, 200 mg, REF 730952

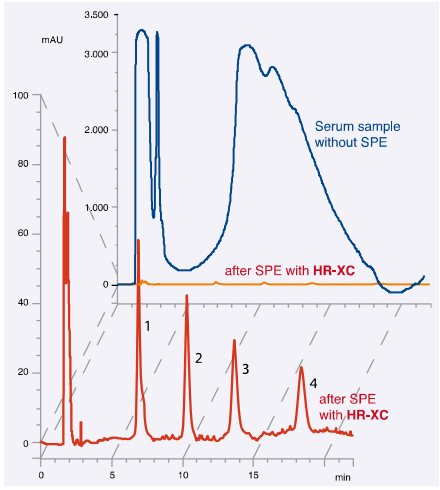
Sample: 1 mL spiked matrix, acidified with 200 µL 2 % H_3PO_4

Column conditioning: 5 mL MeOH, then 5 mL water

Sample application: 1.2 mL spiked serum

Washing: 2 mL 0.1 N HCl in water

Elution: a) 2.5 mL MeOH
Fraction A: neutral and acidic analytes
b) 5 mL MeOH/10 % NH_3
Fraction B: basic analytes



Peaks:
1. Doxepin
2. Imipramine
3. Amitriptyline
4. Trimipramine

Recovery rates [%]:

Fraction A: acidic and neutral analytes

Fraction B: basic analytes

Compound	HR-XC
Suprofen	108
Naproxen	85
Tolmetin	73
Phenobarbital	108
Indomethacin	33
Hexobarbital	80

Compound	HR-XC	Oasis® MCX	Strata™ X-C
Doxepin	101	68	82
Imipramine	95	71	85
Amitriptyline	94	72	78
Trimipramine	92	70	81

Further analysis: HPLC on NUCLEODUR® C18 e.g. C8 Gravity; see MN Appl. No. 122230 and 118520

HR-XA

strong anion exchanger

- strong basic quaternary ammonium anion exchanger
  exchange capacity 0.25 meq/g, pKa ~ 18
- base material polystyrene-divinylbenzene copolymer
  pH stability 1 – 14
- high purity material with highest reproducibility and lowest blank values due to an optimized production process
- spherical particles, size 45 µm and 85 µm (standard); pore size 55 – 65 Å, very large specific surface 850 m^2/g; pore volume 1.4 cm^3/g, RP capacity 350 mg/g (caffeine in water)
- outstanding recovery rates especially for the enrichment of acidic analytes
- recommended application:
  acidic active ingredients from heavily matrix-contaminated samples like e. g. urine, plasma, serum
  phenolic acids
  acidic herbicides
  weak/medium-strength acids with **pKa 2 – 8**

Acidic Pharmaceuticals from serum

MN Appl. No. 305000

Column type: CHROMABOND® HR-XA/3 mL, 200 mg, REF 730951

Sample: 5 mL spiked serum (1 µg/ml each), adjusted basic with 1 N NaOH

Column conditioning: 5 mL MeOH

Equilibration: 5 mL water

Sample application: apply sample

Washing: a) 2.5 mL water
b) 2.5 mL 0,1 N NaOH
c) 5 mL MeOH

Elution: after drying, 5 mL MeOH /1 % formic acid

Further analysis: HPLC on NUCLEODUR® C18 Gravity; see MN Appl.-No. 122840

Recovery rate [%]:

Compound	HR-XA	Oasis® MAX
Ketoprofen	90	85
Fenoprop	104	123
Fenoprofen	98	69
Flurbiprofen	106	98
Ibuprofen	88	58
Carprofen	69	89
Diclofenac	95	94
Meclofenamic acid	92	93

Further applications on CHROMABOND® HR-XA

- Appl. No. 304980 Acidic Herbicides
- Appl. No. 305020 Fractionation of Acidic and Basic Analytes from serum
- Appl. No. 304990 Phenolic Acids

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Standard protocol for CHROMABOND® HR-XA

MN Appl. No. 304970

Column type: CHROMABOND® HR-XA/3 mL, 200 mg, REF 730951

Sample pretreatment: individual sample preparation with reference to analytes and matrix

Conditioning:
5 mL MeOH

Equilibration:
5 mL water

Sample application:
slowly aspirate sample through the column

Washing1:
2 mL 0.1 mol/L NaOH in water

Washing 2:/Elution1:
2 mL MeOH
(elution of neutral and basic compounds)

Elution2:
after drying 5 mL MeOH
1–10 % formic acid
(elution of acidic compounds)

Further analysis: if necessary, evaporate and redissolve in a suitable solvent; HPLC or GC

These conditions are a starting point for SPE method development. Further optimisation may be required to improve results.

CHROMABOND® HR-XCW

HR-XCW

- weak acidic carboxylic acid cation exchanger
  exchange capacity >0.7 meq/g, pKa ~ 5
- base material spherical PS/DVB copolymer
  pH stability 1–14
- high purity material, highest reproducibility and
  lowest blank values due to an optimized production process
- spherical particles, size 45 µm and 85 µm (standard); pore size 50–60 Å
  very large specific surface 850 m^2/g; pore volume 1.2–1.4 cm^3/g
  RP capacity 350 mg/g (caffeine in water)
- outstanding recovery rates especially for enrichment of strongly basic analytes

weak cation exchanger

- recommended application:
  basic compounds like quaternary amines
  active ingredients from heavily matrix-contaminated samples like e.g. urine, plasma, serum
  strong bases with **pKa > 10**

Standard protocol for CHROMABOND® HR-XCW

MN Appl. No. 305300

Column type: CHROMABOND® HR-XCW/3 mL, 200 mg, REF 730739

Sample pretreatment: individual sample preparation with reference to analytes and matrix

Conditioning:
5 mL MeOH

Equilibration:
5 mL water

Sample application:
slowly aspirate sample through the column

Washing1:
2 mL acidified water
(1–5 % formic acid)

Washing 2:/Elution1:
2 mL MeOH
(elution of neutral and acidic compounds)

Elution2:
after drying 2 x 2 mL MeOH / 1–5 % formic acid (elution of strongly basic compounds)

Alternatively elution 2 can be achieved by basic methanol (NH_3), e.g. for primary to tertiary amines. Deprotonation of the analytes interrupts the interaction with the cation exchange resin.

Further analysis: if necessary, evaporate and redissolve in a suitable solvent; HPLC or GC

These conditions are a starting point for SPE method development. Further optimisation may be required to improve results.

Tricyclic Antidepressants

MN Appl. No. 305340

Column type: CHROMABOND® HR-XCW/3 mL, 60 mg REF 730735

Sample: 250 µL spiked Serum, diluted with 1 mL 10 % formic acid in water

Conditioning: 3 mL MeOH

Equilibration: 3 mL water

Sample application: slowly aspirate sample through the column

Washing: 1 mL 5 % formic acid in water, then 1 mL MeOH

Elution: after drying by vaccum (15 min) 3 mL 5 % formic acid in MeOH

Further analysis:
evaporate and redissolve in a suitable solvent for HPLC on NUCLEODUR® C8; Gravity see MN Appl. No. 118520

Recovery rate [%]:

Compound	HR-XCW	HR-XC*	PCA**	Oasis® WCX
Doxepine	**79**	5	11	41
Imipramine	**79**	9	20	67
Amitriptyline	**91**	9	14	46
Trimipramine	**98**	7	14	27

* **HR-XC:** Basic analytes can not be eluted with slightly acidic organic conditions from the strong cation exchanger CHROMABOND® HR-XC, because the eluting power is not sufficient to dissociate the interaction with the ion exchanger. However, with the usage of basic methanol a complete elution can be achieved (please see also MN Appl. No. 304780).

** **PCA:** Due to the missing RP interactions of silica based weak cation exchanger, CHROMABOND® PCA gives only a small enrichment elution of the analytes.

HR-XAW

- weak basic secondary and tertiary ammonium anion exchanger
  exchange capacity >0.5 meq/g, pKa ~ 6
- base material spherical PS/DVB copolymer
  pH stability 1–14
- high purity material with highest reproducibility and
  lowest blank values due to an optimized production process
- spherical particles, size 45 µm and 85 µm (standard); pore size 55–65 Å
  very large specific surface 850 m^2/g; pore volume 1.2–1.4 cm^3/g
  RP capacity 350 mg/g (caffeine in water)
- outstanding recovery rates especially for enrichment of acidic analytes

weak anion exchanger

- recommended application:
  perfluorinated surfactants
  acidic compounds like sulfonates
  active ingredients from heavily matrix-contaminated samples like e.g. urine, plasma, serum
  strong acids with **pKa < 1**

Impregnated with fluorosurfactents?

Surfactants from water

MN Appl. No. 305140

Column type: CHROMABOND® HR-XAW/3 mL, 60 mg, REF 730747

Sample: 500 mL water, spiked with 1 mL standard solution (20 µg/L of each compound)

Conditioning: 2 mL MeOH + 5 % ammonia, then 2 mL methanol

Equilibration: 2 mL water

Sample application: slowly aspirate sample through the column

Washing: 1. 2 mL water
2. 2 mL acetone – acetonitrile – formic acid (50:50:1, v/v/v),
3. 2 mL MeOH

Elution: after drying by vaccum 2 mL methanol + 5 % ammonia

Futher analysis:
evaporate to dryness in a stream of nitrogen under slight heating, and redissolve in a suitable solvent for HPLC on NUCLEODUR® Sphinx RP;
see MN Appl. No. 123340

Recovery rate [%]:

Compound	HR-XAW	Oasis® WAX
Perfluoropropionic acid (PFPrA)	103	76
Perfluoropentanoid acid (PFPeA)	94	81
Perfluorohexanoic acid (PFHxA)	94	91
Perfluoroctanoic acid (PFOA)	95	95
Perfluoroctane sulfonate K salt (PFOS)	81	80
Perfluorododecanoic acid (PFDoDA)	82	34

Application in accordance with **DIN 38407-42.**

Standard protocol for CHROMABOND® HR-XAW

MN Appl. No. 305200

Column type: CHROMABOND® HR-XAW/3 mL, 200 mg, REF 730748

Sample pretreatment: individual sample preparation with reference to analytes and matrix

Conditioning:
5 mL MeOH

Equilibration:
5 mL water

Sample application:
slowly aspirate sample through the column

Washing1:
25 mM ammonium acetate in water

Washing 2:/Elution1:
2 mL MeOH
(elution of neutral and basic compounds)

Elution2:
after drying 2 x 2 mL MeOH / 1–5 % NH_3 (elution of strongly acidic compounds)

Alternatively elution 2 can be achieved by acidified methanol (formic acid). Protonation of the analytes interrupts the interaction with the anion exchange resin.

Further analysis: if necessary, evaporate and redissolve in a suitable solvent; HPLC or GC

These conditions are a starting point for SPE method development. Further optimisation may be required to improve results.

Further CHROMABOND® Applications

Alkaloids from serum

MN Appl. No. 304300

Column type: CHROMABOND® HR-X/3 mL/200 mg REF 730931

Sample pretreatment: Serum samples are spiked with alkaloids (each 2 µg/mL).

Conditioning: 5 mL MeOH

Equilibriation: 5 mL water (Do not let run the column dry!)

Sample application: 1 mL spiked serum sample is passed through the column by vacuum

Washing: 5 mL water

Drying: with nitrogen or air

Elution: after drying 3 x 2 mL acetone

Further analysis: HPLC with NUCLEODUR® C_{18} Gravity, according to MN Appl. No. 117950

Recovery rates [%]:

Compound	HR-X
Atropine	99
Papvarine	97
Noscapine	95
Strychnine	94
Quinine	60

Fungicides in fruit juice

MN Appl. No. 304740

Column type: CHROMABOND® HR-XC/3 mL/200 mg REF 730952

Sample pretreatment: 5 mL spiked apple juice (c=1 µg/mL) + 0.5 mL 0.1 N NaOH

Conditioning: 5 mL MeOH

Equilibriation: 5 mL 2 % NH_3 (Do not let run the column dry!)

Sample application: prepared sample is passed through the column by vacuum.

Washing:

a) 2 mL 2 % NH3

b) 2 mL 30 % MeOH/5 % NH_3

c) 2 mL 0.1 N HCl

d) 2 mL MeOH

Drying: with nitrogen or air

Elution: 4 mL MeOH/5 % NH_3

Evaporation to dryness and reconstitution with 1 mL of mobile phase from subsequent HPLC

Further analysis: HPLC with NUCLEODUR® C_{18} Gravity, according to MN Appl. No. 122200

Recovery rates [%]:

Compound	HR-XC
Carbendazime	89
Thiabendazole	92

Extraction of melamine from milk

MN Appl. No. 304920

Column type: CHROMABOND® HR-XC/3 mL/200 mg REF 730952

Step 1: Add 1 mL 1N HCl to 10 mL spiked milk (1 µg/mL or 0.1 µg/mL); then add 10 mL methylene chloride.

Step 2: vortex to mix, centrifuge for 15 min at 4500 rpm, remove the aqueous layer

add 5 mL 0.1 N HCl to the organic layer

repeat twice step 2

At least combine all aqueous phases for SPE.

Conditioning: 5 mL MeOH

Equilibriation: 5 mL water (Do not let run the column dry!)

Sample application: The prepared sample is passed through the column by gravity or low vacuum.

Washing: 5 mL 0,1 N HCl + 5 mL MeOH

Drying: 10 min vacuum

Elution: 2 x 2.5 mL MeOH/5 % NH_3

Evaporation with nitrogen stream and reconstitution with a suitable solvent for the subsequent analysis

Further analysis: HPLC with NUCLEODUR® 100-5 C_8 ec, according to MN Appl. No. 122670

Recovery rates [%]:

Compound	HR-XC
1 µg melamine in 1 mL milk	99
0.1 µg melamin in 1 mL milk	88

Extraction of campher sulfonic acid from serum

MN Appl. No. 305320

Column type: CHROMABOND® HR-XAW/3 mL/60 mg REF 730747

Sample pretreatment: Stock solution: Camphor sulfonic acid in water (c=0.5 mg/mL)

Standard solution: 5 µL stock solution is diluted to 10 mL (c=0.25 µg/mL)

Sample solution: 5 mL serum is spiked with 5 µL stock solution and diluted with 4 % H_3PO_4 to 10 mL

Conditioning: 2 mL MeOH

Equilibriation: 2 mL water (Do not let run the column dry!)

Sample application: 2 mL sample solution is passed through the column by vacuum.

Washing: 2 mL ormic acid in water, then 2 mL MeOH

Drying: with vacuum

Elution: 2 mL MeOH + 5 % ammonia

Evaporation to dryness (mild nitrogen stream, slightly warming), reconstitution in suitable eluent

Further analysis: HPLC-MS mit NUCLEODUR® C_{18} Gravity, according to MN Appl. No. 123520

Recovery rate: 98.9 %

Allantoin in cosmetic products

MN Appl. No. 305440

Column type: CHROMABOND® HR-XA/3 mL/60 mg
REF 730950

Sample pretreatment: mix 1 g of cosmetic product with 100 mL of water and adjust pH to 10 – 11 with conc. ammonia solution.

Conditioning: 1 mL MeOH

Equilibriation: 1 mL ammonium hydroxide solution ($w(NH_3)$ = 5 %)

Sample application: slowly force or aspirate 1 mL sample through the column.

Washing: 1 mL ammonium hydroxide solution ($w(NH_3)$ = 5 %, then 1 mL MeOH (do not dry the column completely!)

Elution: 2 x 600 µL HCl (w(HCl) = 0.6 %)

Fill up to 10 mL with acetonitrile/30 mM ammonium chloride, pH 3.0 (80:8, v/v) for HPLC analysis.

Further analysis: HPLC with NUCLEODUR® HILIC, according to MN Appl. No. 305446

Desalting and selective Elution of peptides and proteins

MN Appl. No. 305290

Column type: CHROMABOND® HR-X/1 mL/30 mg
REF 730934

Conditioning: 1 mL acetonitrile

Equilibriation: 1 mL de-ionized water (0.1 % TFA)

Sample application: Slowly force or aspirate the sample through the column.

Washing: 2 x 1 mL de-ionized water (0.1 % TFA)

Elution: 250 µL of
40:60 ACN/water (acidic peptides)
70:30 ACN/water (neutral peptides)
70:30 ACN/1.0 % formic acid (basic peptides)

Acidic herbicides

MN Appl. No. 304980

Column type: CHROMABOND® HR-XA/3 mL/200 mg
REF 730951

Sample pretreatment: 1 µg/mL sample adjusted on basic pH with 1 N NaOH

Conditioning: 5 mL MeOH

Equilibriation: 5 mL water (Do not let run the column dry!)

Sample application: prepared sample is passed through the column by vacuum.

Washing:
a) 2.5 mL 50 mM NaOAc
b) 2.5 mL MeOH

Drying: with nitrogen or air

Elution: 5 mL MeOH/10 % formic acid

Evaporation to dryness and reconstitution with 1 mL of mobile phase from subsequent HPLC

Further analysis: HPLC with NUCLEODUR® C_{18} Gravity, according to MN Appl. No. 122820

Recovery rates [%]:

Compound	HR-XA	Oasis® WAX
Dicamba	87	78
Bentazon	79	72
2,4-D	82	80
MCPA	76	72

Extraction of paraquat and diquat from water

MN Appl. No. 305370

Column type: CHROMABOND® HR-XCW/3 mL/60 mg
REF 730735

Conditioning: 1 mL MeOH

Equilibriation: 1 mL water (Do not let run the column dry!)

Sample application: 1 mL spiked water (0.5 µg/mL)

Washing: 1 mL water

Drying: with nitrogen or air

Elution: 1 mL acetonitrile/water + 2 % formic acid (1:1, v/v)

Evaporation and reconstitution is suitable solvent

Further analysis: HPLC with NUCLEODUR® HILIC, according to MN Appl. No. 123060

Recovery rates [%]:

Compound	HR-XA
Paraquat	103
Diquat	113

Ordering information

CHROMABOND® HR-X — hydrophobic RP-phase

Volume	Adsorbent weight						Pack of
CHROMABOND® HR-X polypropylene columns (85 µm)							
	30 mg	60 mg	100 mg	200 mg	500 mg	1 g	
1 mL	730934		730935				30
3 mL		730936		730931	730937		30
6 mL				730938	730939		30
15 mL					730940	730941	20
CHROMABOND® HR-X polypropylene columns (85 µm) · BIGpacks							
3 mL				730931.250			250
6 mL				730938.250	730939.250		250
CHROMABOND® HR-X polypropylene columns (45 µm) · NEW							
1 mL	730934P45		730935P45				30
3 mL		730936P45		730931P45			30
CHROMABOND® LV-HR-X (85 µm)							
	30 mg	60 mg		200 mg			
15 mL	732130	732131		732132			30
CHROMABOND® MULTI 96 HR-X							
	96 x 10 mg (45 µm)	96 x 25 mg (45 µm)	96 x 50 mg (85 µm)		96 x 100 mg (85 µm)		
	738530.010M	738530.025M	738530.050M		738530.100M		1
CHROMABOND® HR-X adsorbent (85 µm)							
					730663		20 g

CHROMABOND® HR-XC — strong cation exchanger

Volume	Adsorbent weight						Pack of
CHROMABOND® HR-XC polypropylene columns (85 µm)							
	30 mg	60 mg	100 mg	150 mg	200 mg	500 mg	
1 mL	730969		730049				30
3 mL		730956			730952	730953	30
6 mL				730957		730955	30
CHROMABOND® HR-XC polypropylene columns (45 µm) · NEW							
1 mL	730969P45		730049P45				30
3 mL		730956P45			730952P45		30
CHROMAFIX® HR-XC cartridges (85 µm)							
Size adsorbent weight Ø	S 155 mg		M 240 mg			L 500 mg	
	731755		731756			731757	50
CHROMABOND® HR-XC adsorbent (85 µm)							
					730664		100 g

CHROMABOND® HR-XA — strong anion exchanger

Volume	Adsorbent weight						Pack of
CHROMABOND® HR-XA polypropylene columns (85 µm)							
	30 mg	60 mg	100 mg	150 mg	200 mg	500 mg	
1 mL	730968		730727				30
3 mL		730950			730951	730954	30
6 mL				730958		730966	30
CHROMABOND® HR-XA polypropylene columns (45 µm) · NEW							
1 mL	730968P45		730727P45				30
3 mL		730950P45			730951P45		30
CHROMAFIX® HR-XA cartridges (85 µm)							
Size adsorbent weight Ø	S 155 mg		M 240 mg			L 500 mg	
	731768		731769			731770	50
CHROMABOND® HR-XA adsorbent (85 µm)							
					730671		100 g

CHROMABOND® HR-XCW — weak cation exchanger

Volume	Adsorbent weight						Pack of
CHROMABOND® HR-XCW polypropylene columns (85 µm)							
	30 mg	60 mg	100 mg	150 mg	200 mg	500 mg	
1 mL	**730731**		**730733**				30
3 mL		**730735**			**730739**	**730741**	30
6 mL				**730737**		**730743**	30
CHROMABOND® HR-XCW polypropylene columns (45 µm) · NEW							
1 mL	**730731P45**		**730733P45**				30
3 mL		**730735P45**			**730739P45**		30
CHROMAFIX® HR-XCW cartridges (85 µm)							
Size		**S**		**M**		**L**	
adsorbent weight ∅		155 mg		240 mg		500 mg	
		731774		**731775**		**731776**	50
CHROMABOND® HR-XCW adsorbent (85 µm)							
						730674	100 g

CHROMABOND® HR-XAW — weak anion exchanger

Volume	Adsorbent weight						Pack of
CHROMABOND® HR-XAW polypropylene columns (85 µm)							
	30 mg	60 mg	100 mg	150 mg	200 mg	500 mg	
1 mL	**730728**		**730729**				30
3 mL		**730747**			**730748**	**730744**	30
6 mL				**730749**		**730745**	30
CHROMABOND® HR-XAW polypropylene columns (45 µm) · NEW							
1 mL	**730728P45**		**730729P45**				30
3 mL		**730747P45**			**730748P45**		30
CHROMAFIX® HR-XAW cartridges (85 µm)							
Size		**S**		**M**		**L**	
adsorbent weight ∅		155 mg		240 mg		500 mg	
		731771		**731772**		**731773**	50
CHROMABOND® HR-XAW adsorbent (85 µm)							
						730673	100 g

CHROMABOND® HR-X*pert* — development kits

			Pack of
CHROMABOND® polypropylene columns			
10 x HR-X and	**Kit I** 3mL/60mg	**Kit II** 3mL/200mg	
5 x HR-XC, HR-XA, HR-XCW, HR-XAW	**730723** (45 µm)	**730726** (85 µm)	30

Further sizes and adsorbent weights for all HR-X*pert* phases on request!

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