

MACHEREY-NAGEL

Modern polymeric SPE phases

Chromatography



CHROMABOND® HLB and HR-Xperts

- Well-defined portfolio of polymeric SPE phases
- Broad application range
- High performance adsorbents

MACHEREY-NAGEL

www.mn-net.com



Modern polymeric CHROMABOND® SPE phases

Do you want to squeeze the best out of your samples?



CHROMABOND® HLB	Hydrophilic-lipophilic balance NVP / DVB copolymer	page 04 – 07
CHROMABOND® HR-X	Hydrophobic PS / DVB copolymer	page 08 – 09
CHROMABOND® HR-XC	Strong mixed-mode cation exchanger on PS / DVB copolymer basis	page 10 – 11
CHROMABOND® HR-XA	Strong mixed-mode anion exchanger on PS / DVB copolymer basis	page 12 – 13
CHROMABOND® HR-XCW	Weak mixed-mode cation exchanger on PS / DVB copolymer basis	page 14 – 15
CHROMABOND® HR-XAW	Weak mixed-mode anion exchanger on PS / DVB copolymer basis	page 16 – 17

Characteristics

- State-of-the-art spherical polymers with different particle sizes to suit sample volume and matrix
- Optimized pore structure and high specific surface
- High purity adsorber material
- Extremely low bleed values
- High specific surface
- pH stability of 1–14

Good to know

Advantages of polymeric based adsorbents compared to silica based:

- Higher capacity of up to 30 wt % (silica gel 3–5 wt %)
- pH stability of 1–14 (silica gel ~ 2–8)
- Optimized flow rates

Benefits for you

Save time and reduce costs

- Well-defined portfolio of polymer phases to suit your application
- Excellent enrichment of neutral, acidic and basic compounds
- Outstanding price/performance ratio

Robust methodology and less pain during method development

- Good reproducibility
- Cleaner samples and protection of your HPLC and GC instruments
- High loadability and outstanding performance
- Ideal flow properties
- Consistent recoveries

No risk

- Test samples available on request.

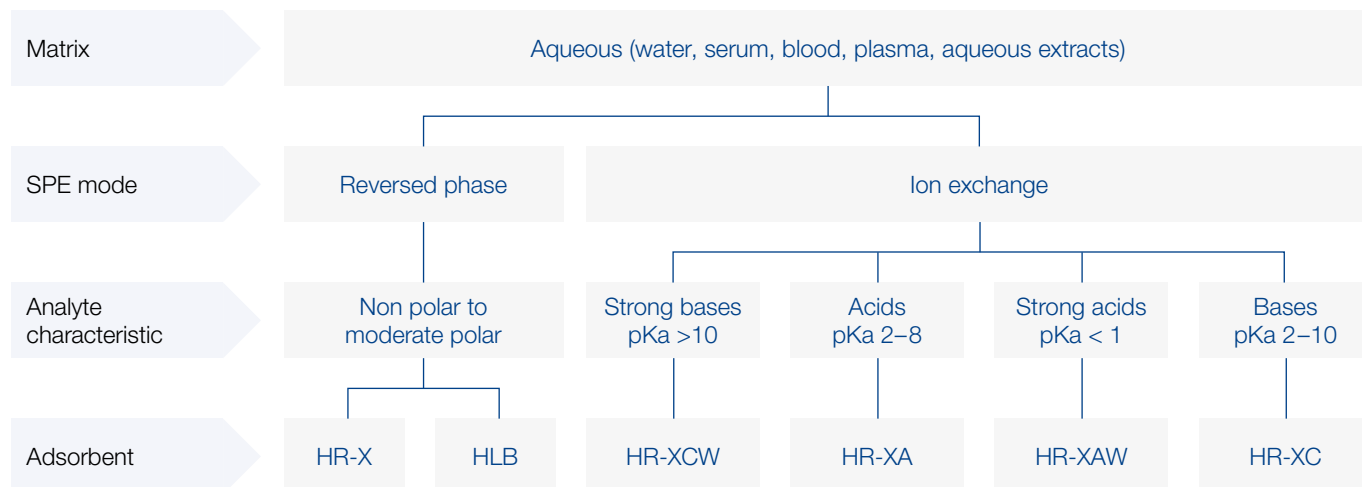


Modern polymeric CHROMABOND® SPE phases

Selection guide

The continuous strive to improve SPE methods led to the development of our portfolio of CHROMABOND® polymer phases.

Stationary phase selection



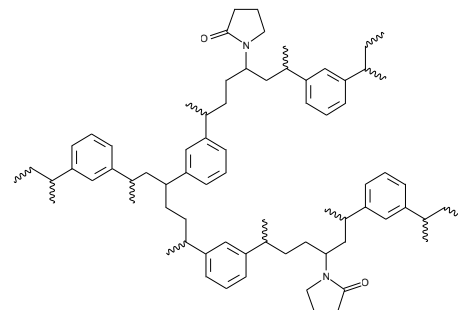
Modern polymeric CHROMABOND® SPE phases

CHROMABOND® HLB

Technical data

Hydrophilic-lipophilic balanced *N*-vinylpyrrolidone-divinylbenzene copolymer (NVP / DVB)

SPE mode:	Reversed phase
Interactions:	Hydrophobic and polar
Particle shape:	Spherical
pH stability:	1–14
Particle size:	60 µm and 30 µm
Pore size:	65 Å
Specific surface:	750 m ² /g



Special characteristics

- Applicable for a wide range of analyte polarities
- High loadability and outstanding performance
- Water wettable – even if bed runs dry, SPE can be continued

Recommended application

- Medium polar organic molecules from polar matrices
- Drugs and pharmaceuticals from urine, blood, serum and plasma
- Tetracyclines and alkaloids from serum
- Pesticides from water

Good to know

A possible replacement for:

- Oasis® HLB
- Strata™-X
- Supel™-Select HLB
- Supra-Poly® HLB
- Isolute® ENV+



Standard SPE procedure for CHROMABOND® HLB (subsequent HPLC analysis)

MN Appl. No. 306300



Column type:
CHROMABOND® HLB / 3 mL / 200 mg, REF 730924

Sample pretreatment:

Individual sample preparation in reference to the compounds and matrix. (Adjust pH value if necessary)

Conditioning:	5 mL methanol, then 5 mL dist. water
Sample application:	Slowly aspirate sample through column
Washing:	5 mL dist. water
Drying:	10 min with applied vacuum
Elution:	8 mL methanol
Evaporation:	Under nitrogen
Reconstitution:	In 1 mL dist. water + 0.1 % formic acid

Standard SPE procedure for CHROMABOND® HLB (subsequent GC analysis)

MN Appl. No. 306310



Column type:
CHROMABOND® HLB / 3 mL / 200 mg, REF 730924

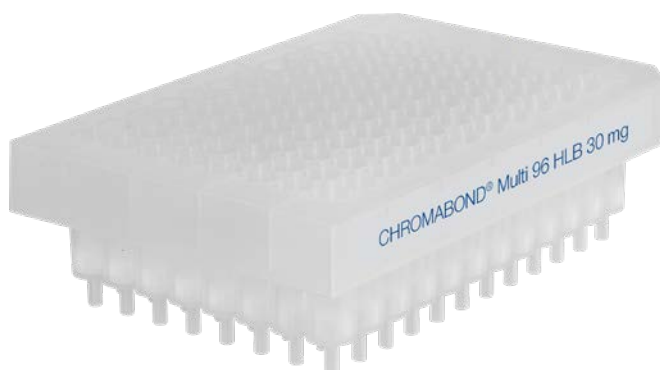
Sample pretreatment:

Individual sample preparation in reference to the compounds and matrix. (Adjust pH value if necessary)

Conditioning:	5 mL solvent (e.g., ethyl acetate), 5 mL methanol, 5 mL dist. water
Sample application:	Slowly aspirate sample through column
Washing:	5 mL dist. water
Drying:	10 min with applied vacuum
Elution:	Solvent ¹⁾ (typical solvents: ethyl acetate, MTBE, methylene chloride)
Evaporation:	Under nitrogen, dry with sodium sulfate ²⁾ , adjust to final volume

¹⁾ usually nonpolar, therefore often 10 % methanol are added

²⁾ e.g., with CHROMAFIX® Dry



Modern polymeric CHROMABOND® SPE phases

Applications

Tetracyclines and alkaloids from serum at pH 5

MN Appl. No. 306380

Chromatographic conditions

Columns: CHROMABOND® HLB / 1 mL / 30 mg
Oasis® HLB / 1 mL / 30 mg
MN REF: 730921
Conditioning: 1 mL methanol, then 1 mL dist. water
Application: 1 mL serum pH 5, adjusted with formic acid
(spiked with 20 µg/mL of each analyte)
Washing: 1 mL dist. water
Drying: 10 min with applied vacuum
Elution: 2 mL methanol
Evaporation: Under nitrogen, 40 °C
Reconstitution: In 1 mL dist. water + 0.1 % formic acid

Further analysis: HPLC, according to MN Appl. No. 128180

Column: EC 50/2 NUCLEOSHELL® RP 18plus, 2.7 µm
MN REF: 763232.20
Eluent: A: dist. water + 0.1 % formic acid
B: acetonitrile + 0.1 % formic acid
Gradient: 2–60 % B in 4 min, 60 % B for 1 min,
60–2 % B in 0.5 min, 2 % B for 3 min
Flowrate: 0.75 mL/min
Temperature: 22 °C
Detection: UV, 330 nm
Injection: 5 µL

Recovery rates ± RSD [%], n = 4

Compound	CHROMABOND® HLB	Oasis® HLB
Berberine	85.4 ± 0.3	82.5 ± 0.6
Chlortetracycline	72.1 ± 1.4	66.3 ± 2.8
Hydrastine	88.9 ± 2.6	99.3 ± 5.7
Oxytetracycline	82.3 ± 1.4	78.7 ± 1.4
Tetracycline	78.1 ± 1.4	70.7 ± 2.6

Mycotoxins in wheat flour

MN Appl. No. 306740

Chromatographic conditions

Columns: CHROMABOND® HLB / 60 µm / 3 mL / 200 mg
MN REF: 730924
Extraction:

- Weigh 4 g homogenized sample in an empty 50 mL centrifuge tube
- Add 8 µL mycotoxin standard mixture (β = 10 µg/mL each analyte in acetonitrile)
- Add 10 mL of water / acetonitrile mixture (20:80, v/v), shake vigorously and wait 10 min
- Add CHROMABOND® QuEChERS extraction Mix XII (REF 730648), shake vigorously for 1 min and cool the mixture down in an ice bath
- Centrifuge at 4500 rpm for 20 min at 20 °C
- Take organic phase for clean-up procedure

Conditioning: 6 mL acetonitrile
Application: 1 mL sample extract was aspirated with low vacuum into a vial
Elution: 4 mL acetonitrile were aspirated with low vacuum into a vial
Evaporation: Combine cleaned sample extract and acetonitrile eluate and evaporate to dryness under nitrogen, 60 °C

Reconstitution: In 1 mL acetonitrile

Analyte	Recovery rate [%]	RSD [%], n = 5
Aflatoxin B1	88	2.6
Aflatoxin B2	91	5.0
Aflatoxin G1	85	2.6
Aflatoxin G2	88	4.5
HT-2 toxin	115	5.7
T-2 toxin	106	5.1
Zearalenone	49	3.4



Modern polymeric CHROMABOND® SPE phases

Applications

Sulfa drugs from serum

MN Appl. No. 306340

Columns*: CHROMABOND® HLB / 60 µm / 1 mL / 30 mg
Oasis® HLB / 60 µm / 1 mL / 30 mg

MN REF: 730921

Conditioning: 1 mL methanol, 1 mL dist. water

Application: 1 mL serum (spiked with 10 µg/mL of each analyte)

Washing: 1 mL dist. water

Drying: 10 min with applied vacuum

Elution: 2 mL methanol

Evaporation: Under nitrogen, 40 °C

Reconstitution: In 1 mL dist. water + 0.1 % formic acid

Further analysis: HPLC, according to MN Appl. No. 128130

Column: EC 150/2 NUCLEODUR® C18 Pyramid, 3 µm

MN REF: 760261.20

Eluent: Dist. water + 0.1 % formic acid / methanol + 0.1 % formic acid (85:15, v/v), 5 min

Flow rate: 0.6 mL/min

Temperature: 25 °C

Detection: UV, 254 nm

Injection: 5 µL

Recovery rates ± RSD [%], n = 5

Compound	CHROMABOND® HLB	Oasis® HLB
Sulfadiazine	97.3 ± 2.9	92.0 ± 3.8
Sulfamerazine	94.4 ± 1.8	92.8 ± 1.6
Sulfathiazole	90.3 ± 2.9	89.6 ± 1.5

Equivalence to Oasis® HLB

CHROMABOND® HLB shows equivalent recovery rates to Oasis® HLB for the three tested sulfa drugs.

Chloramphenicol from honey

MN Appl. No. 306350

Columns*: CHROMABOND® HLB / 60 µm / 3 mL, 200 mg
Oasis® HLB, 3 mL, 200 mg

MN REF: 730924

Sample pretreatment:

Weigh out 5 g of honey. Add 4 mL water and shake rigorously for 30 sec. Spike with 1 mL standard solution (c = 5 ng/mL in methanol) and shake rigorously for 30 sec. Add 15 mL ethyl acetate and shake rigorously for 30 sec. Centrifuge at 3000 rpm for 10 min. Take 12 mL of supernatant for eluent exchange. Evaporate extracts to dryness at 40 °C under a stream of nitrogen. Redissolve residue in 10 mL water.

Conditioning: 3 mL methanol (dispensing speed 1 mL/min), 5 mL dist. water (disp. speed 1 mL/min)

Application: 9 mL water sample (disp. speed 3 mL/min over sample loop)

Washing: 10 mL dist. water (disp. speed 3 mL/min)

Drying: 100 mL air (disp. speed 100 mL/min)

Elution: 5 mL ethyl acetate / methanol (80:20, v/v)

Drying: 100 mL air (disp. speed 100 mL/min)

Evaporation: under nitrogen, 40 °C

Reconstitution: in 1 mL dist. water / acetonitrile (95:5, v/v)

The SPE application was performed with a FREESTYLE® SPE automation system.

Further analysis: LC-MS/MS, according to MN Appl. No. 128140

Column: EC 150/2 NUCLEODUR® π², 5 µm

MN REF: 760624.20

Eluent: A: dist. water
B: acetonitrile
5–95 % B in 7.5 min, 95 % B for 1 min, 95–5 % B in 1 min, 5 % B for 5 min

Flow rate: 0.3 mL/min

Temperature: 35 °C

Detection: MS, Selected Reaction Monitoring (SRM)

Injection: 5 µL

Recovery rates ± RSD [%], n = 5

Compound	CHROMABOND® HLB	Oasis® HLB
Chloramphenicol-d5	90.9 ± 5.4	90.0 ± 9.3

Good to know

Antibiotics and pesticides contamination of agricultural products such as honey has been an issue in the recent years and resulted in stricter guidelines in food safety control.



* Same conditions for all used columns. Due to a better comparability CHROMABOND® HLB and Oasis® HLB adsorbents (60 µm) were packed into equal column hardware. The shown chromatograms may not be representative of other applications.

Modern polymeric CHROMABOND® SPE phases

Applications

Pesticides from tap water

MN Appl. No. 306360



Columns*: CHROMABOND® HLB / 60 µm / 3 mL / 200 mg
Oasis® HLB / 60 µm / 3 mL / 200 mg

MN REF: 730924

Conditioning: 5 mL methanol, 5 mL dist. water

Application: 1000 mL tap water (spiked with 50 ng of each analyte)

Washing: 10 mL dist. water

Drying: 5 min with applied vacuum (-15 psi)

Elution: 6 mL acetonitrile

Evaporation: Under nitrogen, 40 °C

Reconstitution: In 1 mL dist. water / acetonitrile (95:5, v/v)

Further analysis: LC-MS/MS, according to MN Appl. No. 128150



Column: EC 50/2 NUCLEOSHELL® PFP, 2.7 µm

MN REF: 763532.20

Eluent: A: dist. water + 0.1 % formic acid
B: acetonitrile + 0.1 % formic acid
5–95 % B in 15 min, 95 % B for 5 min, 95–5 % B in 1 min, 5 % B for 9 min

Flow rate: 0.3 mL/min

Temperature: 40 °C

Detection: MS, Selected Reaction Monitoring (SRM)

Injection: 5 µL

Recovery rates ± RSD [%], n = 5

Compound	CHROMABOND® HLB	Oasis® HLB
Acetamiprid	73.3 ± 5.0	112.1 ± 9.9
Atrazine	110.3 ± 17.8	114.0 ± 11.6
Azoxystrobin	74.7 ± 5.4	98.1 ± 10.8
Carbaryl	65.7 ± 5.4	69.1 ± 7.1
Chlorotoluron	82.7 ± 5.7	101.2 ± 3.8
Chlorpyrifos	50.3 ± 5.4	47.0 ± 3.7
Clofentezine	27.8 ± 2.7	21.4 ± 3.7
Clothianidin	69.4 ± 6.5	52.9 ± 2.9
Coumaphos	69.8 ± 4.8	82.3 ± 5.2
Cyanazine	99.8 ± 9.3	85.1 ± 7.2
Desethylatrazine	94.8 ± 15.1	87.4 ± 11.4
Desisopropylatrazine	92.5 ± 7.6	N/A
Diazinon	71.5 ± 7.9	73.3 ± 4.7
Difenoconazole	83.9 ± 6.5	28.8 ± 5.0
Diuron	70.0 ± 4.8	80.1 ± 8.4
Ethoprophos	72.4 ± 9.3	85.4 ± 7.2
Hexazinone	88.4 ± 7.7	104.3 ± 7.4
Imazalil	27.3 ± 15.7	N/A
Imidacloprid	93.4 ± 5.1	40.3 ± 5.2
Isoproturon	100.2 ± 4.2	102.8 ± 13.0
Linuron	84.5 ± 7.6	88.3 ± 9.5

Compound	CHROMABOND® HLB	Oasis® HLB
Methabenzthiazuron	72.5 ± 5.3	48.0 ± 3.7
Methomyl	78.8 ± 5.4	83.6 ± 5.6
Metobromuron	73.8 ± 5.6	85.6 ± 9.3
Metolachlor	79.0 ± 5.2	89.2 ± 5.0
Monolinuron	75.4 ± 6.2	97.9 ± 7.2
Myclobutanil	101.8 ± 11.4	88.7 ± 14.5
Phosalone	63.8 ± 7.7	74.0 ± 4.0
Piperonylbutoxide	101.4 ± 8.6	99.7 ± 7.9
Propazine	102.1 ± 13.6	90.9 ± 9.4
Propyzamide	84.8 ± 7.1	86.4 ± 10.6
Terbutylazine	107.9 ± 13.3	100.0 ± 13.6
Thiacloprid	74.1 ± 6.3	86.5 ± 10.8

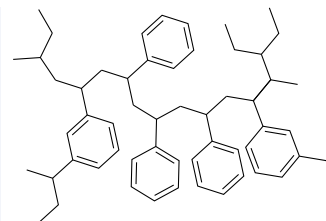


CHROMABOND® HR-X

Technical data

Hydrophobic polystyrene-divinylbenzene copolymer (PS / DVB)

SPE mode:	Reversed phase
Interactions:	Hydrophobic and π - π
Particle shape:	Spherical
pH stability:	1–14
Particle size:	85 μ m and 45 μ m
Pore size:	55–60 Å
Specific surface:	1000 m ² /g
RP capacity:	390 mg/g (caffeine in water)



Recommended application

- Pharmaceuticals / active ingredients from tablets, creams and water
- Drugs and pharmaceuticals from urine, blood, serum and plasma
- Trace analysis of pesticides, herbicides, phenols, PAH and PCBs from water

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 in reference to the compounds and matrix (adjust pH value if necessary).

Conditioning: 5 mL methanol, then 5 mL water
(do not let run the column dry!)

Sample aspiration: The prepared sample is passed through the column by vacuum or pressure (max. 1000 mL sample volume)

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

Drying: With nitrogen or air

Elution: 3 x 2 mL methanol

Further analysis:

Evaporation and reconstitution (if necessary); HPLC or GC

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

Good to know

A possible replacement for:

- Nexus
- ENVI-Chrom P
- Bakerbond H₂O-phobic DVB
- Strata™-X



Modern polymeric CHROMABOND® SPE phases

Applications

Determination of pyrrolizidine alkaloids

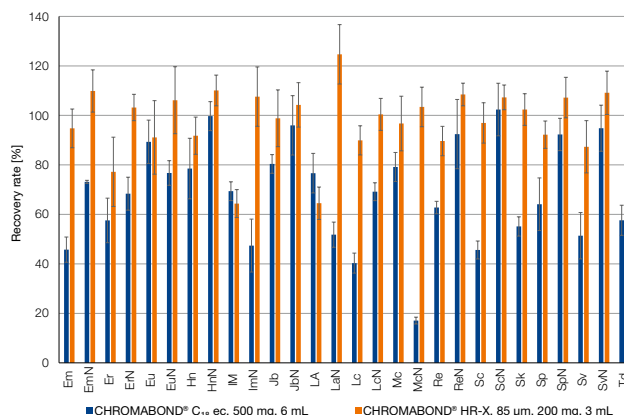
MN Appl. No. 306620

Chromatographic conditions

Columns: CHROMABOND® HR-X/85 µm/3 mL/200 mg
MN REF: 730921
Pretreatment: The following analysis were performed with standard solutions
Conditioning: 5 mL methanol, 5 mL water
Application: 10 mL neutralized standard solution with a flow rate of 3 mL/min
Washing: 2 x 5 mL of water with a flow rate of 3 mL/min
Drying: 5–10 min with vacuum
Elution: 5 mL methanol
Eluent exchange: Add 1.0 mL water as keeper. Evaporate eluate to a volume of 0.5 mL at 40 °C under a stream of nitrogen and fill up to 1.0 mL with water/methanol (95:5, v/v).

Further analysis:

HPLC determination of recovery rates with EC 150/2
 NUCLEOSHELL® RP 18plus, 2.7 µm (REF 763236.20) in reference to MN Appl. No. 127480



Superior to silica based RP phase

CHROMABOND® HR-X shows higher recovery rates for most tested pyrrolizidine alkaloids than CHROMABOND® C18 ec under the given conditions.



Enrichment of opiates

MN Appl. No. 306710

Chromatographic conditions

Columns: CHROMABOND® HR-X/45 µm/3 mL/60 mg
MN REF: 730936P45
Pretreatment: 400 µL methanolic standard solution were diluted with 50 mmol/L phosphate buffer pH 7.0 to 20 mL. 2.5 mL of this solution are equal to 5 ng of each analyte
Conditioning: 3 x 1 mL methanol, 3 x 1 mL water, then 3 x 1 mL 50 mmol/L phosphate buffer pH 7.0
Aspiration: 2.5 mL of pretreated sample solution is passed through the column at a flow of 1–2 mL/min
Washing: 3 x 1 mL 50 mmol/L phosphate buffer pH 7.0, 3 x 1 mL water
Drying: 5 mL air by pushing with a syringe
Elution: 3 x 1 mL 0.1 % formic acid in methanol

Solvent change: Eluate is evaporated to dryness at 30 °C under a stream of nitrogen and then redissolved in organic solvent suited for the subsequent analysis.

Further analysis:

HPLC determination of recovery rates with EC 100/2
 NUCLEOSHELL® Biphenyl, 2.7 µm (REF 763634.20) in reference to MN Appl. No. 128880

Compound	Recovery rate [%]	Standard deviation [%]
Ecgonine methyl ester	94	0
Morphine	77	3
Dihydrocodeine	101	1
Codeine	97	1
6-Acetylmorphine	89	1
Benzoylcodeine	102	0
6-Acetylcodeine	100	0
Cocaine	109	1
Noscapine	95	1
Papaverine	98	2



CHROMABOND® HR-XC

Technical data

Strong cation exchanger based on polystyrene-divinylbenzene copolymer (PS/DVB)

SPE mode: Ion exchange and reversed phase (mixed-mode)

Interactions: Ionic, hydrophobic and π - π

Particle shape: Spherical

pH stability: 1–14

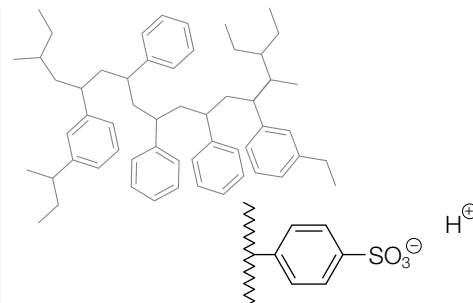
Particle size: 85 μ m and 45 μ m

Pore size: 65–75 Å

Specific surface: 800 m²/g

RP capacity: 300 mg/g (caffeine in water)

Exchange capacity: 1.0 meq/g, pKa < 1



Recommended application

- Basic active ingredients from heavily matrix-contaminated samples, e. g., urine, plasma, serum
- Fungicides from food
- Basic analytes, e. g., amines
- Bases with pKa 2–10

Good to know

A possible replacement for:

- Oasis® MCX
- Strata™-X-C
- StyreScreen® DBX
- HyperSep™ Retain CX

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 in reference to the compounds and matrix (adjust pH value if necessary).

Conditioning: 5 mL methanol, then 5 mL water
(do not let run the column dry!)

Sample aspiration: The prepared sample is passed through the column by vacuum or pressure

Washing 1: 2 mL 0.1 M HCl in water

Washing 2: / Elution 1: 2 mL methanol
(elution of neutral and acidic compounds)

Drying: With nitrogen or air

Elution 2: 5 mL methanol / 5 % NH_3
(elution of basic compounds)

Further analysis:

Evaporation and reconstitution (if necessary); HPLC or GC

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

SPE hardware formats

Check out our different hardware types, e. g., CHROMAFIX® cartridges



Applications

Enrichment of benzodiazepines

MN Appl. No. 306720

Chromatographic conditions

- Columns:** CHROMABOND® HR-XC 45 µm / 3 mL / 60 mg
MN REF: 730956P45
- Pretreatment:** 400 µL methanolic standard solution were diluted with phosphate buffer pH 6.0 to 20 mL
 2.5 mL of this solution are equal to 5 ng of each analyte
- Conditioning:** 2 mL methanol, 2 mL phosphate buffer pH 6.0
- Aspiration:** 2.5 mL of pretreated sample solution is passed through the column at a flow of 1–2 mL/min.
- Washing:** 2 mL phosphate buffer pH 6.0, 2 mL methanol / water (30:70, v/v), 3 mL 0.1 mol/L hydrochloric acid, 2 mL methanol / water (30:70, v/v), 0.1 mL methanol followed by 1 min drying, 2 mL methanol / water (30:70, v/v)
- Drying:** 5 min with a slight nitrogen stream
- Elution:** 2 x 1.5 mL 25 % aqueous ammonia solution / ethylacetate (2:100, v/v)

Solvent change: Eluate is evaporated to dryness at 30 °C under a stream of nitrogen and then redissolved in organic solvent suited for the subsequent analysis.

Further analysis:

HPLC determination of recovery rates with EC 150/2
 NUCLEOSHELL® Bluebird RP 18, 2.7 µm (REF 763436.20)
 in reference to MN Appl. No. 128890

Compound	Recovery rate [%]
Nortetrazepam	85
Tetrazepam	85
α-Hydroxytriazolam	87
Zaleplon	84
Nitrazepam	92
Oxazepam	104
Nordiazepam	83
N-Desmethylflunitrazepam	90
Lorazepam	89
Clonazepam	88
Desalkylflurazepam	102
Temazepam	103
Flunitrazepam	89
Lormetazepam	109
Clobazam	90
Diazepam	98



CHROMABOND® HR-XA

Technical data

Strong anion exchanger based on polystyrene-divinylbenzene copolymer (PS/DVB)

SPE mode: Ion exchange and reversed phase (mixed-mode)

Interactions: Ionic, hydrophobic and π - π

Particle shape: Spherical

pH stability: 1–14

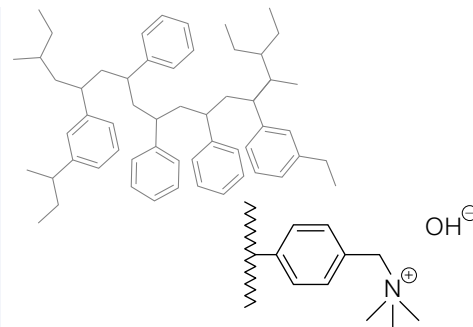
Particle size: 85 μ m and 45 μ m

Pore size: 55–65 Å

Specific surface: 850 m²/g

RP capacity: 350 mg/g (caffeine in water)

Exchange capacity: 0.25 meq/g, pKa ~ 18



Recommended application

- Acidic active ingredients from heavily matrix-contaminated samples, e. g., urine, plasma, serum
- Phenolic acids
- Acidic herbicides
- Weak/medium-strength acids with pKa 2–8

Good to know

A possible replacement for:

- Oasis® MAX
- Strata™-X-A
- HyperSep™ Retain AX
- StyreScreen® QAX

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 in reference to the compounds and matrix (adjust a basic pH value).

Conditioning: 5 mL methanol, then 5 mL water
(do not let run the column dry!)

Sample aspiration: The basic sample is passed through the column by vacuum or pressure (max. 1000 mL sample volume)

Washing 1: 2 mL 0.1 M NaOH in water

Washing 2: / Elution 1: 2 mL methanol
(elution of neutral and basic compounds)

Drying: With nitrogen or air

Elution 2: 5 mL methanol / 1–10 % formic acid
(elution of acidic compounds)

Further analysis:

Evaporation and reconstitution (if necessary); HPLC or GC

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

Successful filtration

We recommend to use CHROMAFIL® Xtra syringe filters in combination with our SPE columns. For further information, please visit www.mn-net.com/chromafil.



Modern polymeric CHROMABOND® SPE phases

Applications

Fractions of acidic and basic analytes from serum

MN Appl. No. 305020

Chromatographic conditions

Column: CHROMABOND® HR-XA / 85 µm / 3 mL / 200 mg

MN REF: 730951

Pretreatment: 1 µg/mL analytes in serum, adjusted on basic pH with 1 N NaOH

Conditioning: 5 mL methanol, then 5 mL water (Do not let run the column dry!)

Aspiration: The prepared sample is passed through the column by vacuum

Washing: With 2.5 mL water impurities are removed

Drying: With nitrogen or air

Elution: Fraction A (basic analytes) is eluted with 5.0 mL methanol
Fraction B (acidic analytes) with 5.0 mL methanol / 10 % formic acid

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

Washing: 1.6 mL acetonitrile, 20 µL/s

Subsequent analysis:

Fraction A: HPLC determination on EC 125/4 NUCLEODUR® C8 Gravity, 5 µm (REF 760751.40) in reference to MN Appl. No. 118520

Fraction B: HPLC determination on EC 125/4 NUCLEODUR® C18 Gravity, 5 µm (REF 760100.40) in reference to MN Appl. No. 122230

Recovery rates:

Fraction A	Recovery [%]	Fraction B	Recovery [%]
Protriptyline	75	Suprofen	96
Nortriptyline	69	Naproxen	86
Doxepine	72	Tolmetin	85
Imipramine	80		
Amitriptyline	78		
Trimipramine	73		

Acidic pharmaceuticals from serum

MN Appl. No. 305000

Chromatographic conditions

Column: CHROMABOND® HR-XA / 85 µm / 3 mL / 200 mg

MN REF: 730951

Pretreatment: 1 µg/mL pharmaceuticals in serum, adjusted on basic pH with 1 N NaOH

Conditioning: 5 mL methanol, then 5 mL water (Do not let run the column dry!)

Aspiration: The prepared sample is passed through the column by vacuum

Washing: With the following washing mixtures impurities are removed: a) 2.5 mL water · b) 2.5 mL 0.1 N NaOH · c) 5.0 mL methanol

Drying: With nitrogen or air

Elution: Analytes are eluted with 5 mL methanol / 1 % formic acid

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

Subsequent analysis:

HPLC determination of recovery rates with EC 125/4 NUCLEODUR® C18 Gravity, 5 µm (REF 760100.40) in reference to MN Appl. No. 122840

Recovery rates:

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



CHROMABOND® HR-XCW

Technical data

Weak cation exchanger based on polystyrene-divinylbenzene copolymer (PS / DVB)

SPE mode: Ion exchange and reversed phase (mixed-mode)

Interactions: Ionic, hydrophobic and π - π

Particle shape: Spherical

pH stability: 1–14

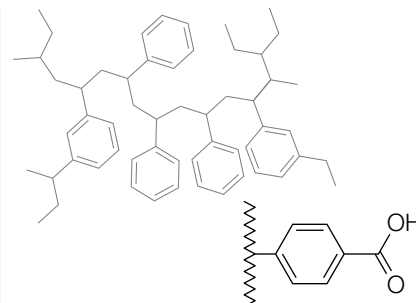
Particle size: 85 μm and 45 μm

Pore size: 50–60 Å

Specific surface: 850 m^2/g

RP capacity: 350 mg/g (caffeine in water)

Exchange capacity: > 0.7 meq/g , $\text{pK}_a \sim 5$



Recommended application

- Basic compounds like quaternary amines
- Active ingredients from heavily matrix-contaminated samples, e.g., urine, plasma, serum
- Strong bases with $\text{pK}_a > 10$

Good to know

A possible replacement for:

- Oasis® WCX
- Strata™-X-CW



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 in reference to the compounds and matrix.

Conditioning: 5 mL methanol, then 5 mL water
(do not let run the column dry!)

Sample aspiration: The sample is passed through the column by
vacuum or pressure (max. 1000 mL sample
volume)

Washing 1: 2 mL 5 % aq. NH_4OH solution

Washing 2: / Elution 1: 2 mL methanol
(elution of neutral and acidic compounds)

Drying: With nitrogen or air

Elution 2: 2 x 2 mL 1–5 % formic acid in methanol
(elution of strongly basic compounds)

Basic methanol (NH_3) can be used alternatively for elution 2 (e.g., for
primary to tertiary amines). Here an interruption of the interactions with
the cation exchanger results by a deprotonation of the analyte.

Further analysis:

Evaporation and reconstitution (if necessary); HPLC or GC

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



HPLC columns

Are you looking for HPLC columns for subsequent analysis?
Find an overview of our HPLC columns under the following link
www.mn-net.com/hplc.



Modern polymeric CHROMABOND® SPE phases

Applications

Tricyclic Antidepressants

MN Appl. No. 305340



Column type:
CHROMABOND® HR-XCW / 85 µm / 3 mL / 60 mg

MN REF: 730735

Pretreatment: 250 µL spiked serum, diluted with 1 mL 10 % formic acid in water

Conditioning: 3 mL MeOH

Equilibration: 3 mL water

Application: Slowly aspirate sample through the column

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

Elution: After drying by vacuum (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 rates:

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.



Modern polymeric CHROMABOND® SPE phases

CHROMABOND® HR-XAW

Technical data

Weak anion exchanger based on polystyrene-divinylbenzene copolymer (PS/DVB)

SPE mode: Ion exchange and reversed phase (mixed-mode)

Interactions: Ionic, hydrophobic and π - π

Particle shape: Spherical

pH stability: 1–14

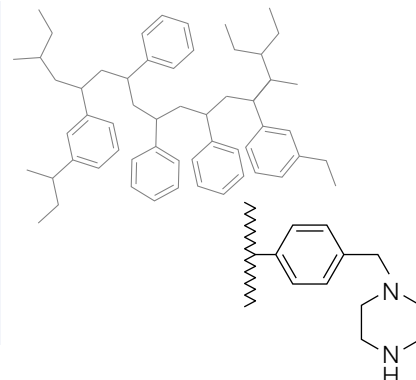
Particle size: 85 μ m and 45 μ m

Pore size: 55–65 Å

Specific surface: 850 m²/g

RP capacity: 350 mg/g (caffeine in water)

Exchange capacity: > 0.5 meq/g, pKa ~ 6



Recommended application

- Perfluorinated surfactants
- Acidic compounds like sulfonates
- Active ingredients from heavily matrix-contaminated samples, e. g., urine, plasma, serum
- Strong acids with pKa < 1

Good to know

A possible replacement for:

- Oasis® WAX
- Strata™-X-AW



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 in reference to the compounds and matrix.

Conditioning: 5 mL methanol, then 5 mL water
(do not let the column run dry!)

Sample aspiration: The sample is passed through the column by vacuum or pressure (max. 1000 mL sample volume)

Washing 1: 25 mM ammonium acetate in water

Washing 2: / Elution 1: 2 mL methanol
(elution of neutral and basic compounds)

Drying: With nitrogen or air

Elution 2: 2 x 2 mL 1–5 % ammonia in methanol
(elution of strongly acidic compounds)

Acidic methanol (formic acid) can be used alternatively for elution 2.
Here an interruption of the interactions with the anion exchanger results by a protonation of the analyte.

Further analysis:

Evaporation and reconstitution (if necessary); HPLC or GC

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



GC columns

For more information on our high performance GC capillary columns, please visit www.mn-net.com/optima.

Modern polymeric CHROMABOND® SPE phases

Applications

Polyfluorinated compounds (PFCs) from fresh and sea water

MN Appl. No. 306730

Chromatographic conditions

- Columns:** CHROMABOND® HR-XAW / 85 µm / 3 mL / 60 mg
MN REF: 730747
- Pretreatment:** 50 mL water sample spiked with PFC standard mixture ($\beta = 0.5$ ng for each analyt in 50 mL water), adjusted to pH value 7–8
- Conditioning:** 2 mL 0.1 % ammonium hydroxide in methanol, 2 mL methanol, 2 mL water
- Aspiration:** Pretreated sample solution is passed through the column at a flow of 5–10 mL/min
- Washing:** 2 mL water, 2 mL 1.0 % formic acid in acetone / acetonitrile (50:50, v/v), 2 mL methanol
- Drying:** No drying
- Elution:** 2.4 mL 0.1 % ammonium hydroxide in methanol
- Solvent change:** Evaporate eluate to dryness at 40 °C under a stream of nitrogen and reconstitute in 0.5 mL water / methanol (40:60, v/v)

Did you know?

Properties of PFCs:

- Persistent in the environment
- Water-, dirt- and fat-repellent; resistant against aggressive chemicals
- Often toxic; many PFCs are bioaccumulative
- Thermally and chemically stable

Daily use of PFCs:

- Fire-fighting foam
- Paper finishing
- Fibre coating
- Textile coating, e.g., seat covers, carpets, outdoor clothing
- Cookware
- Food packaging, e.g., pizza cartons, paper cups
- Building material, e.g., water resistant lacquer

Recovery rates:

Matrix Analyte	Water		Seawater	
	Recovery [%]	RSD [%; n = 3]	Recovery [%]	RSD [%; n = 3]
PFPeA	98	2.9	84	1.6
PFHxA	96	1.7	91	1.3
PFHpA	106	2.9	82	2.4
PFOA	99	2.3	86	2.5
PFNA	114	2.7	93	2.0
PFDA	110	2.6	90	2.3
PFUdA	96	5.3	85	3.5
PFDoA	84	1.6	76	2.1
PFTrDA	75	2.9	70	2.6
PFTeDA	66	4.3	74	4.0
L-PFBS	96	1.6	91	0.7
PFHxS	100	1.6	84	0.8
L-PFHpS	104	1.8	90	3.2
PFOS	103	2.0	84	2.3
L-PFDS	72	4.8	75	3.4
FOSA*	0	–	0	–
N-MeFOSAA*	3	–	0	–
N-EtFOSAA*	2	–	0	–
4:2 FTS	96	1.3	46	2.0
6:2 FTS	108	2.4	53	0.8
8:2 FTS	105	5.2	63	4.5
PFBA**	356	3.6	65	1.8
M ₄ -PFBA**	139	4.0	64	1.4
M ₄ -PFOA	101	3.7	89	2.8
M ₂ -PFHxA	95	2.2	84	0.5
M ₄ -PFHxS	96	2.2	84	1.7
M ₅ -PFNA	107	3.5	90	1.8
M ₄ -PFOS	101	2.4	82	1.2
M ₂ -PFDA	103	3.6	87	3.3
M ₂ -PFDoA	79	3.3	75	2.1
M ₂ -PFUdA	90	3.3	82	2.3

* Due to the organic washing steps, these analytes were eluted into waste.

** In accordance to the properties of the analyte molecules, a not satisfying S/N ratio is received resulting in an improper integration for calculating the recovery rate.

Note: An LC-MS/MS method for determination of polyfluorinated compounds is shown in MN Appl. No 128900



Modern polymeric CHROMABOND® SPE phases

Ordering information

CHROMABOND® HLB

	Volume	Adsorbent weight						Pack of
		30 mg	60 mg	100 mg	150 mg	200 mg	500 mg	1 g
	CHROMABOND® HLB polypropylene columns (60 µm)							
	1 mL	730921		730922				30
	3 mL		730923			730924	730925	30
	6 mL				730944	730926	730927	30
	15 mL						730928	730929 20
	CHROMABOND® HLB polypropylene columns (60 µm) · BIGpacks							
	3 mL		730923.250			730924.250		250
	6 mL					730926.250	730927.250	250
	CHROMABOND® HLB polypropylene columns (30 µm)							
	1 mL	730921P30		730922P30				30
	3 mL		730923P30			730924P30		30
	6 mL				730944P30			30
	CHROMABOND® LV-HLB (30 µm)							
	15 mL	732140	732141					30
	CHROMAFIX® HLB cartridges (60 µm)							
			731921		731922		731923	50
	CHROMABOND® MULTI 96 HLB (60 µm)							
			96 x 10 mg		96 x 30 mg		96 x 60 mg	
							738920.060M	1
	CHROMAFIX® MULTI 96 HLB (30 µm)							
				738921.010M		738921.030M		1

CHROMABOND® HR-X

	Volume	Adsorbent weight 30 mg	60 mg	100 mg	200 mg	500 mg	1 g	Pack of
	CHROMABOND® HR-X polypropylene columns (85 µm)							
	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)							
	1 mL	730934P45		730935P45				30
	3 mL		730936P45		730931P45			30
	CHROMABOND® LV-HR-X (85 µm)							
	15 mL	732130						30
	Adsorbent weight			96 x 100 mg				
	CHROMABOND® MULTI 96 HR-X (85 µm)							
				738530.100M				1

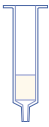
Modern polymeric CHROMABOND® SPE phases

Ordering information (cont.)

CHROMABOND® HR-XC

	Volume	Adsorbent weight					Pack of
		30 mg	60 mg	100 mg	150 mg	200 mg	500 mg
	CHROMABOND® HR-XC polypropylene columns (85 µm)						
	1 mL	730969		730049			30
	3 mL		730956			730952	730953
	6 mL				730957		730955
	CHROMABOND® HR-XC polypropylene columns (45 µm)						
	1 mL	730969P45		730049P45			30
	3 mL		730956P45			730952P45	30
	Size	S		M		L	Pack of
	Minimum adsorbent weight	50 mg		140 mg		400 mg	
	CHROMAFIX® HR-XC cartridges (85 µm)						
		731755		731756		731757	50

CHROMABOND® HR-XA

	Volume	Adsorbent weight					Pack of
		30 mg	60 mg	100 mg	150 mg	200 mg	500 mg
	CHROMABOND® HR-X polypropylene columns (85 µm)						
	1 mL	730968		730727			30
	3 mL		730950			730951	730954
	6 mL				730958		730966
	CHROMABOND® HR-XA polypropylene columns (45 µm)						
	1 mL	730968P45		730727P45			30
	3 mL		730950P45			730951P45	30
	Size	S		M		L	Pack of
	Minimum adsorbent weight	70 mg		180 mg		510 mg	
	CHROMAFIX® HR-XA cartridges (85 µm)						
		731768		731769		731770	50

CHROMABOND® HR-XCW

	Volume	Adsorbent weight					Pack of
		30 mg	60 mg	100 mg	150 mg	200 mg	500 mg
	CHROMABOND® HR-XCW polypropylene columns (85 µm)						
	1 mL	730731		730733			30
	3 mL		730735			730739	730741
	6 mL				730737		730743
	CHROMABOND® HR-XCW polypropylene columns (45 µm)						
	1 mL	730731P45		730733P45			30
	3 mL		730735P45			730739P45	30
	Size	S		M		L	Pack of
	Minimum adsorbent weight	60 mg		160 mg		450 mg	
	CHROMAFIX® HR-XCW cartridges (85 µm)						
		731774		731775		731776	50

Modern polymeric CHROMABOND® SPE phases

Ordering information (cont.)

CHROMABOND® HR-XAW

	Volume	Adsorbent weight					Pack of
		30 mg	60 mg	100 mg	150 mg	200 mg	500 mg
	CHROMABOND® HR-XAW polypropylene columns (85 µm)						
	1 mL	730728		730729			30
	3 mL		730747			730748	30
	6 mL				730749		30
	CHROMABOND® HR-XAW polypropylene columns (45 µm)						
	1 mL	730728P45		730729P45			30
	3 mL		730747P45			730748P45	30
	Size	S		M		L	Pack of
	Minimum adsorbent weight	50 mg		120 mg		360 mg	
	CHROMAFIX® HR-XAW cartridges (85 µm)						
		731771		731772		731773	50

Registered trademarks

Oasis®	Waters Corp. (USA)
CHROMABOND®	MACHEREY-NAGEL GmbH & Co. KG (Germany)
CHROMAFIX®	MACHEREY-NAGEL GmbH & Co. KG (Germany)
FREESTYLE®	LCTech GmbH (Germany)
Strata™	Phenomenex Inc. (USA)
Isolute®	Biotage® AB (Sweden)
Supelclean™ ENVI™	Sigma-Aldrich Inc. (part of Merck KGaA, Germany)
BakerBond®	J. T. Baker® (part of Avantor™) (USA)
Supra-Poly®	PerkinElmer® Inc. (USA)
StyreScreen®	United Chemical Technologies (USA)
HyperSep™	Thermo Fisher Scientific Inc. (USA)
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