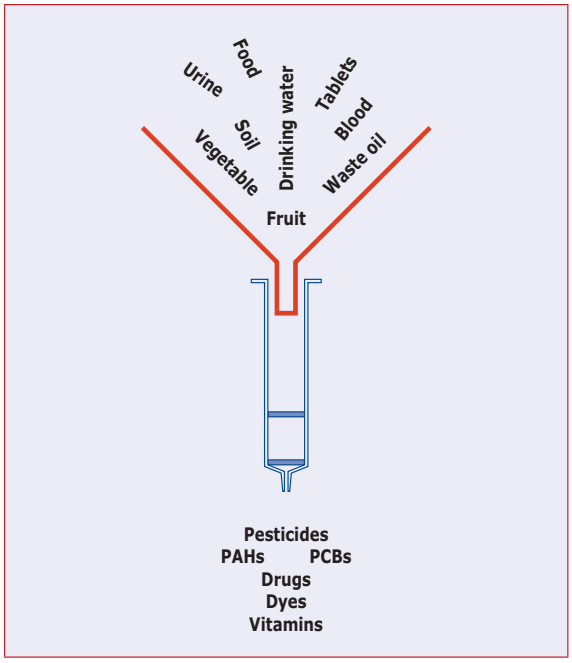
Basic principles of SPE

Solid phase extraction (SPE) is a powerful method for sample preparation and is used by most chromatographers today.

More than 20 years ago MACHEREY-NAGEL designed and introduced CHROMABOND® SPE cartridges containing silica-based adsorbents. Since then we developed the widest range of phases and products for SPE based on silica and polymeric materials.

SPE has capabilities in a broad range of applications:

- environmental analyses
- pharmaceutical and biochemical analyses
- organic chemistry
- food analysis



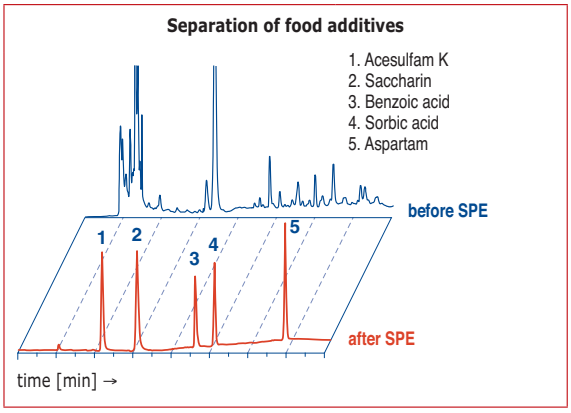
SPE is a form of digital (step-wise) chromatography designed to extract, partition, and/or adsorb one or more components from a liquid phase (sample) onto a stationary phase (adsorbent or resin). An adsorbed substance can be removed from the adsorbent by step-wise increase of elution strength of the eluent (step gradient technique). SPE extends a chromatographic system's lifetime, improves qualitative and quantitative analysis, and the demand placed on an analytical instrument is considerably lessened.

In general, SPE is used for three important purposes in state-of-the-art analyses:

- concentration of the analyte
  (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, UV or IR spectroscopy, ...)
- changing an analyte's environment to a simpler matrix more suitable for subsequent analyses

Advantages of SPE compared to classical liquid-liquid extraction:

- lower consumption of solvents
- faster – enormous time savings
- lower costs per sample
- potential for automation
- high consistency in individual sample handling
- more specific selectivity because of the broad range of adsorbents and different retention mechanisms
- optimisation of extraction by variation or adjusting of the solid phase and chromatographic conditions

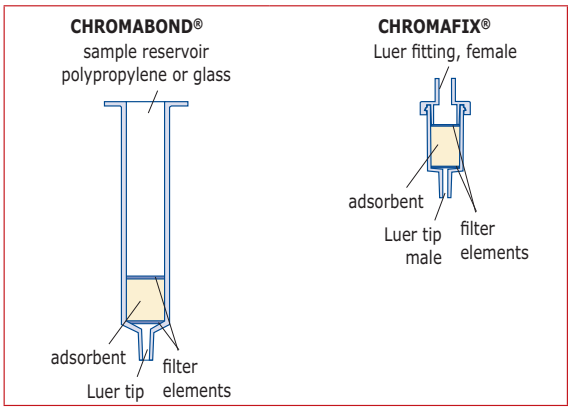


Solid phase extraction

Basic principles and summary of MN phases for SPE

Design of columns and cartridges

All CHROMABOND® columns and CHROMADIX® cartridges are manufactured from polypropylene (PP) with lowest content of extractables (plasticizers, stabilisers, ...) offering blank value free results by usage of most common solvents. The high quality CHROMABOND® adsorbents are kept in place by chemically very inert polyethylene filter elements (PE, standard pore size 20 µm). Funnel-shaped large volume columns, 96-well plates and cartridges for automated and on-line SPE packed with CHROMABOND® adsorbents are available on request.



CHROMABOND® polypropylene columns

- PP columns with PE filter elements
- different sizes from 1, 3, 6 up to 150 ml
- adsorbent weights from 20 mg to 50 g
- male luer tip as exit
- compatible with most robots (e.g., Gilson ASPEC™, Caliper AutoTrace®)

CHROMABOND® glass columns are available on request.

CHROMAFIX® cartridges

- PP cartridges with PE filter elements
- three different sizes with different adsorbent weights: **S**mall (0.4 ml), **M**edium (0.8 ml), **L**arge (1.8 ml))
- female Luer tip at the inlet, male Luer tip as exit
- offers alternative way of handling using positive pressure by syringes or peristaltic pumps
- especially suited for convenient SPE of small sample volumes

Solid phase extraction

Phase	Matrix	Modification / Application	Similar phases*
RP phases			
HR-X	PS/DVB		ENVI-Chrom P, Strata™-X, Oasis® HLB, Nexus
PS-RP	PS/DVB	removal of organic components	Strata™ SDB-L, Bond Elut® ENV, Bond Elut® LMS, DCS-PS/DVB, ENV PS-DVB, Bakerbond™ H_2O-phobic DVB, Isolute® 101
C_{18}	silica	octadecyl, not endcapped	Strata™ C18-U, Accubond® C18, Bakerbond™ PolarPlus, Isolute® C18, LiChrolut® RP-18
C_{18} Hydra	silica	octadecyl, not endcapped, for polar analytes	
Normal phases			
SiOH	silica	unmodified	Strata™ Si-1, Bond Elut® silica, DSC-Si, LC-Si, CLEAN-UP® silica, Accubond® silica, Bakerbond™ silica gel, Isolute® silica, LiChrolut® Si
NH_2	silica	aminopropyl	Strata™ NH_2, Sep-Pak® NH_2, Bond Elut NH_2, DSC-NH_2, LC-NH_2, CLEAN-UP® aminopropyl, Accubond® NH_2, Bakerbond™ amino, Isolute® NH_2, LiChrolut® NH_2
OH	silica	diol	DSC-Diol, LC-Diol, Accubond® Diol (OH)
Alox A/N/B	aluminium oxide	acidic / neutral / basic	LC-Alumina-A/N/B, Accubond® aluminium oxide A/N/B
Florisil®	magnesium silicate		Strata™ FL-PR, Sep-Pak® Florisil®, Bond Elut® Florisil®, ENVI-Florisil®, LC-Florisil®, CLEAN-UP® Florisil®, Accubond® Florisil®, Bakerbond™ Florisil®, Isolute® FL, LiChrolut® Florisil®
PA	polyamide 6		DPA-6S
Ion exchangers			
SA	silica	benzenesulphonic acid cation exchanger (SCX)	Strata™ SCX, Bond Elut® SCX, DSC-SCX, LC-SCX, CLEAN-UP® Benzenesulfonic Acid, Accubond® SCX, Bakerbond™ Aromatic Sulfonic Acid, Isolute® SCX, LiChrolut® SCX
PSA	silica	propylsulphonic acid cation exchanger	
PS-OH^-	PS/DVB	strong anion exchanger, OH^- form	Oasis® MAX
PS-H^+	PS/DVB	strong cation exchanger, H^+ form	Oasis® MCX, Strata™ X-C
PS-Ag^+	PS/DVB	strong cation exchanger, Ag^+ form	
PS-Ba^{2+}	PS/DVB	strong cation exchanger, Ba^{2+} form	
Phases for special applications			
Drug	silica	bifunctional C_8/SA, for enrichment of drugs from urine	Strata™ Screen-C, Bond Elut® Certify I, DSC-MCAX, Clean Screen® DAU, Accubond® Evidex, Bakerbond™ Narc-2, Isolute® HCX, LiChrolut® TSC
Drug II	silica	bifunctional C_8/SB, for extraction of THC and derivatives as well as acidic analytes from biological fluids	Strata™ Screen-A, Bond Elut Certify II, Clean Screen® THC, Bakerbond® Narc-1, Isolute® HAX
CN/SiOH	silica	combination phase for PAHs from soil	
NH_2/C_{18}	silica	combination phase for PAHs from water	
Na_2SO_4/Florisil®		combination phase for extraction of hydrocarbons from water (DIN H-53 / ISO DIS 9377-4)	
SA/SiOH	silica	combination phase for PCBs from waste oil	
Diamino	silica	**p**rimary and **s**econdary amine functions (PSA), for determination of pesticides in food (QuEChERS method)	Supelclean PSA, Bond Elut PSA

* phases which provide a similar selectivity based on chemical or physical properties (list not complete)

1

1 CHROMABOND® HR-X

hydrophobic polystyrene-divinylbenzene copolymer *MACHEREY-NAGEL*
pH stability 1 to14
high-purity material with highest reproducibility and lowest blank values due to a novel manufacturing process
spherical particles 85µm; pore size 55 to 60Å
very high surface 1000m²/g
capacity 390mg/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 optimised pore structure

recommended applications:
pharmaceuticals/active ingredients from tablets, creams and water/waste water
drugs and pharmaceuticals from urine, blood, serum and plasma trace analysis of pesticides.

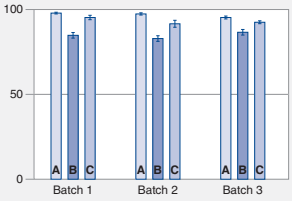
Capacity ml	Capacity mg	PK	Cat. No.
1	30	30	**4.003 808**
3	60	30	**4.003 811**
1	100	30	**4.003 809**
6	200	30	**4.003 814**
3	500	30	**4.003 813**
15	500	20	**4.003 819**
15	1000	20	**4.003 820**
3	200	250	**4.003 806**
6	200	250	**4.003 815**

BIGpacks: 4.003 806/4.003 815
Cartridges in further sizes and phases of the HR-X poduct range available on request.

Highest reproducibility

✓ within each batch ✓ from batch to batch

Compounds:
A phenobarbital
B pentobarbital
C hexobarbital



Barbiturates from serum

Column type:
CHROMABOND® HR-X
3 ml / 200 mg
Sample: 100 ng/ml each in serum
Column conditioning: 5 ml methanol, 5 ml dist. water
Sample application: 1 ml spiked serum
Column washing: 5 ml water
Elution: after drying 3 x 2 ml methanol

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

MN Appl. No. 304290

Standard protocol for method development with CHROMABOND® HR-X

Column type:
CHROMABOND® HR-X
3 ml / 200 mg
Sample pretreatment: if necessary, adjust pH value
Column conditioning: 5 ml methanol
Equilibration: 5 ml water
Sample application: slowly aspirate the sample through the column
Column washing: 5 ml water – methanol (95:5, v/v)
Elution: after drying 3 x 2 ml methanol

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

MN Appl. No. 304310

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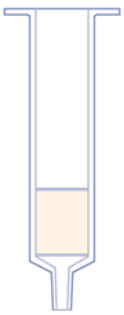

2 CHROMABOND® C18

Octadecyl modified silica phase for SPE, not endcapped *MACHEREY-NAGEL*
Base material silica, pore size 60Å, particle size 45µm for C18, specific surface 500m²/g,
pH stability 2 to 8 octadecyl phases, not endcapped, carbon content 14% possesses more free silanols (SiOH), which allow secondary interactions with polar groups of the analytes

Recommended applications:
non-polar compounds
pesticides

Capacity ml	Capacity mg	PK	Cat. No.
1	100	100	**6.226 798**
3	200	50	**9.003 487**
3*	500	50	**4.003 434**
6*	500	30	**4.003 436**
6*	1000	30	**4.003 438**
6	2000	30	**4.003 512**
15	2000	20	**4.003 464**
45	5000	20	**6.700 747**
70	10000	10	**4.003 561**
3	500	250	**4.003 433**
6	500	250	**4.003 435**
6	1000	250	**4.003 437**

*Glass columns, Polipropylene columns available on request.

1 CHROMAFIX® C18

Octadecyl modified silica phase, not endcapped. *MACHEREY-NAGEL*

Size	Capacity mg	PK	Cat. No.
S	270	50	**7.083 665**
M	530	50	**7.079 617**
L	950	50	**4.003 838**

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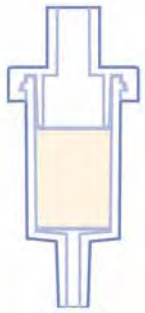

2 CHROMABOND® C18 Hydra

octadecyl modified silica phase for SPE of polar analytes *MACHEREY-NAGEL*

base material silica, pore size 60Å, particle size 45µm, specific surface 500m²/g, pH stability 2 to 8, special octadecyl phase for polar analytes, not endcapped, carbon content 15%.

Recommended applications:
more polar compounds like pesticides and their polar degradation products, phenols, phenoxycarboxylic acids, nitroaromatics, pharmaceuticals.

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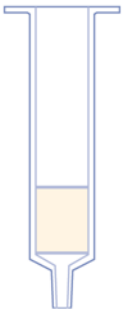

Capacity ml	Capacity mg	PK	Cat. No.
1	50	100	**4.003 565**
1	100	100	**4.003 566**
3	200	50	**4.003 567**
3	500	50	**4.003 569**
6	500	30	**4.003 573**
3	1000	50	**4.003 571**
6	1000	30	**4.003 575**
6	2000	30	**4.003 576**
6	3000	30	**4.003 577**

3 CHROMABOND® NH_2

aminopropyl modified silica phase for SPE *MACHEREY-NAGEL*

base material silica, pore size 60Å, particle size 45µm, specific surface 500m²/g, pH stability 2 to 8, aminopropyl phase, carbon content 3.5%, polar, weak anion exchanger.

Recommended applications:
- trace elements
- lipids

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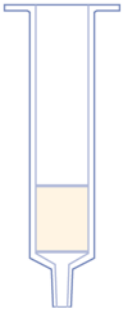

Capacity ml	Capacity mg	PK	Cat. No.
1	100	100	**4.003 465**
3	200	50	**4.003 609**
3*	500	50	**6.205 143**
6*	500	30	**4.003 533**
6*	1000	30	**4.003 678**
3	500	250	**4.003 466**

*Glass columns, Polipropylene columns available on request.
BIGpacks: 4.003 466

4 CHROMABOND® OH

diol modified silica phase for SPE *MACHEREY-NAGEL*

Base material silica, pore size 60Å, particle size 45µm, specific surface 500m²/g, pH stability 2 to 8, diol phase, carbon content 5.5%, polar properties similar to SiOH.

Recommended application:
- antibiotics
- prostaglandins

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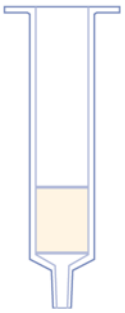

Capacity ml	Capacity mg	PK	Cat. No.
1	100	100	**4.003 470**
3	200	50	**4.003 610**
3	500	50	**4.003 471**
6	500	30	**6.224 847**

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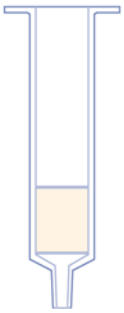

1 CHROMABOND® SiOH

unmodified silica phase for SPE *MACHEREY-NAGEL*

unmodified, weakly acidic silica, pore size 60Å, particle size 45µm, specific surface 500m²/g, pH stability 2 to 8, very polar, adsorbs humidity from air, for this reason it should be kept well closed and if necessary dried before use due to its high affinity for polar compounds it should not be conditioned with polar (e.g. methanol) or water-containing solvents.

Recommended applications:
- aflatoxins
- chloramphenicol
- pesticides
- steroids
- vitamins

Capacity ml	Capacity mg	PK	Cat. No.
3*	200	50	**4.003 548**
3*	500	50	**4.003 479**
6*	500	30	**4.003 475**
6	1000	30	**4.003 481**
6	2000	30	**4.003 498**
15	2000	20	**4.003 550**
45	5000	20	**4.003 605**
70	10000	10	**6.202 850**
150	50000	10	**4.003 630**
3	500	250	**4.003 477**
6	1000	250	**4.003 482**
6	2000	250	**4.003 499**

*Glass columns, Polipropylene columns available on request.
BIGpacks: 4.003 477/4.003 482/4.003 499

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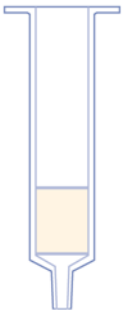

2 CHROMABOND® Alox A/Alox N/Alox B

aluminium oxide, acidic, neutral, basic *MACHEREY-NAGEL*

aluminium oxide, high purity, pore volume 0.90ml/g, particle size 60 to 150µm, specific surface 150m²/g, acidic, pH 4 ±0.5, recommended application: together with phase SA for PCB and pesticides

Alox A: acidic, pH 4 ±0.5
Alox N: neutral, pH 7 ±0.5
Alox B: basic, pH 9.5 ±0.5

Phase	Capacity ml	Capacity mg	PK	Cat. No.
Alox A	3	500	50	**4.003 621**
Alox A	6	500	30	**4.003 622**
Alox A	6	1000	30	**4.003 456**
Alox A	45	4000	20	**4.003 623**
Alox N	3	500	50	**4.003 619**
Alox N	6	500	30	**4.003 620**
Alox N*	6	1000	30	**4.003 515**
Alox N	45	4000	20	**6.226 917**
Alox B	3	500	50	**4.003 615**
Alox B	6	500	30	**4.003 628**
Alox B	6	1000	30	**4.003 458**
Alox B	45	4000	20	**4.003 629**

*Glass columns, Polipropylene columns available on request.

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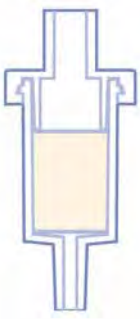

3 CHROMAFIX® Alox N

Aluminium oxide, neutral, pH 7 ±0.5 *MACHEREY-NAGEL*

Size	Capacity mg	PK	Cat. No.
M	850	50	**4.003 853**
L	1700	50	**4.003 854**

1 CHROMABOND® Florisil®

magnesium silicate for SPE *MACHEREY-NAGEL*

matrix magnesium silicate (MgO to SiOH 15:85), high purity, particle size 150 to 250µm.

Recommended application:
- organic tin compounds
- aliphatic carboxylic acids
- PCB, PAH

Capacity ml	Capacity mg	PK	Cat. No.
3	200	50	**4.003 624**
3	500	50	**4.003 488**
6	500	30	**4.003 557**
6	1000	30	**6.224 842**
6	1000	250	**4.003 489**
6*	1000	30	**4.003 490**

BIGpacks: 4.003 489
*Glass columns

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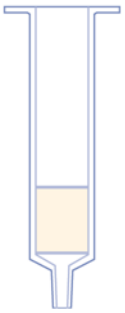

2 CHROMABOND® PA

polyamide 6 for SPE *MACHEREY-NAGEL*

matrix polyamide 6, unmodified, high purity, particle size 40 to 80µm.

Recommended application:
- flavonoids
- PAH

Capacity ml	Capacity mg	PK	Cat. No.
3	200	50	**4.003 595**
3	500	50	**4.003 511**
6	500	30	**7.089 089**
6	1000	30	**7.400 537**

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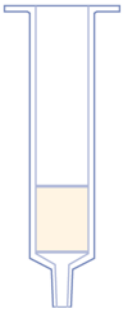

3 CHROMABOND® PSA

propylsulphonic acid modified silica cation exchanger for SPE *MACHEREY-NAGEL*

Base material silica, pore size 60Å, particle size 45µm, specific surface 500m²/g, pH stability 2 to 8, propylsulphonic acid modified silica, very strong cation exchanger (capacity ~ 0.7meq/g), contrary to the SA phase no Π-Π interactions.

Recommended applications:
- weak cations

Capacity ml	Capacity mg	PK	Cat. No.
1	100	100	**4.003 626**
3	500	50	**4.003 627**
6	1000	30	**6.206 233**

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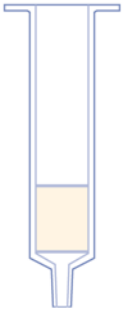

4 CHROMABOND® SA

benzenesulphonic acid modified silica cation exchanger for SPE (SCX) *MACHEREY-NAGEL*

base material silica, pore size 60Å, particle size 45µm, specific surface 500m²/g, pH stability 2 to 8, benzenesulphonic acid modified silica, strongly acidic cation exchanger (capacity ~ 0.5meq/g). Adsorbent with hydrophobic and Π-Π interactions (benzene ring). Ion exchange of organic compounds from aqueous matrix. Elution of interesting compounds with solvent systems, which compensate the ionic and nonpolar interactions, e.g. methanolic HCl.

Recommended application:
- amino acids
- amines
- chlorophyll
- PCB

Capacity ml	Capacity mg	PK	Cat. No.
1	100	100	**6.314 563**
3	200	50	**4.003 563**
3	500	50	**7.051 056**
6	500	30	**4.003 613**
6	1000	30	**6.224 846**
3	500	250	**4.003 485**

BIGpack: 4.003 485

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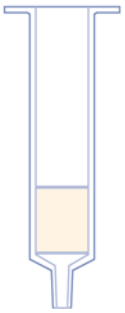

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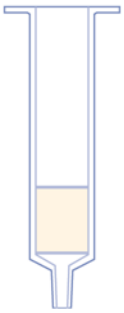


1 SPE phases for RP/ ion chromatography

SPE phases for polymer-based RP and ion chromatography *MACHEREY-NAGEL*

Base material: high-purity polystyrene-divinylbenzene copolymers (PS/DVB)
pore size 100 Å, paricle size 100µm. Verly low degree of swelling, thus very well suited for chromatography. Reliable function over the whole pH range from 0-14. Different modifications for different applications from elimination of nonpolar compounds up to removal of specific polar comonents.

Recommended application:
Removal of interfering compounds
- improves chromatographic separtions, if the interfering components overlap with the analyte in the chromatogram
- improves lifetime of the chromatographic column, since interfering components can irreversibly block the column packing

Enrichment of the analytes

removal of organic interfering components from water	PS-RP hydrophobic PS/DVB-copolymer
removal or concentration of anions from water increasing the pH value in acidic samples removal or concentration of cations from water	PS-OH^- strong PS/DVB anion exanger, OH^- form capacity 0.6 meq/g
decreasing the pH value in basic samples	PS-H^+ strong PS/DVB cation exchanger, H^+ form, capacity 2.9 meg/g
removal of halide ions from water	PS-Ag^+ strong PS/DVB cation exchanger, Ag^+ form
removal of sulphate ions from water	PS-Ba^{2+} strong PS/DVB cation exchanger, Ba^{2+} Form

Phase	Capacity ml	Capacity mg	PK	Cat. No.
PS-OH-	3	200	30	**4.003 600**
PS-H+	3	200	30	**4.003 702**
PS-OH-	3	500	30	**4.003 581**
PS-H+	3	500	30	**4.003 589**
PS-OH-	6	500	30	**4.003 591**
PS-H+	6	500	30	**4.003 590**

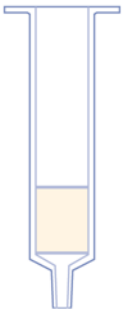
2

2 CHROMAFIX® PS

MACHEREY-NAGEL

Phase	Size	Capacity mg	PK	Cat. No.
PS-RP	S	200	50	**4.003 869**
PS-OH-	S	200	50	**4.003 867**
PS-H+	S	230	50	**4.003 866**
PS-Ag+	S	240	50	**4.003 865**
PS-Ba2+	S	280	50	**4.003 868**
PS-RP	M	320	50	**6.228 258**
PS-OH-	M	380	50	**4.003 861**
PS-H+	M	430	50	**7.401 474**
PS-Ag+	M	480	50	**4.003 864**
PS-Ba2+	M	550	50	**7.402 218**
PS-OH	L	800	50	**4.003 862**
PS-H+	L	900	50	**4.003 863**

3



3 CHROMABOND® Drug

special silica phase for SPE enrichment of drugs from urine or plasma *MACHEREY-NAGEL*

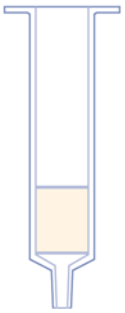
base material silica, pore size 60Å, particle size 45µm, specific surface 500m^2/g, pH stability 2 to 8, special bifunctional modification - C_8/SA (strong cation exchanger - benzenesulphonic acid).

Recommended application:
- enrichment of acidic
- neutral and basic drugs from urine or plasma

Capacity ml	Capacity mg	PK	Cat. No.
1	100	100	**4.003 696**
3	200	50	**6.802 715**
3	500	50	**4.003 699**
6	500	30	**4.003 697**

1 CHROMABOND® Drug II

1



Special silica phase for SPE of THC and derivatives acidic analytes from biological fluids (urine, blood ...) *MACHEREY-NAGEL*

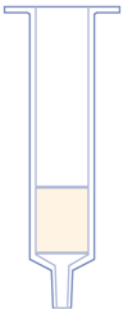
base material silica, pore size 60Å, particle size 45µm, specific surface 500m²/g, pH stability 2 to 8, special bifunctional modification - C8/SB (strong anion exchanger - quaternary amine).

Recommended applications:
- extraction of THC and derivatives from urine, blood, serum, plasma
- acidic analytes from biological fluids

Capacity ml	Capacity mg	PK	Cat. No.
1	100	100	**4.003 700**
3	200	50	**4.003 695**
3	500	50	**4.003 701**
6	500	30	**4.003 698**

2 CHROMABOND® NH_2/C18

2



Combination phase for SPE analysis of PAH from water containing humic acids *MACHEREY-NAGEL*

Special combination phase: aminopropyl phase for removal of interfering humic acids, octadecyl phase for enrichment of PAH

Recommended application:
- PAH from water containing humic acids

Capacity ml	Capacity ml / mg	PK	Cat. No.
6	500 / 500	30	**6.228 257**
6	500 / 1000	30	**4.003 675**

Glass columns available on request.

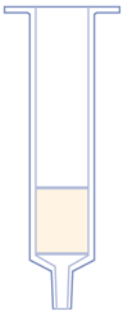
PAHs from water containing humic acids

Column type:
CHROMABOND® NH_2/C18, 6 ml, 500 mg/1 g glass column
Sample pretreatment:
mix 500 ml water sample with 25 ml 2-propanol
Column conditioning: 10 ml dichloromethane, 10 ml methanol, then 10 ml dist. water – 2-propanol (9:1, v/v)
Sample application: aspirate 500 ml of the pretreated water sample through the column (~ 5 ml/min)
Washing: 2 ml dist. water – 2-propanol (9:1, v/v), then dry column (about 20 min, vacuum)
Elution: 4 x 0.5 ml CH_2Cl_2 (percolate first 0.5 ml into the column packing without vacuum, the apply light vacuum), if necessary evaporate in a stream of nitrogen and fill up with a suitable solvent

MN Appl. No. 301260

3 CHROMABOND® CN/SiOH

3



Combination phase for SPE analysis of PAH *MACHEREY-NAGEL*

special combination phase, cyanopropyl phase for selective adsorption of polycyclic aromatics via Π-Π interactions, unmodified silica phase for removal of polar compounds.

Recommended applications:
- extraction of the 16 PAHs according to EPA from soil samples

Capacity ml	Capacity ml / mg	PK	Cat. No.
3	500 / 1000	50	**4.003 507**
6	500 / 1000	30	**6.233 128**
6	500 / 1000	250	**4.003 514**

BIGpack: 4.003 514

PAHs from soil

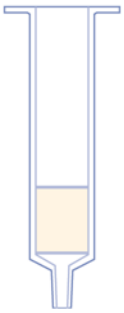
Column type:
CHROMABOND® CN/SiOH,
6 ml, 500/1000 mg
Sample pretreatment: dry 30 g soil with sodium sulphate and reflux 4 h with 250 ml petroleum ether in a Soxhlet extractor. For low PAH contents (colourless or weakly coloured extracts) concentrate extract to 1/10 of its volume in a rotation evaporator.
Column conditioning: 4 ml petroleum ether

MN Appl. Nr. 301310

Sample application:
aspirate 20 ml of the extract through the column
Washing: 2 ml petroleum ether
Elution: 2 x 2 ml acetonitrile / toluene (3:1, v/v), then evaporate or fill to the volume required

Further analysis: HPLC, e.g. with column 250 x 3 mm NUCLEOSIL® 5 C_{18} PAH
recovery rates see application 301310 at *www.mn-net.com*

1



1 CHROMABOND® Na_2SO_4/Florisil®

Combination phase for SPE of hydrocarbons from water acc. to DIN H53/ISO DIS 9377-4 *MACHEREY-NAGEL*

Special combination phase of sodium sulphate and Florisil®.

Recommended application:
- hydrocarbons from drinking, surface and waste waters

Capacity ml	Capacity ml / mg	PK	Cat. No.
6	2000 / 2000	30	**4.003 558**
6*	2000 / 2000	30	**6.900 415**
6*	2000 / 2000	250	**4.003 559**

BIGpacks: 4.003 559
*Glass columns

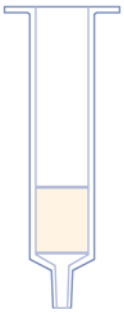
Hydrocarbons from water

Column type:
CHROMABOND® Na_2SO_4/Florisil®,
2000/2000 mg, 6 ml glass column
Internal standard solution: dissolve 20 mg *n*-tetracontane ($C_{40}H_{82}$) in petroleum ether, add 20 ml *n*-decane ($C_{10}H_{22}$) and fill up to 1 litre with petroleum ether. For perparation of the extraction solution dilute standard solution 1:10 with petroleum ether.
Sample pretreatment: adjust 900 ml water (10 °C) with HCl (12 mol/l) to pH 2 and add 80 g $MgSO_4$. Add 50 ml of the extraction solution, close the bottle and stir the suspension intensely for 30 min.

Add enough dist. water to separate the organic from the aqueous phase.
Column conditioning: 5 ml petroleum ether
Sample application:
slowly aspirate or force the sample through the column
Elution: wash with 10 ml petroleum ether. Evaporate the combined solutions from sample application and elution to 1 ml at about 75 °C. If necessary, fill up to 1 ml again. (If the hydrocarbon content is high, evaporation to 1 ml may not be necessary.)
Recovery rate: must be > 80 % for *n*-tetracontane.

MN Appl. No. 302090

2



2 CHROMABOND® SA/SiOH

Combination phase for SPE analysis of PCB *MACHEREY-NAGEL*

Special combination phase:
SA: strongly acidic cation exchanger based on silica with benzenesulphonic acid modification
SiOH: unmodified silica for removal of polar compounds

Recommended application:
- extraction of PCB from waste oil (hexane extract)

Capacity ml	Capacity ml / mg	PK	Cat. No.
3	500 / 500	50	**6.901 798**
3	500 / 500	250	**4.003 513**

BIGpack: 4.003 513

PCBs from waste oil

Column type:
CHROMABOND® SA/SiOH, 3 ml, 500/500 mg

Column conditioning: 1 ml *n*-hexane
Sample application:
apply 250 µl waste oil to the column and aspirate or force it into the adsorbent with 2 x 1 ml *n*-hexane

MN Appl. No. 301390

Elution: aspirate or force another 2 x 500 µl *n*-hexane through the column; collect all *n*-hexane fractions and if necessary adjust to a concentration suitable for subsequent analysis by either evaporting *n*-hexane in a stream of nitrogen or by dilution with *n*-hexane

Recovery rates:
PCB-28 97 %, PCB-52 96 %. PCB-101 95 %, PCB-138 90 %, PCB-153 95 %, PCB-180 96 %, PCB-209 100 %

1 Adsorbent CHROMABOND® Diamino

MACHEREY-NAGEL

Weight g	PK	Cat. No.
100	100	**4.003 688**
20	20	**4.003 689**

1

2 Accessories CHROMABOND® QuEchERS

MACHEREY-NAGEL

Type	PK	Cat. No.
50ml PP-centrifuge tube with crew cap	50	**4.003 552**

2

3 CHROMABOND® vacuum manifolds and accessories

for simultaneous preparation of up to 12, 16 or 24 samples
replacement parts and accessories for special applications

MACHEREY-NAGEL

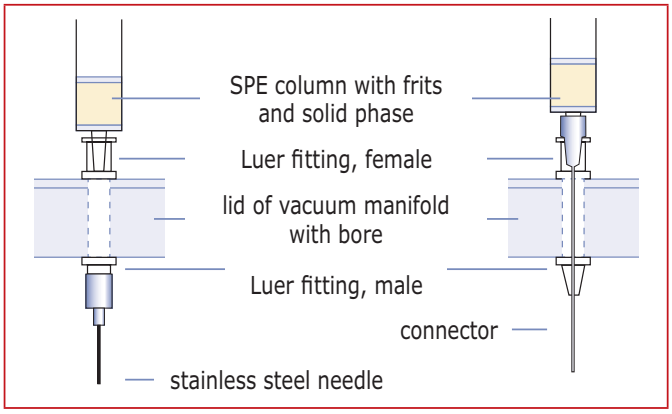
Vacuum manifold complete consists of: glass cabinet with lid and lid gasket, removable needles on lower side of lid, vacuum gauge, control valve, valves and caps, variable rack.

3

Description	PK	Cat. No.
Vacuum manifold complete for up to 12 columns or cartridges (incl. reservoir tank)	1	**9.003 479**
Vacuum manifold complete for up to 16 columns or cartridges	1	**4.003 586**
Vacuum manifold complete for up to 24 columns or cartridges	1	**7.056 914**
Lids with gaskets for 12 columns (incl. Luer fittings and valves), plastic	1	**4.003 530**
Gaskets for lid with 12 positions (4.003 530, 9.003 479)	2	**6.801 608**
Luer fittings for lid, female	12	**4.003 534**
Luer fittings for lid, male	12	**4.003 535**
Valves, plastic	12	**7.089 161**
Stainless steel needles	12	**7.079 432**
Drying attachment for 12 columns	1	**4.003 536**
Products for protection from cross contamination valve, brass, tarnished	1	**4.003 538**
Products for protection from cross contamination valve as above	12	**7.089 162**
Products for protection from cross contamination stainless steel connectors	12	**7.079 431**
Tubing adaptor for 1,3 and 6 ml polypropylene columns (PTFE)	4	**6.900 713**

Protection from cross contamination

For special applications, which require maximum protection from cross contamination we supply chrome-plated brass valves and stainless steel or PTFE connectors, the application of which is shown below. These special connectors are fitted through the lid; thus the sample only has contact with the inert connector and not with the lid, directly flowing into the receptacle.



Drying attachment

If the eluate has to be evaporated, this can be performed with the so-called drying attachment (**11**, see below). This special lid has a gas connector on one side (**12**), from which the gas is fed simultaneously to the 12 or 24 stations (**13**). Thus 12 or 24 eluates can be evaporated simultaneously by just changing the lid and applying a stream of inert gas, e. g. nitrogen.

Syringe filters CHROMAFIL®

Sample clarification

Syringe filters are used for filtration of suspended matter from liquid samples or gases. With CHROMAFIL®, rapid purification and removal of particles is very simple: just place the filter on the syringe, and you are ready for filtration. Special manipulations are not required. Contamination of sensitive instrumentation by solid impurities can be avoided, thus increasing lifetime of chromatographic columns and equipment.

Advantages:

- **Polypropylene housing**
  considerably better solvent stability compared to acrylate and polystyrene filters, low content of extractable substances
- **Housing ultrasonically sealed, not glued**
  no extractable components from glues
- The special **thick rim** of the housing is ideal for use of the filters in laboratory robots (e.g. Benchmate™).
- **Filtration in both directions** possible, the liquid cannot bypass the membrane
- **Luer lock on side of entry**
  safe connection on the "high pressure" side
- **Luer exit**
  standard luer for 25 mm filters, minispike luer with low dead volume and small OD for 15 mm filters. Filter inlet and filter exit can be fitted to the CHROMABOND® columns for selective sample preparation with the aid of a special adaptor.
- **Deflector**
  the stream of liquid is broken and distributed, and does not directly hit the membrane: this prevents rupture of the membrane
- **Star-shaped distribution device**
  the liquid is evenly distributed to the whole membrane surface: this results in a better utilisation of the total area; the filter is not plugged up rapidly; high flow efficiency
- **Colour coded filters**
  filters with 0.2 µm pores have a yellow upper shell, that of filters with 0.45 µm pores is colourless; the different membrane types are distinguished by different colours of the lower shell
- Available **pore sizes** 0.2 and 0.45 µm (exceptions: PET filters with 1.2 µm, glass fibre filters with 1 µm, PES filters with 5 µm)
- **Filter sizes:** 25 and 15 mm diameter The small diameter filters are especially recommended for very small samples, which require extremely low dead volumes: 80 µl for 25 mm ∅, 12 µl for 15 mm ∅
- All filters can be **autoclaved** at **121 °C** and **1.1 bar** for 30 min.

Recommended filter size depending on sample volume

sample volume	recommended filter diameter
1 – 10 ml	15 mm
10 – 100 ml	25 mm

CHROMAFIL® BIG-BOXES

- 400 (25 mm) or 800 (15 mm) quality syringe filters
- food safe PE box with screw cap
- economical price

Depending on your filtration task you can choose filter membranes made from different materials:

Material
Polyester (PET) with or without glass fibre prefilter
Regenerated cellulose (RC)
Teflon® (PTFE)
Cellulose mixed esters (MV)
Cellulose acetate (CA) · sterile and non-sterile
Polyamide / Nylon (PA)
Polyethersulfone (PES) · sterile
Polyvinylidene difluoride (PVDF) with or without glass fibre prefilter
Glass fibre (GF)

Chemical compatibility of filter materials

The following table lists the chemical compatibility of our CHROMAFIL® materials. The chemical compatibility depends on several parameters such as time, pressure, temperature and concentration.

In most cases, CHROMAFIL® filters will have only short contact with a solvent. In these cases they may be used despite of limited compatibility.

For example, a PTFE filter with PP housing does not liberate any UV-detectable substances during filtration of 5 ml THF, although PP shows only limited resistance towards THF.

Solvent	Material									
	MV	CA	RC	PA	PTFE	PVDF	PES	PET	GF	PP
Acetaldehyde	⊖	⊖	⊕	○	⊕	⊕		⊕	⊕	○
Acetic acid, 100 %	⊖	⊖	⊖	⊖	⊕	⊕	⊕	⊕	⊕	⊕
Acetone	⊖	⊖	⊕	⊕	⊕	⊖	⊖	⊕	⊕	⊕
Acetonitrile	⊖	⊖	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕
Ammonia, 25 %	⊖	⊖	○	⊖	⊕	⊕	⊕	○	⊕	⊕
Benzene	⊕	⊕	⊕	⊕	⊕	○		⊕	⊕	○
n–Butanol	⊕	⊕	⊕	○	⊕	⊕	⊕	⊕	⊕	⊕
Cyclohexane	⊕	⊕	⊕	○	⊕	⊕	⊕	⊕	⊕	⊕
Dichloromethane	⊕	⊖	⊕	⊖	⊕	⊕	⊖	⊕	⊕	⊖
Diethyl ether	○	○	⊕	⊕	⊕	⊕	⊕	⊕	⊕	○
Dimethylformamide	⊖	⊖	○	⊕	⊕	⊖	⊖	⊕	⊕	⊕
1,4–Dioxane	⊖	⊖	⊕	⊕	⊕	○	⊖	⊕	⊕	○
Ethanol	⊖	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕
Ethyl acetate	⊖	⊖	⊕	⊕	⊕	⊕	⊕	⊕	⊕	○
Ethylene glycol	○	○	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕
Formic acid, 100 %	⊕	⊖	○	⊖	⊕	⊕	⊕	○	⊕	⊕
Hydrochloric acid, 30 %	⊖	⊖	⊖	⊖	⊕	⊕	⊕	⊖	⊕	⊕
Methanol	⊖	⊖	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕
Nitric acid, 65 %	⊖	⊖	⊖	⊖	○	○		○	⊕	⊖
Oxalic acid, 10 % aqueous	⊕	⊖	⊕	⊖	⊕	⊕		⊕	⊕	⊕
Petroleum ether	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕
Phosphoric acid, 80 %	⊖	⊖	○	⊖	⊕	○		⊕	⊕	⊕
Potassium hydroxide, 1 mol/l	⊖	⊖	○	⊕	⊕	○	⊕	○	⊕	⊕
2–Propanol	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕
Sodium hydroxide, 1 mol/l	⊖	⊖	○	⊕	⊕	○	○	○	○	⊕
Tetrachloromethane	⊕	⊖	⊕	⊕	⊕	○		⊕	⊕	○
Tetrahydrofuran	⊖	⊖	⊕	○	⊕	⊕	⊖	⊕	⊕	○
Toluene	⊕	⊖	⊕	⊕	⊕	⊕	⊕	⊕	⊕	○
Trichloroethene	⊕	⊕	⊕	○	⊕	⊕		⊕	⊕	○
Trichloromethane	⊕	⊖	⊕	⊖	⊕	⊕	⊖	⊕	⊕	⊖
Urea	⊕	⊕	⊕	⊕	⊕	⊕		⊕	⊕	⊕
Water	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕	⊕
Xylene	⊕	⊕	⊕	⊕	⊕	○		⊕	⊕	○

Data not guaranteed. ⊕ resistant, ⊖ not resistant, ○ limited resistance

MV = cellulose mixed esters, CA = cellulose acetate, RC = regenerated cellulose, PA = polyamide, PTFE = polytetrafluoroethylene (Teflon), PVDF = polyvinylidene difluoride, PES = polyethersulfone, PET = polyester, GF = glass fibre, PP = polypropylene (housing material)

Syringe filters CHROMAFIL®

CHROMAFIL® Xtra *MACHEREY-NAGEL*

labelled for method validation and cerification

Xtra: imprint for direct identification of membrane type, diameter and pore size
Xtra: low bleeding polypropylene housing
Xtra: colour-free plain polypropylene

CHROMAFIL® syringe filters with polyester (PET) membrane

hydrophilic multipurpose membrane *MACHEREY-NAGEL*

For polar as well as non-polar solvents. The HPLC filter, especially suited for mixtures of water and organic solvents; for TOC/DOC determination; not cytotoxic, does not inhibit the growth of microorganisms and higher cells.

Polyester filter with integrated glass fibre prefilter (GF/PET): recommended for solutions with a high load of particulate matter or for highly viscous solutions.

Type	Pore size µm	Membrane dia. mm	Housing colour top	Housing colour base	PK	Cat. No.
PET-20/25	0.20	25	labelled		100	**4.003 417**
PET-45/25	0.45	25	labelled		100	**6.232 548**
PET-120/25	1.20	25	labelled		100	**6.232 549**
PET-20/25	0.20	25	labelled		400	**4.003 418**
PET-45/25	0.45	25	labelled		400	**4.003 416**
PET-120/25	1.20	25	labelled		400	**6.233 172**
PET-20/15 MS*	0.20	15	yellow	orange	800	**4.003 397**
PET-45/15 MS*	0.45	15	colourless	orange	800	**4.003 398**
GF/PET-20/25	1.0/0.20	25	blue	orange	100	**9.049 079**
GF/PET-45/25	1.0/0.45	25	black	orange	100	**9.049 080**
GF/PET-20/25	1.0/0.20	25	blue	orange	400	**9.049 020**
GF/PET-45/25	1.0/0.45	25	black	orange	400	**9.049 021**

MS = minispike on filter exit
CHROMAFIL® Xtra: 4.003 417/6.232 548/6.232 549/4.003 418/4.003 416/6.233 172
BIG-BOX: 4.003 418/4.003 416/6.233 172/4.003 397/4.003 398/9.049 020/9.049 021
*also available as small pack with 100 pieces

1 CHROMAFIL® syringe filters with regenerated cellulose (RC) membrane

1

hydrophilic membrane with very low adsorption *MACHEREY-NAGEL*

for aqueous and organic/aqueous liquids i.e. polar and medium polar sample solutions. Binding capacity for proteins 84µg/25mm filter.

Type	Pore size µm	Membrane dia. mm	Housing colour top	Housing colour base	PK	Cat. No.
RC-20/25	0.20	25	labelled		100	**4.003 424**
RC-45/25	0.45	25	labelled		100	**4.003 426**
RC-20/25	0.20	25	labelled		400	**4.003 425**
RC-45/25	0.45	25	labelled		400	**6.233 891**
RC-20/15 MS	0.20	15	yellow	blue	100	**9.049 025**
RC-20/15 MS	0.20	15	yellow	blue	800	**4.003 399**
RC-45/15 MS	0.45	15	colourless	blue	100	**9.049 026**
RC-45/15 MS	0.45	15	colourless	blue	800	**4.003 400**

MS = minispike on filter exit
BIG-BOX: 4.003 425/6.233 891/4.003 399/4.003 400
CHROMAFIL® Xtra: 4.003 424/4.003 426/4.003 425/6.233 891

Syringe Filters please see page 663.

1

1 CHROMAFIL® PTFE (Polytetrafluorethylene)

hydrophobic membrane *MACHEREY-NAGEL*

for nonpolar liquids and gases very resistant towards all kinds of solvents as well as acids and bases flushing with alcohol, followed by water, makes the originally hydrophobic membrane more hydrophilic.

Type	Pore size µm	Membrane dia. mm	Housing colour top	Housing colour base	PK	Cat. No.
PTFE-20/25	0.20	25	labelled		100	**4.003 409**
PTFE-45/25	0.45	25	labelled		100	**9.049 059**
PTFE-20/25	0.20	25	labelled		400	**4.003 410**
PTFE-45/25	0.45	25	labelled		400	**9.049 060**
O-20/3	0.20	3	natural	natural	100	**9.049 053**
O-45/3	0.45	3	natural	natural	100	**9.049 054**
O-20/15 MS	0.20	15	yellow	natural	100	**9.049 055**
O-45/15 MS	0.45	15	natural	natural	100	**9.049 056**
O-20/15 MS	0.20	15	yellow	natural	800	**4.003 394**
O-45/15 MS	0.45	15	natural	natural	800	**4.003 395**

MS = minispike on filter exit
BIG-BOX: 4.003 410/9.049 060/4.003 394/4.003 395
CHROMAFIL® Xtra: 4.003 409/9.049 059/4.003 410/9.049 060

2

2 CHROMAFIL® MV (cellulose mixed membrane)

Hydrophilic membrane *MACHEREY-NAGEL*

- for aqueous or polar solutions
- CHROMAFIL® Xtra

Type	Pore size µm	Membrane dia. mm	PK	Cat. No.
MV-20/25	0.20	25	100	**4.003 407**
MV-45/25	0.45	25	100	**4.003 405**
MV-20/25	0.20	25	400	**4.003 408**
MV-45/25	0.45	25	400	**4.003 406**

BIG-BOX: 4.003 408/4.003 406

3

3 CHROMAFIL® CA (cellulose acetate)

hydrophilic membrane *MACHEREY-NAGEL*

for filtration of water-soluble oligomers and polymers, especially suited for biological macromolecules. Very high shape stability in aqueous solutions
extremely low binding capacity for proteins (21µg/ 25mm filter).

Available in a sterile package (S) for filtration under sterile conditions (each filter individually sealed).

Type	Pore size µm	Membrane dia. mm	Housing colour top	Housing colour base	PK	Cat. No.
CA-20/25	0.20	25	labelled		100	**4.003 419**
CA-45/25	0.45	25	labelled		100	**4.003 421**
CA-20/25	0.20	25	labelled		400	**4.003 420**
CA-45/25	0.45	25	labelled		400	**4.003 422**
CA-20/25 S*	0.20	25	yellow	red	50	**9.049 036**
CA-45/25 S*	0.45	25	natural	red	50	**9.049 037**

BIG-BOX: 4.003 420/4.003 422
*sterile pack
CHROMAFIL® Xtra: 4.003 419/4.003 421/4.003 420/4.003 422

4

4 CHROMAFIL® PA (Polyamide = Nylon)

rather hydrophilic membrane *MACHEREY-NAGEL*

for aqueous and organic/aqueous medium polar liquids

Type	Pore size µm	Membrane dia. mm	Housing colour top	Housing colour base	PK	Cat. No.
PA-20/25	0.20	25	labelled		100	**4.003 411**
PA-45/25	0.45	25	labelled		100	**6.232 389**
PA-20/25	0.20	25	labelled		400	**4.003 412**
PA-45/25	0.45	25	labelled		400	**6.234 011**
AO-20/3	0.20	3	light beige	light beige	100	**9.049 047**
AO-45/3	0.45	3	light beige	light beige	100	**9.049 048**

BIG-BOX: 4.003 412/6.234 011
CHROMAFIL® Xtra: 4.003 411/6.232 389/4.003 412/6.234 011

1 CHROMAFIL® Polyvinylidene difluoride (PVDF)

hydrophilic membrane *MACHEREY-NAGEL*

for polar and nonpolar solutions, water-soluble oligomers and polymers like proteins
binding capacity for proteins 82µg/25mm filter. The PVDF filter with integrated glass fibre prefilter (GF/P) is recommended for filtration of biological samples with high particle loads. This filter features a high binding capacity for proteins. Also suited for filtration of polar and non-polar solutions.

Type	Pore size µm	Membrane dia. mm	Housing colour top	Housing colour base	PK	Cat. No.
PVDF-20/25	0,20	25	labelled		100	**4.003 413**
PVDF-45/25	0,45	25	labelled		100	**9.049 063**
PVDF-20/25	0,20	25	labelled		400	**4.003 414**
PVDF-45/25	0,45	25	labelled		400	**4.003 415**
GF/P-45/25	1,0/0,45	25	black	white	400	**4.003 402**
GF/P-45/25	1,0/0,45	25	black	white	100	**4.003 401**

CHROMAFIL® Xtra: 4.003 413/9.049 063/4.003 414/4.003 415 BIG-BOX: 4.003 414/4.003 402/4.003 415

2 CHROMAFIL® Glass fibre (GF)

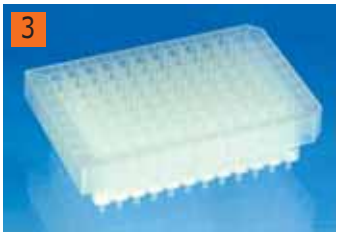
inert filter *MACHEREY-NAGEL*

nominal pore size 1µm, allows higher flow rates than smaller pore filters; for solutions
with high loads of particulate matter or for highly viscous solutions (e. g. soil samples, fermentation broths) as prefilters for other CHROMAFIL® filters, they prevent plugging of the membrane.

Type	Pore size µm	Membrane dia. mm	Housing colour top	Housing colour base	PK	Cat. No.
GF- 100/25	nom. 1.0	25	labelled		100	**6.232 362**
GF- 100/25	nom. 1.0	25	labelled		400	**4.003 423**
GF- 100/15 MS	nom. 1.0	15	blue	natural	100	**9.049 077**

MS = minispike on filter exit CHROMAFIL® Xtra: 6.232 362/4.003 423 BIG-BOX: 4.003 423

3 CHROMAFIL® MULTI 96 filter plates



96-well polypropylene plates for simultaneous filtration of 96 samples *MACHEREY-NAGEL*

advantages of this high-throughput system are:
- economical by saving time and solvent
- use of multi-channel pipettors facilitates liquid transfer steps
- readily adaptable to all common automated/robotic handling systems
- minimised dead volume (≤ 40µl)
- membrane materials correspond to the respective CHROMAFIL® syringe filters

Description	PK	Cat. No.
Filter plates with cellulose mixed ester filter elements (0.20 µm)	1	**4.003 976**
Filter plates with cellulose mixed ester filter elements (0.45 µm)	1	**4.003 977**
Filter plates with RC filter elements (regenerated cellulose, 0.2 µm)	1	**4.003 971**
Filter plates with RC filter elements (regenerated cellulose, 0.45 µm)	1	**6.227 345**
Filter plates with PTFE filter elements (0.2 µm)	1	**6.227 343**
Filter plates with PTFE filter elements (0.45 µm)	1	**6.227 344**
Filter plates with PTFE filter elements (1.0 µm)	1	**4.003 974**
Filter plates with PTFE filter elements (3.0 µm)	1	**4.003 975**
Filter plates with PE filter elements (20 µm)	1	**4.003 970**
Filter plates with PE filter elements (50 µm)	1	**4.003 973**
Filter plates with glass fibre filter elements (nominal 1 µm)	1	**6.227 346**
Filter plates with glass fibre filter elements (nominal 3 µm)	1	**4.003 972**
Vacuum manifold for monoblocks, with reservoir tank, vacuum gauge and control valve, required for filtration with 96-well filter plates	1	**4.003 962**