

Chromatography

SPE

CHROMABOND®
Vakuumkammern
Vacuum manifolds

Gebrauchsanleitung · User Manual

MACHEREY-NAGEL

www.mn-net.com



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CHROMABOND® vacuum manifolds

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Basics of Solid Phase Extraction (SPE)

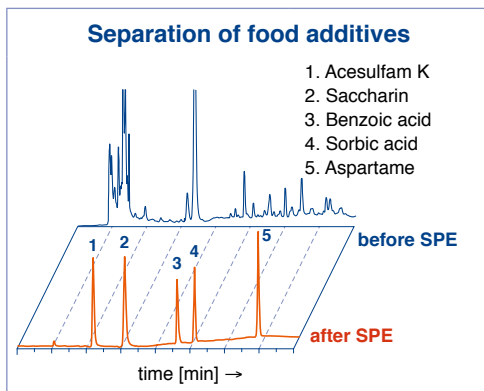
Solid phase extraction (SPE) is a powerful method for sample preparation and is used by most chromatographers today. For more than 20 years MACHEREY-NAGEL offers CHROMABOND® SPE cartridges based on silica and polymeric materials.

In modern analyses SPE fulfills three 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

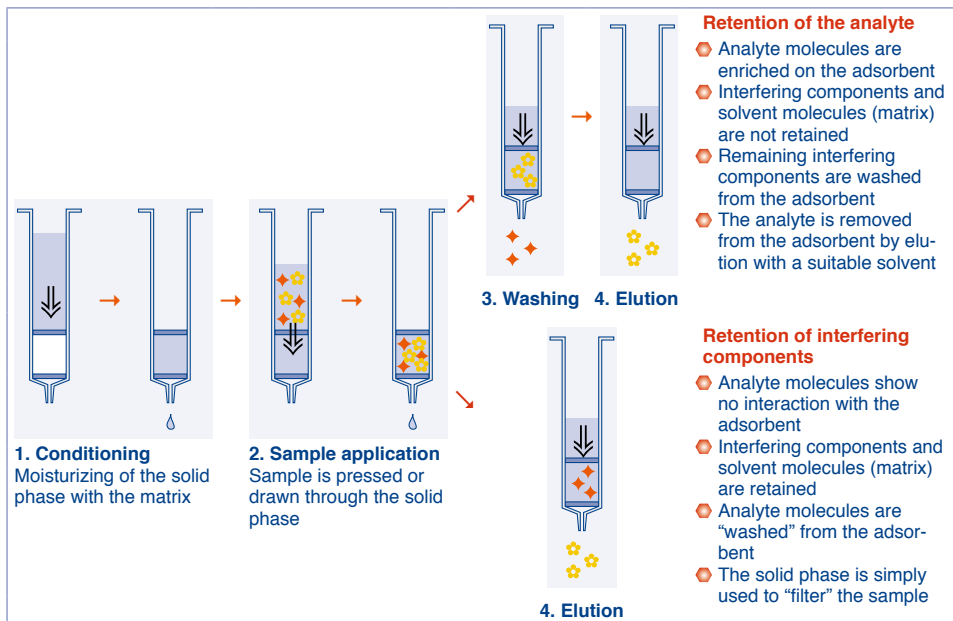
Advantages of SPE compared to classical liquid-liquid extraction:

- ⇒ 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
- ⇒ Optimization of extraction by variation or adjusting of the solid phase and chromatographic conditions



Principle of Solid Phase Extraction (SPE)

Since analytes can be either adsorbed on the SPE packing material or directly flow through while the interfering substances are retained, two general separation procedures are possible – both cases are shown in the figure.



CHROMABOND® vacuum manifolds

1. General information

CHROMABOND® vacuum manifolds for simultaneous preparation of 12, 16 or 24 samples

If several samples are to be treated simultaneously, we recommend our vacuum manifolds (Figure 1).

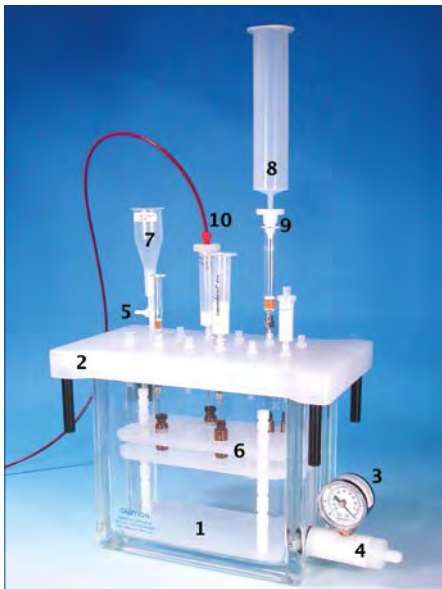


Figure 1: Vacuum manifold for 12 cartridges

MACHEREY-NAGEL supplies such manifolds in 3 different versions for up to 12, 16 or 24 CHROMABOND® columns or CHROMAFIX® cartridges, respectively. The manifolds consist of a rectangular glass cabinet (1) with vacuum gauge (3) and a polypropylene lid (2), which can hold the columns or cartridges. The replaceable valves/stop cocks (5) on the lid allow individual vacuum control for each solid phase extraction column, if required. The cabinet is fitted with a variable rack (6) with exchangeable partitions, which accept a wide variety of vessels like test tubes, measuring flasks, scintillation vials, autosampler vials, plastic vials and many more.

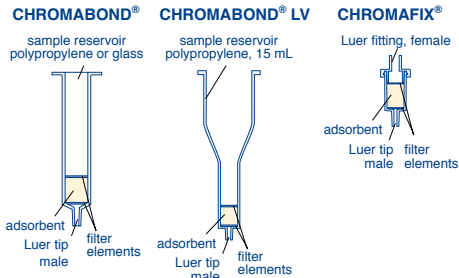


Figure 2: Hardware options for CHROMABOND® and CHROMAFIX® cartridges

CHROMABOND® vacuum manifolds

With the control valve (4) the vacuum in the chamber can be adjusted and read from the gauge. There are several possibilities for applying different sample volumes: small samples can be applied directly to the CHROMABOND® column. For medium size samples we have developed our CHROMABOND® LV columns (7) with 15 mL sample reservoir. Especially for this column type we offer the vacuum manifold with 16 positions, because with the manifold for 24 columns only every second position can be used. Alternatively, you may use the polypropylene sample reservoirs (30 or 70 mL) (8) from our program of SPE accessories, which can be fitted onto the CHROMABOND® column with the aid of an adaptor (9). Sample reservoirs fit directly onto the upper Luer fitting of the CHROMAFIX® cartridges.

For large sample volumes we recommend our CHROMABOND® tubing adaptors (10), which fit onto the CHROMABOND® columns. The other end of the tubing is placed into the sample, which, by applying vacuum, is continuously drawn into the CHROMABOND® column.

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 in figure 3. These special needles are fitted through the

lid; thus the sample only has contact with the inert needle and can flow directly into the receptacle.

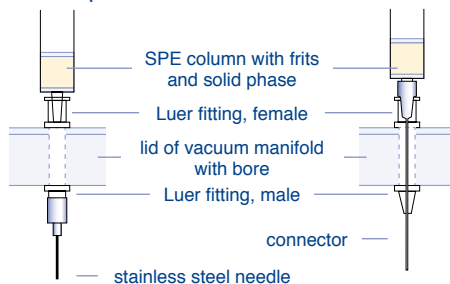


Figure 3 : Cross contamination free elution with stainless steel or PTFE connectors (right) compared to the standard configuration (left)

CHROMABOND® vacuum manifolds

If the eluate has to be evaporated, this can be performed with the so-called drying attachment (11, see Figure 4). 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.



Figure 4: Vacuum manifold with drying attachment

2. Operating instructions

Caution: Do not operate this manifold without first reading and fully understanding the operating instructions.

2.1 Contents

CHROMABOND® vacuum manifold

REF	Positions / Ports
730150	12
730360	16
730151	24

Contents

- Glass cabinet
- Lid with gasket
- Stainless steel needles
- Vacuum gauge with valve
- Plastic valves, luer
- Variable rack
- Waste tank (polypropylene (PP), only for REF 730150)

CHROMABOND® vacuum manifolds

2.2 Assembly

Manifold lid, gasket, needles and flow control valves

1. Attach the four black legs to the manifold lid.



2. Check to ensure that the white plastic lid gasket is properly seated in the lid.



3. Attach the stainless steel needles to the male luer connection fittings on the underside of the manifold lid.



4. Insert the flow control valves (stop cocks) into the female luer fittings on the lid.



5. Rotate the valves slightly to ensure positive seating.

Rack and adjustable shelves

1. The rack and shelf assembly consists of three attachment posts, and a support platform.



2. There are five shelves supplied with the 12 port manifolds, and three with the 16 and 24 port units.

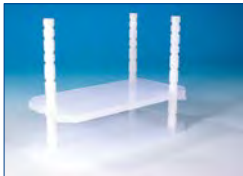


CHROMABOND® vacuum manifolds

3. Select and position one or more of the shelves supplied with the unit that best accommodates your collection vessel. Align the three small holes in the shelf with the three posts attached to the platform. Adjust the height of the shelf so that the delivery needles in the manifold lid will be inside the collection vessels.



4. The shelves are then secured on the rack posts by attaching the “C” shaped support clips to the slots in the support posts directly under the shelf that is to be supported.



5. The dimple shelves are used when utilizing test tubes as collection vessels.
6. If you are using collection vessels to collect sample preparation solvents, place the rack and shelves with appropriate collection vessels into the glass vacuum chamber. Attach the lid, and you are now ready to attach your SPE columns in the stop cocks, and proceed with sample preparation.



CHROMABOND® vacuum manifolds

Using the disposable solvent waste tank

1. For 12 port manifolds only, there is an optional disposable solvent waste collection tank (REF 730233, supplied with the manifold) available. To collect sample preparation solvents in the tank, place it in the glass vacuum chamber. Attach the lid and you are ready to place your SPE columns in the stopcocks and proceed with your sample preparation.



2. Just before you do your final elution, remove the lid, and take the waste tank containing the waste solvents out of the glass vacuum chamber. There are small handles at each end of the waste tank to facilitate its removal.
3. Place the rack and shelf set containing the sample collection vessels into the glass vacuum chamber.

4. Replace the lid, being careful to ensure the needles are inside each collection vessel. Proceed with your final elution.



5. Waste solvents should be properly discarded from the waste tank. The tank can be rinsed and re-used a number of times before discarding. Using the waste tank will save time, and greatly simplify manifold clean-up following a sample run, by eliminating the necessity for cleaning the vacuum chamber between sample runs.

CHROMABOND® vacuum manifolds

Vacuum connection and manifold operation

1. Install a liquid trap between the manifold vacuum chamber and the vacuum source.
2. Use vacuum tubing to connect the vacuum source to the filter or trap, and from the trap or filter to the manifold.
3. Apply vacuum and adjust vacuum at the manifold by using the knurled bleed valve ring adjacent to the vacuum gauge on the manifold.



Do not allow vacuum to go below 20 inches of mercury (68 kPa, 680 mbar, 510 Torr, 0.66 atm, 0.67 bar, 9.8 psi). Exceeding this vacuum will void the manifold warranty. For most applications a region of 25–27 inches of Hg (84–91 kPa) is satisfactory for proper flow through the CHROMABOND® SPE columns. The bleed valve ring also permits vacuum release for the purpose of changing collection vessels

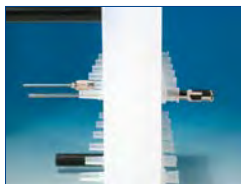
4. Proper operation of the manifold involves regulation of vacuum levels by using the vacuum bleed valve to achieve the desired flow rates. Individual flow control valves (stop cocks) at

each port must also be regulated to control the flow through each individual column.

5. The individual stop cocks should be in the closed position prior to removal of SPE columns or cartridges when under vacuum. Failure to completely bleed the vacuum from the chamber prior to venting the manifold may result in splashing or spillage of collected eluates.

Use of optional connectors

Disposable stainless steel or PTFE connectors (REF 730106, 730564) are designed to fit through the manifold lid, via the luer fittings. These needles deliver the eluate directly from the SPE extraction column or cartridge into the collection vessel in the vacuum chamber without contaminating the manifold lid. This combination is especially useful for critical sample analysis, such as environmental samples.



CHROMABOND® vacuum manifolds

Use of optional drying attachment

Drying attachments (REF 730187, 730188) for the 12 and 24 port vacuum manifolds will direct a flow of air or nitrogen into the collection vessels to dry eluates prior to further analysis.



Adapters also permit connection of the drying attachment directly to SPE columns to dry the tube prior to final elution.

2.3 Summary

Vacuum should not exceed 20 inches of mercury (68 kPa, 680 mbar, 510 Torr, 0.66 atm, 0.67 bar, 9.8 psi). Further information about the recommended working range can be found on page 38.

A vacuum bleed valve and gauge assembly installed in the glass chamber permits controlling and monitoring vacuum. Solvent resistant polypropylene luer fittings in the lid make the manifold compatible with any male luer terminated SPE device.

Stop cock valves on each port control liquid flow into the manifold chamber and col-

lection vessel. Optional PTFE needles are available when the sample requires an inert flow-path through the manifold lid into the collection vessel. Disposable waste containers are also available for the 12 port manifold to collect waste solvents prior to the final sample elution.

Sample filtration

Following column preparation and sample clean-up, and prior to final sample elution, the final eluate can be filtered prior to collection and injection. To filter the final eluate, remove the CHROMABOND® SPE column from the female luer of each stop-cock. A CHROMAFIL® 25 mm syringe filter is then inserted into the female luer of the stop cock, and the CHROMABOND® SPE column, cartridge or disk is placed into the female luer of the filter.



CHROMABOND® vacuum manifolds

Eluate collection vessels are placed in the rack in the glass vacuum chamber, and the system is now ready to deliver a filtered final eluate. It is important that filters must NOT be placed on the manifold until the final elution step. If filters are in place during column preparation or sample clean-up, they may air lock and not permit passage of any final eluate.

3. Storage and clean up notes

The glass cabinet of the CHROMABOND® vacuum manifold is made of standard laboratory glass, not from borosilicate glass. Therefore the mechanical and thermal properties can not be compared to usual laboratory glass products.

This glass cabinet must be cleaned by hand; the use of a Laboratory dish washer can cause damage or even destruction.

The lid of the CHROMABOND® vacuum manifold has a limited lifetime.

For normal use, we can give a one year guarantee. Depending on the solvents used, and contamination by acids or bases, the lifetime may be even shorter.

We recommend, not to let the lid get in direct contact to solvents and acids/bases. In order to achieve this, we offer our stainless steel or PTFE connectors (REF 730106, 730564). They prevent the direct contact of solvents with the lid, or the plastic fittings.

An additional advantage is that the lid is not contaminated, and a cleaning of the lid after use is therefore not necessary.

If cleaning of the lid with organic solvents is necessary, do not leave it in a solvent bath for more than 10 minutes! The lid may become twisted after this treatment!

Do not store or use the CHROMABOND® vacuum manifold in direct sun light (e.g. on a window-sill). Furthermore UV light or temperatures above 40 °C can cause an irreversible twisting of the lid.

4. Ordering information

Description	Pack of	REF
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		
for up to 12 columns or cartridges (including PP tank)	1	730150
for up to 16 LV columns	1	730360
for up to 24 columns or cartridges	1	730151
Glass cabinets without accessories		
for 12 columns (small)	1	730173
for 16 LV or 24 columns (large)	1	730174
Lids with gaskets		
for 12 columns (including Luer fittings and valves)	1	730175
for 16 LV columns (including Luer fittings and valves)	1	730365
for 24 columns (including Luer fittings and valves)	1	730176
Gaskets for lid, for 12 columns	2	730177
Gaskets for lid, for 16 or 24 columns respectively	2	730178
General accessories for vacuum manifolds		
Luer stoppers for vacuum manifold, blue	12	730194
Luer fittings for lid, female	12	730183.12
Luer fittings for lid, male	12	730184.12
Valves, plastic	12	730185
Stainless steel needles	12	730152
Polypropylene needles	12	730154
Waste tanks (PP) for vacuum manifold for up to 12 columns (not available for 16- or 24-position manifold)	2	730233
Vacuum gauge, complete with accessories	1	730179



female



male

CHROMABOND® vacuum manifolds

Description	Pack of	REF
Drying attachment and collecting racks for evaporation of eluates		
Drying attachment for 12 columns	1	730187
Drying attachment for 16 columns	1	730990
Drying attachment for 24 columns	1	730188
Collecting rack for 12 columns	1	730157
Collecting rack for 16 LV columns	1	730366
Collecting rack for 24 columns	1	730153
Products for protection from cross-contamination		
Valve, brass, tarnished	1	730189.1
Valves, as above	12	730189.12
Stainless steel connectors	12	730106
PTFE connectors	12	730564
Tubing adaptors for application of large sample volumes		
for 3 and 6 mL glass columns	4	730387
for 1, 3 and 6 mL polypropylene columns	4	730243
for 15, 45 and 70 mL polypropylene columns (PTFE tube length approx. 1 m)	4	730386
Adaptors for connection of e.g. Luer syringes		
Adaptor (PVDF) for glass columns (3 and 6 mL)	1	730104
Adaptors as above	10	730105
Adaptor (PP) for polypropylene columns (1, 3 and 6 mL)	1	730100
Adaptors as above	10	730101
Adaptor (PE) for polypropylene columns (15, 45 and 70 mL)	1	730350
Adaptors as above	10	730385
Adaptor (PE) for polypropylene columns (30 and 70 mL)	1	730566

CHROMABOND® vacuum manifolds

Description	Pack of	REF
Reservoir columns for application of medium–size samples		
Reservoir column 30 mL, polypropylene, with one adaptor for 1, 3, 6 mL CHROMABOND® polypropylene columns	1	730102
10 Reservoir columns 30 mL, polypropylene with one adaptor for 1, 3, 6 mL CHROMABOND® polypropylene columns	1 kit	730103
Reservoir column 70 mL, polypropylene, with one adaptor for 1, 3, 6 mL CHROMABOND® polypropylene columns	1	730381
10 Reservoir columns 70 mL, polypropylene with one adaptor for 1, 3, 6 mL CHROMABOND® polypropylene columns	1 kit	730382
Reservoir column 70 mL, polypropylene, with one adaptor for 15, 45, 70 mL CHROMABOND® polypropylene columns	1	730388
10 Reservoir columns 70 mL, polypropylene with one adaptor for 15, 45, 70 mL CHROMABOND® polypropylene columns	1 kit	730389

Polymer-based CHROMABOND® SPE phases

CHROMABOND® HR-Xpert...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

All **CHROMABOND® HR-Xpert** phases are based on high-purity and spherical polymeric resin and provide:

- ⇒ Excellent recovery rates and highest reproducibility
- ⇒ Reliable and cost-efficient analyses
- ⇒ Robust retention mechanism even for aggressive washing procedures
- ⇒ Low limits of detection also for critical matrices

CHROMABOND® polypropylene columns					REF
volume ads. weight pack of	1 mL		3 mL		6 mL
	30 mg 30	100 mg 30	60 mg 30	200 mg 30	500 mg 30
HR-X	730934	730935	730936	730931	730939
HR-XC	730969	730049	730956	730952	730955
HR-XA	730968	730727	730950	730951	730966
HR-XCW	730731	730733	730735	730739	730743
HR-XAW	730728	730729	730747	730748	730745

Details and standard protocols for the **CHROMABOND® HR-Xpert** concept can be found on our website www.mn-net.com/HR-Xpert.

Silica-based CHROMABOND® SPE phases

Traditional silica-based phases for reliable routine analysis

CHROMABOND® polypropylene columns					REF
volume	1 mL	3 mL		6 mL	
ads. weight	100 mg	200 mg	500 mg	500 mg	1000 mg
pack of	100	50	50	30	30
C18 ec	730011	730012	730013	730014	730015
NH ₂	730031	730413	730033	730180	730626
SiOH	730071	730214	730073	730070	730075
SA (SCX)	730076	730275	730077	730425	730212
SB (SAX)	730078	730322	730079	730426	730323

Other dimensions or phases (also for special applications) such as Florisil®, Alox (N, A, B), Drug, Drug II, CN/SiOH, NH₂/C18 und QuEChERS can be found on our website www.mn-net.com/SPE.



Umrechnungstabelle/pressure conversion chart

Umrechnungstabelle/pressure conversion chart

% Vacuum	mbar	Torr (mm Mercury)	psi (lb/in ²) abs	Inches Mercury Absolute	Inches Mercury Gauge	kPa abs
0.0	1013	760.0	14.7	29.92	0.00	101.4
1.3	1000	750.0	14.5	29.5	0.42	99.9
1.9	981	735.6	14.2	28.9	1.02	97.7
7.9	933	700.0	13.5	27.6	2.32	93.5
21.0	800	600.0	11.6	23.6	6.32	79.9
34.0	666	500.0	9.7	19.7	10.22	66.7
47.0	533	400.0	7.7	15.7	14.22	53.2
61.0	400	300.0	5.8	11.8	18.12	40.0
74.0	266	200.0	3.9	7.85	22.07	26.6
87.0	133	100.0	1.93	3.94	25.98	13.3
89.5	107	80.0	1.55	3.15	26.77	10.7
92.1	80	60.0	1.16	2.36	27.56	8.0
94.8	53	40.0	0.77	1.57	28.35	5.3
97.4	27	20.0	0.39	0.785	29.14	2.7
100.0	0	0.0	0.0	0.0	29.92	0.0

allowed vacuum
erlaubtes Vakuum

not allowed vacuum
nicht erlaubtes Vakuum

for CHROMABOND® vacuum manifolds
für CHROMABOND® Vakuumkammern

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- Kostenlose Applikationsdatenbank (in Englisch, mit mehr als 3.000 Chromatographie-Applikationen)

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MN Application database

HPLC - TLC - SPE

How to use the MN Application database

Search criteria:

Application type: SPE TLC HPLC GC

Results:

Title	Phase	Material
SPE Adsorbent and activation of the LC adsorbent material for the purification of pharmaceuticals (18 pharmaceuticals in benzene, chloroform and water)	HPLC	NUCLEOSORB
SPE Solid phase extraction of organic acids from pharmaceutical systems for chromatography	HPLC	NUCLEOSORB
SPE Rapid purification procedure from water	HPLC	NUCLEOSORB
SPE Desorbent recovery rates for Chromasorb® (PM)	HPLC	NUCLEOSORB
SPE Removal from water of the following six American and 8 EU for food and consumer goods (USA)	HPLC	NUCLEOSORB
SPE Removal from water of the following six German and 8 EU for food and consumer goods (Germany)	HPLC	NUCLEOSORB
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1.2.24.937



HPLC



GC



TLC



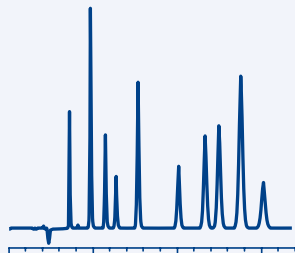
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