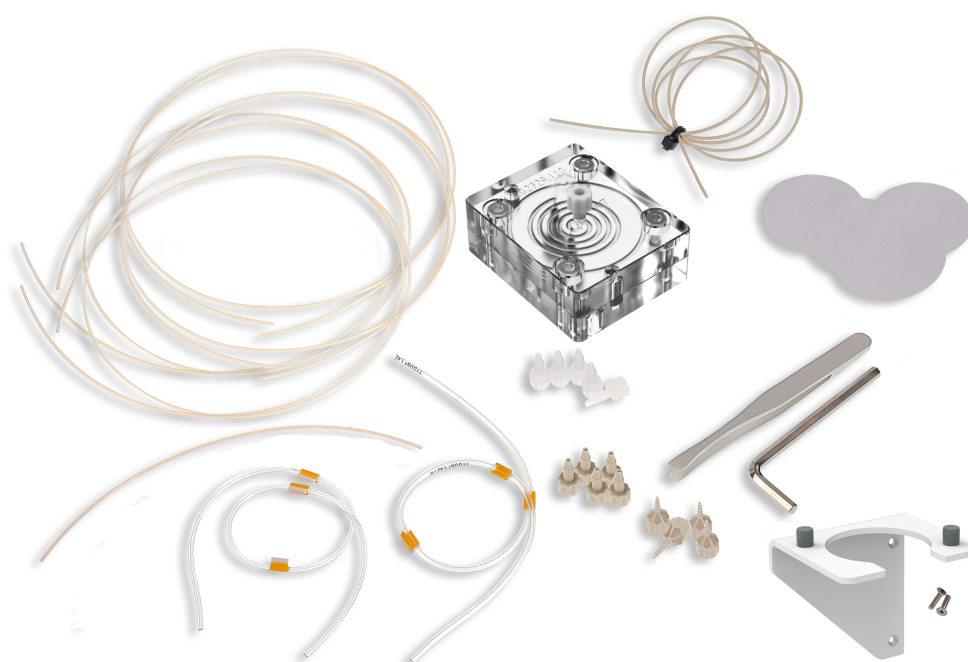


IC equipment



IC equipment: Inline Ultrafiltration 2 - pull mode

Manual

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IC equipment

IC equipment: Inline Ultrafiltration 2 - pull mode

6.05330.010

Manual

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This documentation has been prepared with great care. However, errors can never be entirely ruled out. Please send comments regarding possible errors to the address above.

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1 Introduction

1.1 Description of the IC equipment: Inline Ultrafiltration 2 - pull mode

The IC equipment: Inline Ultrafiltration 2 - pull mode (6.05330.010) contains all accessories required for Inline Ultrafiltration directly before injection.

The main component of the IC equipment: Inline Ultrafiltration 2 - pull mode is the ultrafiltration cell. This cell is suitable for the filtration of samples with high requirements regarding filtration effectiveness and sample throughput.

2 peristaltic pumps are needed in addition to the IC equipment: Inline Ultrafiltration 2 - pull mode for pumping the sample and the filtrate.

1.2 About the documentation

This manual describes the correct assembly and maintenance of the IC equipment: Inline Ultrafiltration 2 - pull mode, the installation of the capillary connections and the assembly of the holder to the sample changer.

The installation of the peristaltic pump is not described in this manual. The installation of the peristaltic pump is described in the respective manuals for the ion chromatograph or for the sample changer.









CAUTION

Please read through this documentation carefully before putting the IC equipment: Inline Ultrafiltration 2 - pull mode into operation. The documentation contains information and warnings which the user must follow in order to ensure safe operation of the IC equipment: Inline Ultrafiltration 2 - pull mode.

1.3 Symbols and conventions

The following symbols and formatting may appear in this documentation:

(5-12)	Cross-reference to figure legend
	The first number refers to the figure number, the second to the instrument part in the figure.
1	Instruction step
	Carry out these steps in the sequence shown.
Method	Dialog text, parameter in the software
File ► New	Menu or menu item
[Next]	Button or key
	WARNING
	This symbol draws attention to a possible life-threatening hazard or risk of injury.
	WARNING
	This symbol draws attention to a possible hazard due to electrical current.
	WARNING
	This symbol draws attention to a possible hazard due to heat or hot instrument parts.
	WARNING
	This symbol draws attention to a possible biological hazard.
	CAUTION
	This symbol draws attention to possible damage to instruments or instrument parts.
	NOTE
	This symbol highlights additional information and tips.

2 Mode of operation of ultrafiltration

One peristaltic pump delivers a continuous flow of sample solution at a high rate through the sample chamber of the ultrafiltration cell. The sample flows along the filtration membrane to the sample outlet and into the waste container.

At the same time, a second peristaltic pump generates a vacuum in the filtrate chamber of the ultrafiltration cell, thus aspirating the sample solution through the filtration membrane. The filtrate reaches the sample loop and is then injected.

20 to 25% of the original sample solution volume is analyzed as filtrate. The remainder flows into the waste container.

There are 2 options to connect the ultrafiltration cell. The push mode (*see chapter 2.1, page 3*) and the pull mode (*see chapter 2.2, page 3*).

2.1 Mode of operation of the push mode

The basic principle of the push mode is that the sample is delivered by pushing it.

In push mode, one peristaltic pump is installed between the sample changer and the sample inlet of the ultrafiltration cell. The other peristaltic pump is installed between the filtrate outlet and the injector. The sample flows through both peristaltic pump tubings before analysis. As the peristaltic pump tubings have a high volume, the required sample volume is higher in the push mode than in the pull mode (*see chapter 2.3, page 4*).

2.2 Mode of operation of the pull mode

The basic principle of the pull mode is that the sample is delivered by pulling it.

In pull mode, one peristaltic pump is installed between the sample outlet of the ultrafiltration cell and the waste container. The other peristaltic pump is installed between the injector and the waste container. Rinsing the additional volume of the peristaltic pump tubings is not necessary. Thus, the required sample volume is lower in the pull mode than in the push mode (*see chapter 2.3, page 4*).



2.3 Sample volume

The sample volume depends on the following factors:

- Volume of the sample needle
The lower the volume of the sample needle, the smaller the required sample volume.
- Length of the capillaries
The shorter the length of the capillaries, the smaller the required sample volume.
- Inner diameter of the capillaries
The smaller the inner diameter of the capillaries, the smaller the required sample volume.
- Volume of the peristaltic pump tubings
The lower the volume of the peristaltic pump tubings, the smaller the required sample volume.
- Volume of the ultrafiltration cell
The lower the volume of the ultrafiltration cell, the smaller the required sample volume.
- Volume of the sample loop
The lower the volume of the sample loop, the smaller the required sample volume.
- Ratio of the flow rate on the sample side to the flow rate on the filtration side
On the sample side, a high flow rate is required to pump a high sample volume through the sample chamber. On the filtration side, a lower flow rate is required as less filtrate needs to be pumped through the filtrate chamber than sample through the sample chamber.
The smaller the ratio of the flow rate on the sample side is to the flow rate on the filtration side, the smaller the required sample volume.

The pull mode and the push mode differ with regards to the length of the capillaries and the volume of the peristaltic pump tubings. The volume of the peristaltic pump tubings in particular carries weight.

Due to the additional volume of the peristaltic pump tubings, the required volume is considerably higher in the push mode than in the pull mode.

3 Overview

3.1 Parts of the IC equipment: Inline Ultrafiltration 2 - pull mode

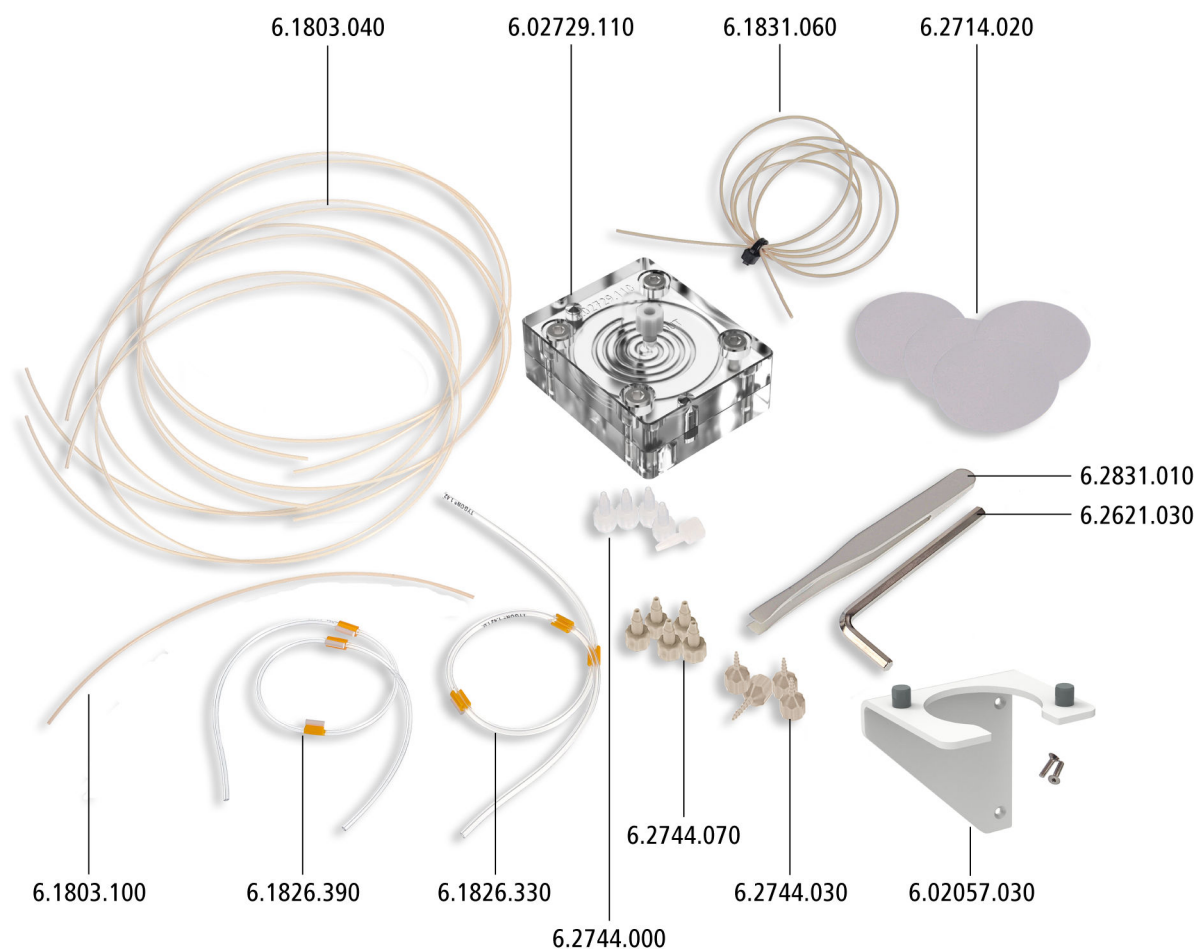


Figure 1 IC equipment: Inline Ultrafiltration 2 - pull mode – Parts

Table 1 Parts of the IC equipment: Inline Ultrafiltration 2 - pull mode

Article number	Article name	Number
6.1803.040	PTFE capillary, 0.5 mm ID / 1 m	4
6.02729.110	Ultrafiltration cell 2	1
6.1831.060	PEEK capillary, 0.5 mm ID / 1 m	1
6.2714.020	Filtration membrane 1	1
6.2831.010	Plastic tweezers	1

3.1 Parts of the IC equipment: Inline Ultrafiltration 2 - pull mode



Article number	Article name	Number
6.2621.030	Hex key 4 mm	1
6.02057.030	Filtration cell holder	1
6.2744.030	Coupling olive/UNF 10/32, 4x	1
6.2744.070	Pressure screw, short, 5x	1
6.2744.000	Pressure screw PVDF, 5x	1
6.1826.330	Pump tubing LFL (orange/white), 3 stoppers	1
6.1826.390	Pump tubing LFL (yellow/yellow), 3 stoppers	1
6.1803.100	PTFE capillary, 0.5 mm ID / 40 cm	1



3.3 Components of the ultrafiltration cell

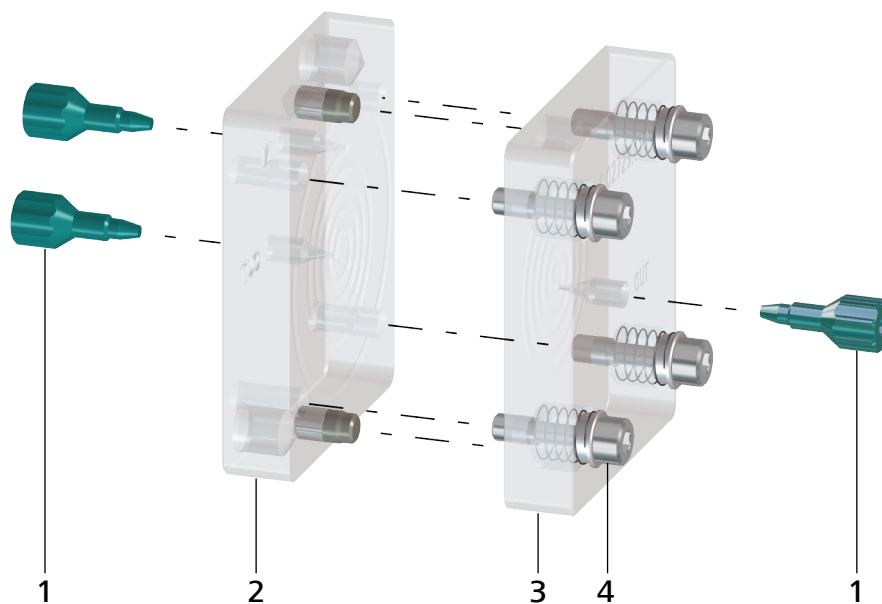


Figure 3 Ultrafiltration cell – Parts

1	Stopper	2	Sample chamber
3	Filtrate chamber	4	Screws for screwing together the sample chamber and the filtrate chamber

3.4 Connectors of the ultrafiltration cell

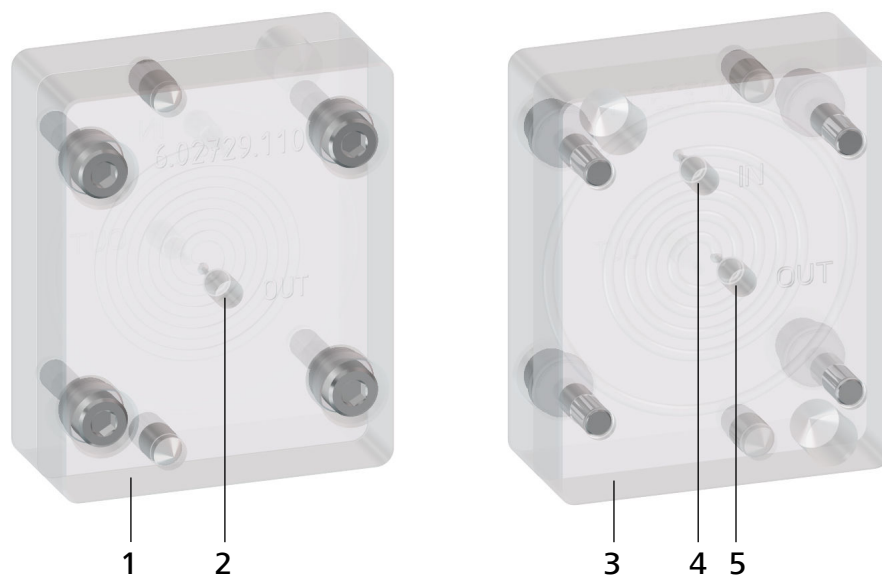


Figure 4 Ultrafiltration cell – Connectors

1	Filtrate chamber	2	Filtrate outlet labeled <i>OUT</i>
3	Sample chamber	4	Sample inlet labeled <i>IN</i>
5	Sample outlet labeled <i>OUT</i>		

4 Installation

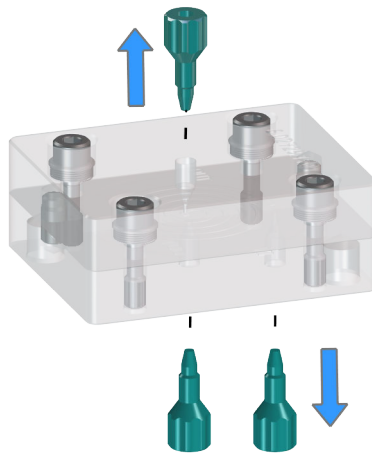
4.1 Preparing the ultrafiltration cell

Inserting the filtration membrane

Accessories

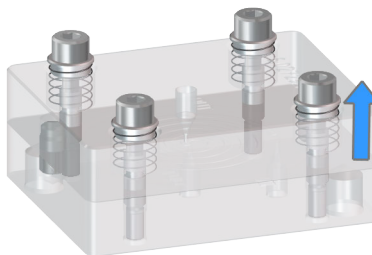
- Ultrafiltration cell 2 (6.02729.110)
- Filtration membrane 1 (6.2714.020)
- 4 mm hex key (6.2621.030)
- Plastic tweezers (6.2831.010)

1 Removing the stoppers



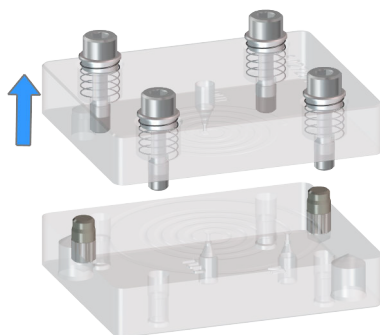
- Remove the 3 green stoppers by hand.
- Place the ultrafiltration cell on the table with the filtrate chamber facing upwards. The screws face upwards.

2 Removing the screws



- Loosen the screws with the hex key.
The screws cannot be removed from the filtrate chamber.

3 Disassembling the ultrafiltration cell



Remove the filtrate chamber of the ultrafiltration cell.

4 Cleaning the ultrafiltration cell

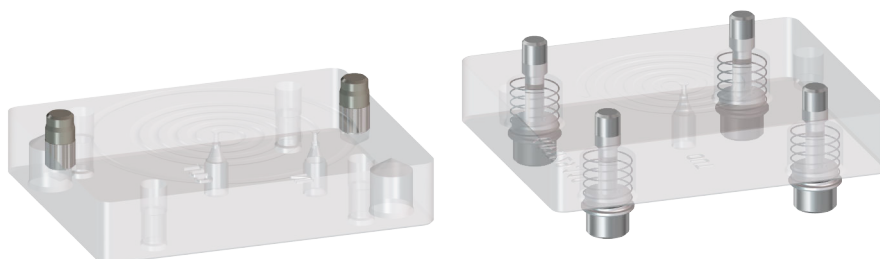


CAUTION

Damage to the ultrafiltration cell

Organic solvents (e.g. acetone) corrode and damage the ultrafiltration cell material (PMMA).

Use ultrapure water or a water-ethanol mixture (70:30) for cleaning the ultrafiltration cell.



- Rinse both chambers with ultrapure water.
- Dry both chambers with a lint-free cloth.

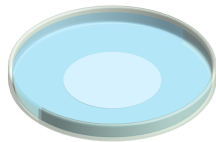
5 Wetting the filtration membrane



NOTICE

The package of the filtration membranes contains sheets of different thicknesses and colors:

- The thin white sheets are the filtration membranes. Only insert filtration membranes into the ultrafiltration cell.
- The firm white cardboard is a cover. The cover protects the filtration membranes. Do not insert the cover into the ultrafiltration cell.
- The thin light-blue sheets are separation sheets. The separation sheets are placed between 2 filtration membranes. Do not insert the separation sheets into the ultrafiltration cell.



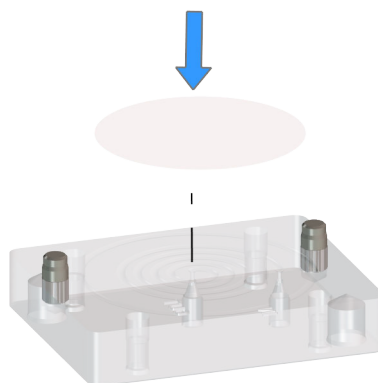
- Using the tweezers, take a filtration membrane out of the package.
- Place the filtration membrane in a petri dish filled with ultrapure water and allow to hydrate for approx. 2 minutes.

6 Inserting the filtration membrane



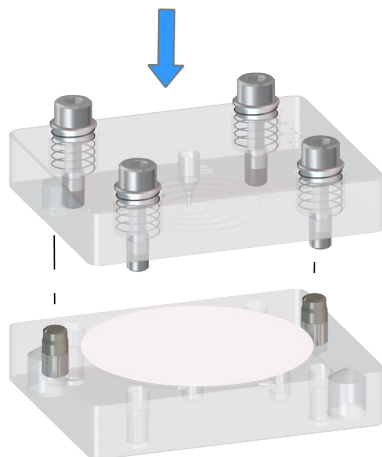
NOTICE

The filtration membrane must not dry out before it is inserted!



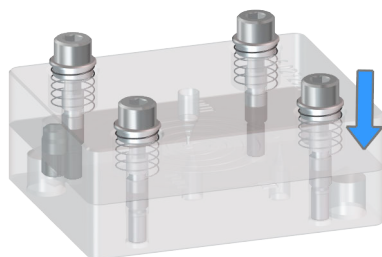
Using the tweezers, place the wet filtration membrane within the circular engraving on the sample chamber.

7 Assembling the ultrafiltration cell



Place the filtrate chamber on the sample chamber in such a way that the 2 guide bolts of the sample chamber fit into the bore holes of the filtrate chamber.

8 Screwing the ultrafiltration cell together



- Screw the screws entirely into the ultrafiltration cell using a hex key.
- Additionally, tighten each screw by a $\frac{1}{4}$ rotation.

- 9 If the ultrafiltration cell is not used immediately, seal the sample inlet, the sample outlet and the filtrate outlet with the 3 green stoppers. This allows for the filtration membrane to stay moist.

4.2 Connecting the ultrafiltration cell

This chapter describes how to establish the capillary connections in the filtration system. This chapter does not describe how the peristaltic pumps are connected. Information on the installation of the peristaltic pumps can be found in the *chapter "Installing the peristaltic pump" in the manual for the ion chromatograph or in the manual for the sample changer.*

The following figure shows a schematic overview of all capillary connections in the filtration system.

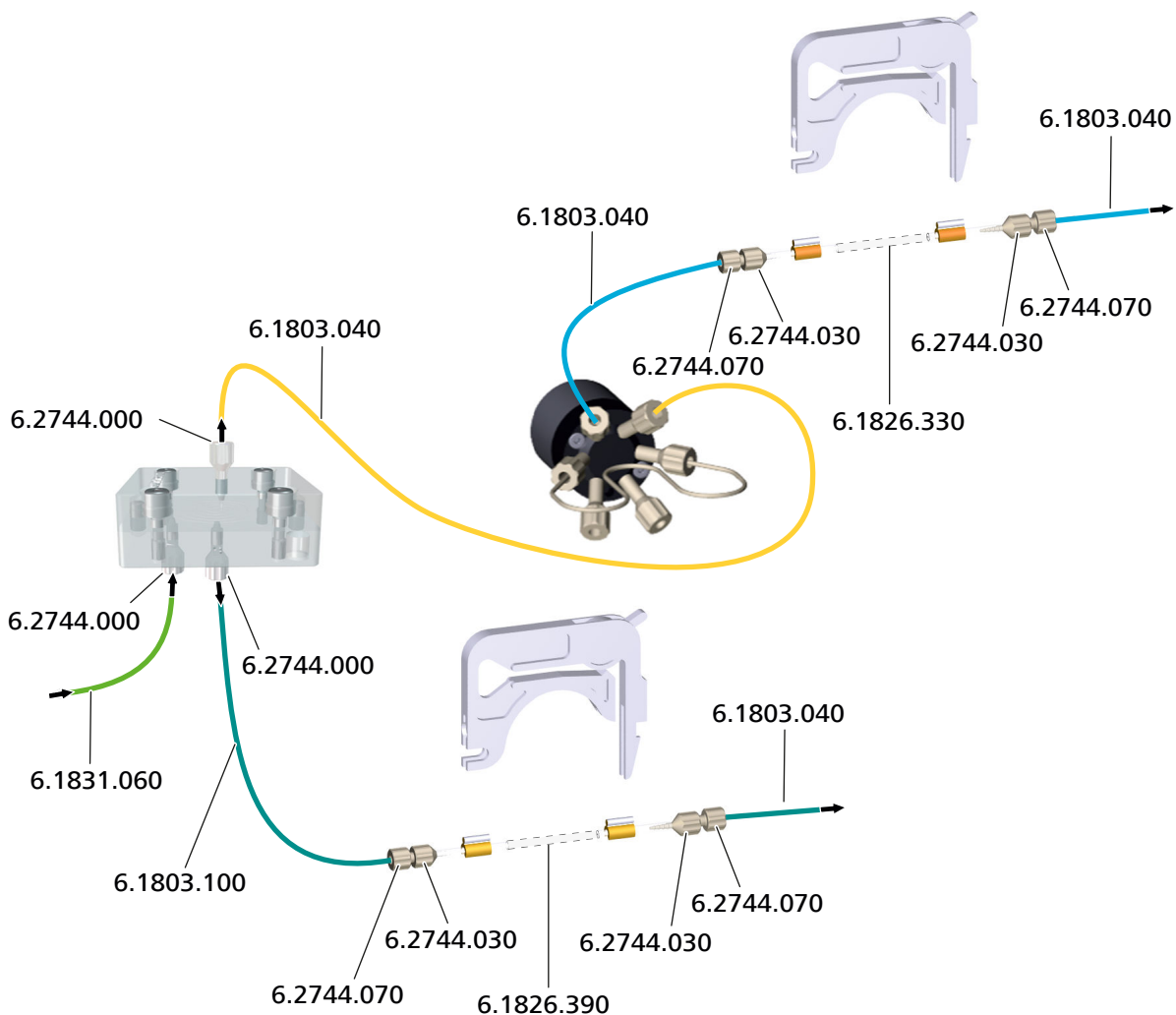


Figure 5 pull mode – Connecting the ultrafiltration cell



NOTICE

- In order to keep dead volume to a minimum, the capillaries of the IC equipment: Inline Ultrafiltration 2 - pull mode are as short as possible.
- To prevent the capillaries leading into the ion chromatograph from being pinched, always guide them through the capillary feed-throughs provided for this purpose (*see the manual for the ion chromatograph*).

Connecting the ultrafiltration cell

Accessories

- Ultrafiltration cell 2 (6.02729.110)
 - PEEK capillary, 0.5 mm ID / 1 m (6.1831.060)
 - PTFE capillary, 0.5 mm ID / 40 cm (6.1803.100)
 - 4 x PTFE capillary 0.5 mm ID / 1 m (6.1803.040)
 - Pump tubing LFL (orange/white), 3 stoppers (6.1826.330)
 - Pump tubing LFL (yellow/yellow), 3 stoppers (6.1826.390)
 - Pressure screw PVDF (6.2744.000)
 - Coupling olive/UNF 10/32 (6.2744.030)
 - Pressure screw, short (6.2744.070)
 - Pressure screw (6.2744.014)
- The pressure screw (6.2744.014) is not part of the IC equipment: Inline Ultrafiltration 2 - pull mode.



CAUTION

Damage to the ultrafiltration cell caused by using the wrong pressure screws

PEEK pressure screws are too hard. PEEK pressure screws can damage the ultrafiltration cell material.

- Use only transparent PVDF pressure screws (6.2744.000) to tighten capillaries to the ultrafiltration cell.

1 Preparing the pump tubing for the sample

Use the pump tubing with yellow stoppers (6.1826.390) for conveying the sample.

- Attach the coupling olive/UNF 10/32 (6.2744.030) to the inlet.
- Attach the coupling olive/UNF 10/32 (6.2744.030) to the outlet.



2 Preparing the pump tubing for the filtrate

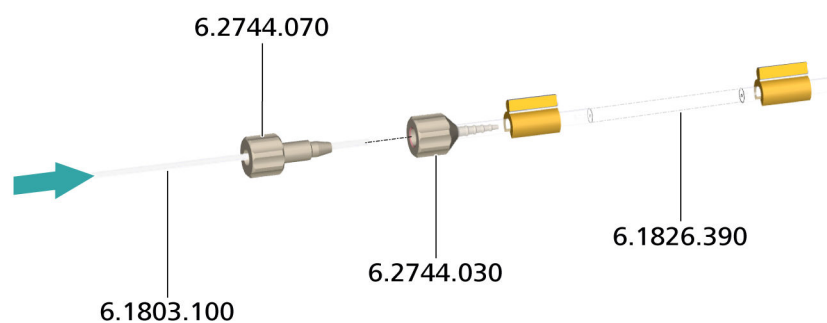
Use the pump tubing with orange/white stoppers (6.1826.330) for conveying the filtrate.

- Attach the coupling olive/UNF 10/32 (6.2744.030) to the inlet.
- Attach the coupling olive/UNF 10/32 (6.2744.030) to the outlet.

3 Connecting the sample changer to the sample chamber

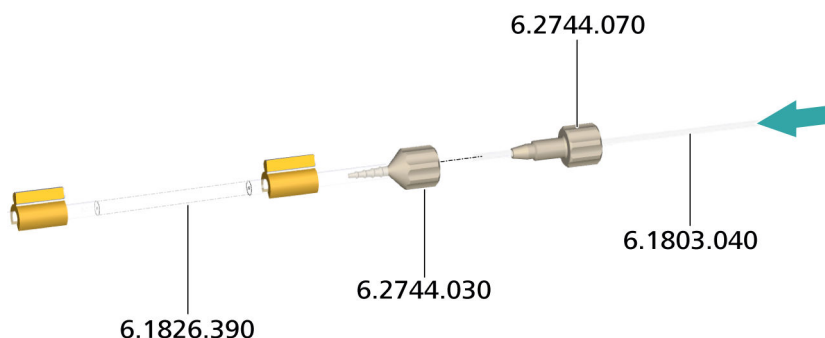
- Tighten one end of a PEEK capillary (6.1831.060) to the sample needle using a pressure screw (6.2744.014) (*see the manual for the sample changer*).
- Tighten the other end of the PEEK capillary (6.1831.060) to the inlet *IN* of the sample chamber using a PVDF pressure screw (6.2744.000).

4 Connecting the sample chamber to the peristaltic pump for the sample



- Tighten one end of the PTFE capillary (6.1803.100) to the outlet *OUT* of the sample chamber using a PVDF pressure screw (6.2744.000).
- Tighten the other end of the PTFE capillary (6.1803.100) to a coupling olive/UNF 10/32 (6.2744.030) on the inlet of a pump tubing with the yellow stoppers (6.1826.390) using a pressure screw (6.2744.070).

5 Connecting the peristaltic pump for the sample to the waste collector

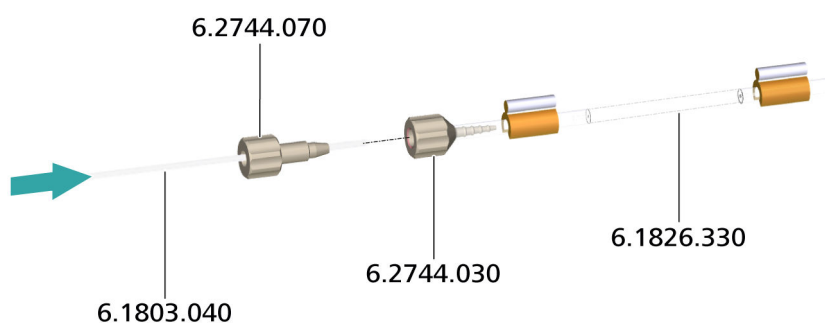


- Tighten one end of the PTFE capillary (6.1803.040) to a coupling olive/UNF 10/32 (6.2744.030) at the outlet of the pump tubing with the yellow stoppers (6.1826.390) using a pressure screw (6.2744.070).
- Tighten the other end of the PTFE capillary (6.1803.040) to the waste collector using a PVDF pressure screw (6.2744.000).

6 Connecting the filtrate chamber to the injector

- Tighten one end of the PTFE capillary (6.1803.040) to the outlet *OUT* of the filtrate chamber using a PVDF pressure screw (6.2744.000).
- Tighten the other end of the PTFE capillary (6.1803.040) to Port 1 of the injector using a pressure screw (6.2744.014) (*see the manual for the ion chromatograph*).

7 Connecting the injector to the peristaltic pump for the filtrate

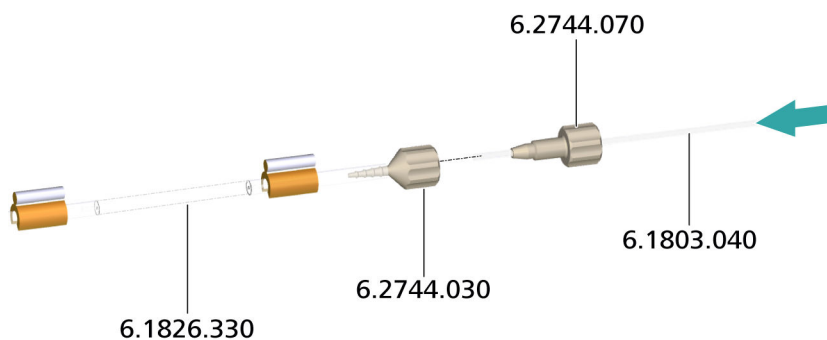


- Tighten one end of the PTFE capillary (6.1803.040) to Port 2 of the injector using a pressure screw (6.2744.014) (*see the manual for the ion chromatograph*).



- Tighten the other end of the PTFE capillary (6.1803.040) to the coupling olive/UNF 10/32 (6.2744.030) at the inlet of the pump tubing with the orange/white stoppers (6.1826.330) using a pressure screw (6.2744.070).

8 Connecting the peristaltic pump for the filtrate to the waste collector



- Tighten 1 PTFE capillary (6.1803.040) to a coupling olive/UNF 10/32 (6.2744.030) at the outlet of the pump tubing with the orange/white stoppers (6.1826.330) using a pressure screw (6.2744.070).
- Tighten the other end of the PTFE capillary (6.1803.040) to the waste collector using a PVDF pressure screw (6.2744.000).

4.3 Inserting the ultrafiltration cell

Fastening the ultrafiltration cell to the sample changer

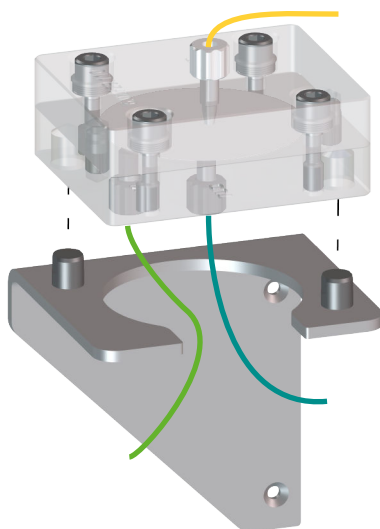
Accessories

- Filtration cell holder (6.02057.030)
- Ultrafiltration cell 2 (6.02729.110)

1 Attaching the holder

Tighten the filtration cell holder (6.02057.030) to the sample changer (*see manual for the sample changer*).

2 Inserting the ultrafiltration cell



Insert the ultrafiltration cell in such a way that the positioning pins of the filtration cell holder are located in the openings of the filtration cell provided for this purpose.

4.4 Purging the ultrafiltration cell

Every time a new filtration membrane is inserted, purge the filtration cell. To accomplish this, rinse all capillaries with e.g. ultrapure water. The entire filtration system must be completely connected prior to this procedure.

Rinsing the ultrafiltration cell

Accessories

- Sealing cap (6.2744.100)
The sealing cap (6.2744.100) is not part of the IC equipment: Inline Ultrafiltration 2 - pull mode.

1 Settings in the software

- Immerse the sample needle in the rinsing solution.
- Switch on the peristaltic pump.
- Rinse the filtration system with ultrapure water for approx. 5 min.

2 Monitoring the rinsing process

- Check whether equal amounts of solution are emerging from both feed lines to the waste container.
- Check whether all capillary connections are tight.
If liquid is escaping somewhere, then tighten the corresponding connection or redo the connection.



3 Removing air bubbles

- Check whether any air bubbles remain trapped in the ultrafiltration cell.

If air bubbles are trapped in the cell, proceed as follows:

- A capillary (6.1803.040) connects the peristaltic pump for the sample to the waste collector. Remove this capillary from the waste collector and seal it with a sealing cap (6.2744.100).
- If the air bubbles have not yet escaped, increase the rate of the peristaltic pump to 4.

5 Operation

5.1 Ultrafiltration time and sample volume

The ultrafiltration cell was tested with the IC equipment: Inline Ultrafiltration 2 - pull mode. The following values were determined for the ultrafiltration time and sample volume:

- Ultrafiltration time: 140 s
- Sample volume: 3.5 mL

These values are guidelines. The ultrafiltration time and the sample volume of your installation may deviate from these values. This is why the actual values for the ultrafiltration time and the sample volume must be determined for each installation.

5.2 Selecting the filtration membrane

Existing sample preparation procedures can be applied to ultrafiltration cell 2 (6.02729.110). If filtration membrane 1 (6.2714.020) is not used, a membrane with suitable pore size does not automatically yield the desired results even if the particle size is known.

Our investigations have shown that the retention capacity of conventional filtration membranes does not always correspond to their specified pore size. The table *Selection of the filtration membrane* shows the qualitative filtration action of filtration membranes with different nominal pore sizes. Aqueous solutions containing silica particles with particle sizes of 1.5 µm and 5 µm were used in the test.

Table 2 Selection of the filtration membrane

Test solutions: Silica particles in water	Pore size of the filtration membrane ¹	Effect
0.5%, 5 µm	0.15 µm	no permeation
0.5%, 5 µm	3 µm	no permeation
0.5%, 5 µm	8 µm	no permeation
0.5%, 5 µm	10 µm	permeation ²
0.5%, 5 µm	12 µm	no permeation
0.5%, 1.5 µm	0.15 µm	no permeation
0.5%, 1.5 µm	3 µm	permeation

¹ Nominal pore size according to manufacturer's statement.



² Except for this membrane, all membranes were from the same manufacturer.

Due to the lower filter thickness, the retention capacity of filtration membranes may be lower than that of filters with the same pore size but a higher filter thickness. This must be taken into account when selecting an appropriate filtration membrane.

6 Maintenance

6.1 Service life of the filtration membrane

One common problem in filtration is that solid substances in the sample are deposited on the filtration membrane, causing it to become blocked over time. This effect is reduced with the following design measures.

- The ultrafiltration cell has a symmetrical design.
- The ultrafiltration cell is placed horizontally in the sample changer.
- The sample flows through the sample chamber of the ultrafiltration cell. The filtrate is aspirated on the filtration side. In this way, solid substances adhere less to the membrane.

Monitor the filtration process and replace the filtration membrane if needed.

A declining recovery rate in standard solution analyses is an indicator for an imminent blockage of a filtration membrane. Prepare standard solutions ideally with the sample matrix to be analyzed.

If a large number of samples is analyzed, we, at Metrohm, recommend measuring check standards regularly. In the case of samples with high particle loads, measure check standards after every 5th to 10th sample. No general prediction regarding the number of filtration cycles can be made. Also, the change in the recovery rate may be different with more samples being analyzed. Whereas the recovery rate with one sample matrix may remain constant over many samples and then suddenly drop off severely, its decline may be slow and continuous with a different sample composition.

At which time a filtration membrane needs to be replaced depends on the sample matrix and the specifications of the analysis method applied. Experience has shown that minuscule particles and suspended substances in the sample matrix will lead to blockage of the filtration membrane faster than coarser particles will. Coarser particles are more readily propelled past the membrane in the flow of sample material.

The table *Service life of the filtration membrane* contains samples that were filtered with the ultrafiltration cell and a filtration membrane 1 (6.2714.020) with a pore size of 0.2 µm and analyzed on a Metrohm ion chromatograph.

The listed results were obtained with Inline Ultrafiltration (push mode). The results are to be seen as examples, not guidelines. Deviating values for the service life can be found depending on the actual sample matrix. In pull mode, a smaller sample amount is needed than in push mode. Accord-



ingly, the filtration membrane is less contaminated. Therefore, a significantly longer service life can be expected in pull mode.

The concentration of the following 7 anions was determined for each sample type: F^- , Cl^- , NO_2^- , Br^- , NO_3^- , HPO_4^{2-} , SO_4^{2-} .

Table 3 Service life of the filtration membrane

Sample designation	Number of samples per filter
Orange juice with fruit pulp	40
Surface water	500
Drinking water	1,000
Ground water	500
Waste water 1	1,000
Waste water 2	130
Waste water 3	40
Waste water 4	80
NaCl solution (1%)	5,000
Schöniger absorption solution	100
Acidic earth extracts	1,000
Aqueous earth extracts	200

6.2 Replacing the filtration membrane

The filtration membrane used has to be in perfect condition in order to ensure a consistent quality of the analysis results. Therefore, replace the filtration membrane in regular intervals (*see chapter 6.1, page 23*).

Replacing the filtration membrane

Accessories

- Filtration membrane 1 (6.2714.020)
- 4 mm hex key (6.2621.030)
- Plastic tweezers (6.2831.010)

1 Taking the ultrafiltration cell out of the system

- In the software, stop the system and wait until the pressure has been released.
- Remove all capillaries from the ultrafiltration cell.
- Take the ultrafiltration cell out of the holder.

2 Cleaning the ultrafiltration cell

Carry out the instruction steps 2 to 8 of the procedure *Inserting the filtration membrane on page 10*.

3 Connecting capillaries to the ultrafiltration cell

Carry out the instruction steps 3, 4 and 6 of the procedure *Connecting the ultrafiltration cell on page 15*.

4 Inserting the ultrafiltration cell into the holder

Place the ultrafiltration cell back in the holder (*see chapter 4.3, page 18*).

5 Purging the ultrafiltration cell

Purge the ultrafiltration cell (*see chapter 4.4, page 19*).



7 Technical specifications

7.1 Ultrafiltration cell 2 (6.02729.110)

<i>Material</i>	PMMA (poly(methyl methacrylate))
<i>Solvent compatibility</i>	Water or water-ethanol mixture (70:30) (no other organic solvents)
<i>Cell volume</i>	Sample chamber: 240 μ L Filtrate chamber: 120 μ L

7.2 Filtration membrane 1 (6.2714.020)

<i>Pore diameter</i>	0.2 μ m
<i>Membrane diameter</i>	47 mm
<i>Material</i>	Regenerated cellulose

8 Accessories

Up-to-date information on the scope of delivery and optional accessories for your product can be found on the Internet. You can download this information using the article number as follows:

Downloading the accessories list

- 1** Enter <https://www.metrohm.com/> into your Internet browser.
- 2** Enter the article number (e.g. **6.05330.010**) into the search field.
The search result is displayed.
- 3** Click on the product.
Detailed information regarding the product is shown on various tabs.
- 4** On the **Included parts** tab, click on **Download the PDF**.
The PDF file with the accessories data is created.



NOTICE

Once you have received your new product, we recommend downloading the accessories list from the Internet, printing it out and keeping it together with the manual for reference purposes.



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