

Azura

SMB systems Instructions



Document no. V6675



Note: For your own safety, read the instructions and observe the warnings and safety information on the device and in the instructions. Keep the instructions for future reference.

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1. Product information

1.1 Intended use



Note: Only use the system for applications that fall within the range of the intended use. Otherwise, the protective and safety equipment of the system could fail.

Application	Separation and extraction
Pharmaceutical chemistry	Chiral compound (cis-trans phytol, steroids, peptides, antibiotics, etc.)
Food chemistry	Fatty acids, carbohydrate mixtures (sucrose/molasses, fructose/glucose, etc.)
Biochemistry	Phenylalanine, fermentation/cell culture products (citric acid, sugars, antibodies, enzymes, etc.)
Petrochemistry	C8-Hydrocarbon (xylene/toluene, etc.)

1.2 AZURA® SMB system variants

1.2.1 AZURA® SMB Lab

This SMB system is optimized for separation tasks on a scale of several hundred grams. The standard configuration consists of four AZURA® assistants ASM 2.1L with seven multi-position valves and four AZURA® pumps P 4.1S as well as our user-friendly software PurityChrom® MCC including required IT hardware.

Depending on the special requirements of every separation the SMB system can be freely configured via valve switch (e.g. closed-loop, open-loop) and is upgradable with detectors and flow meters.

Individual configuration is available on request.

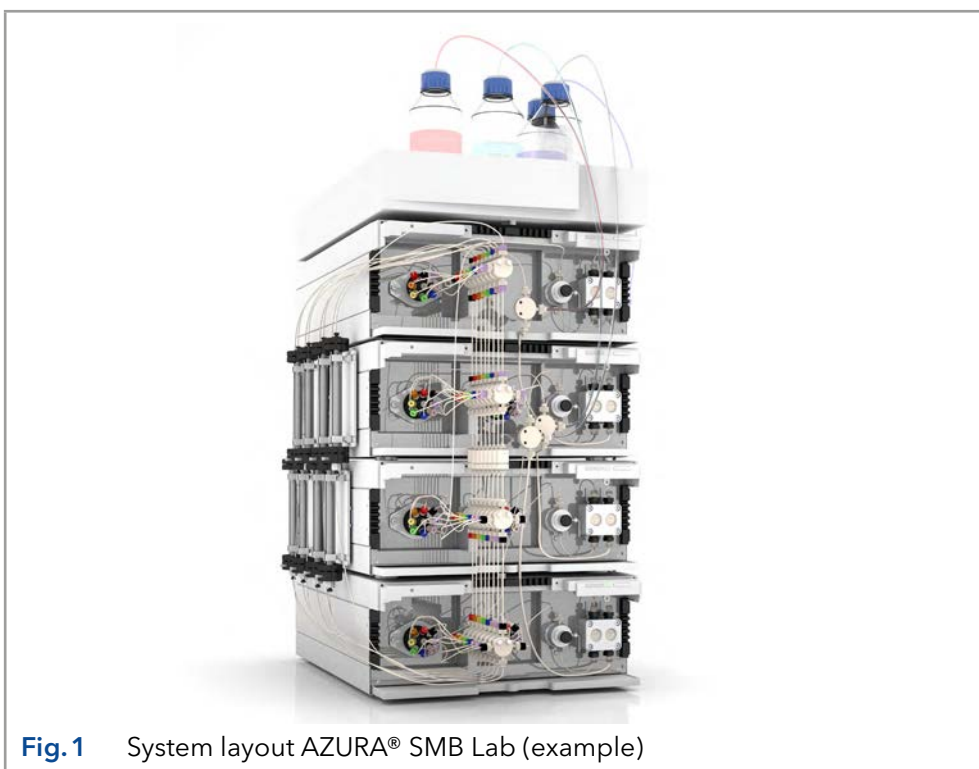


Fig. 1 System layout AZURA® SMB Lab (example)

1.2.2 AZURA® SMB Pilot

The AZURA® SMB Pilot is designed for the separation of binary mixtures on a hundred gram to kilogram scale and is typically used with columns up to 50 or 100 mm ID. Its special emphasis is put on the continuous operation mode and highest productivity.

The SMB standard configuration consists of four AZURA® pumps P 2.1L and seven 8-port multiposition valves integrated into four AZURA® assistants ASM 2.1L. Our user-friendly software PurityChrom® MCC and the required IT hardware are also included.

We offer several variations of the standard system configuration.

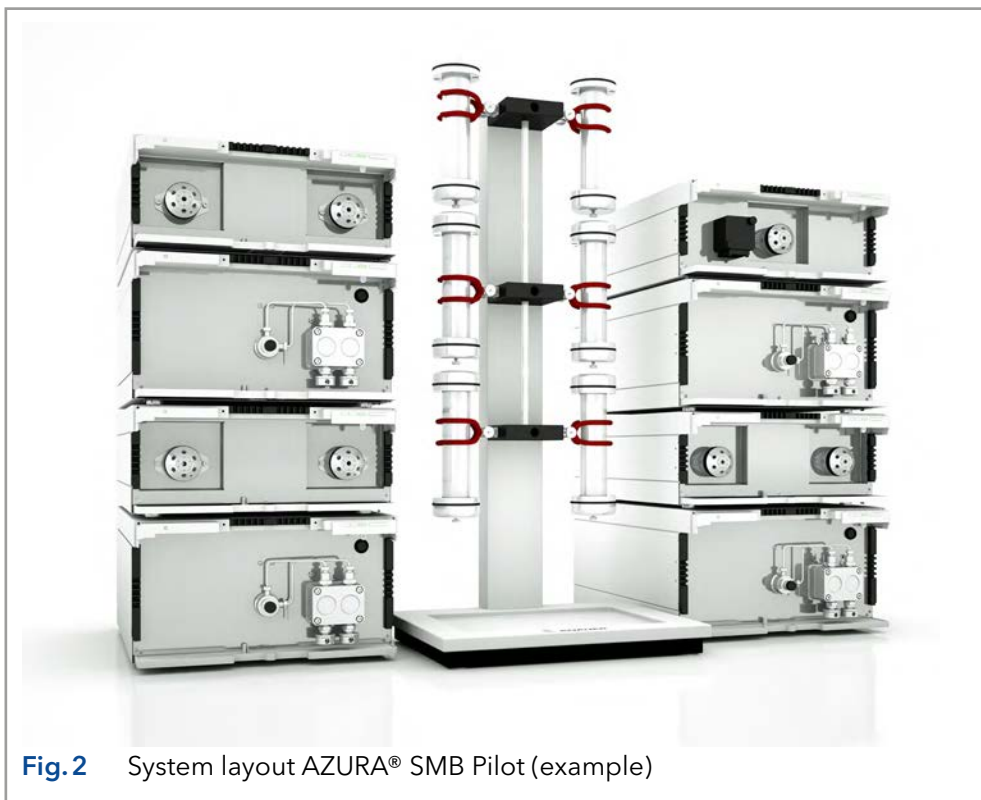


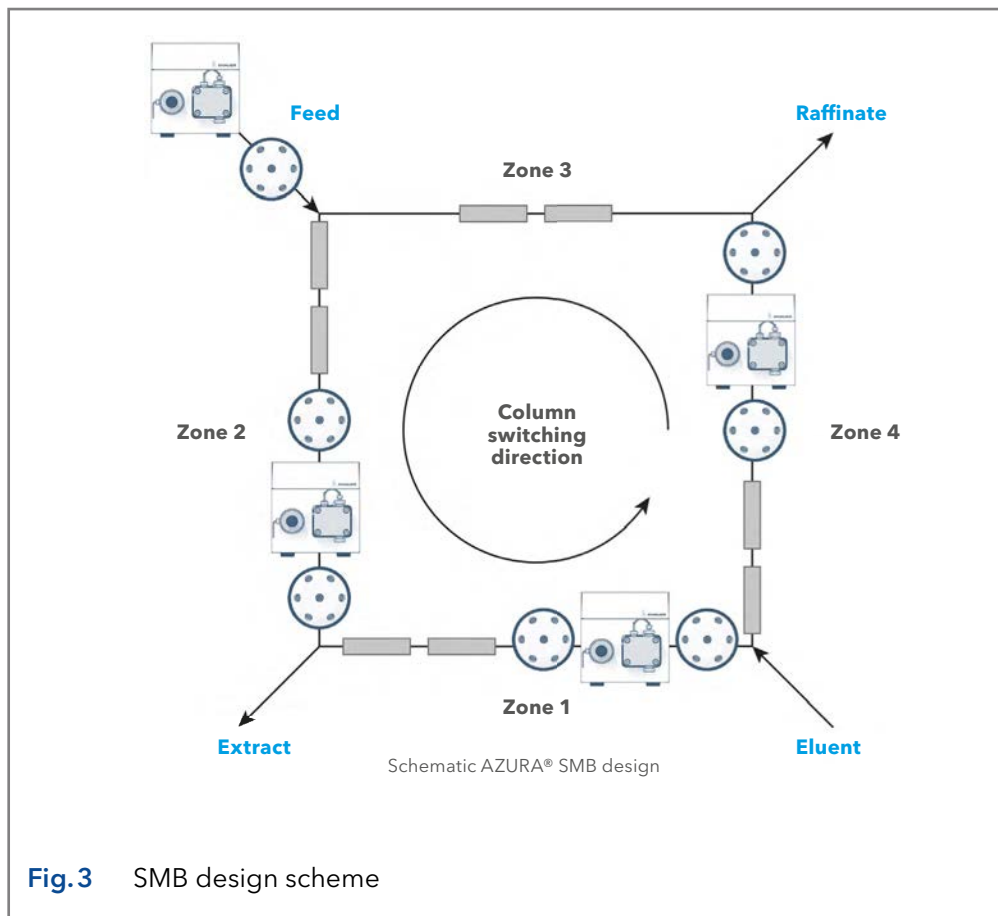
Fig.2 System layout AZURA® SMB Pilot (example)

1.3 System configuration

The standard AZURA® SMB systems consists of four pumps and seven multi-position valves.

The devices are arranged as follows:

- Three pumps (Extract, Raffinate, Eluent) are placed inside the SMB cycle.
- The feed pump is placed outside the SMB cycle.
- Four valves are placed at the pump outlets.
- Three valves are placed at the pump inlets. The feed pump inlet is not connected to a multiposition valve.



1.3.1 AZURA® SMB Lab

Number of columns	Max. flow rate	Max. pressure	Description	Article no.
8	50 ml/min	130 bar	Stainless steel	A29000
			Biocompatible (PAEK, ceramic)	A29001

1.3.2 AZURA® SMB Pilot

Number of columns	Max. flow rate	Max. pressure	Description	Article no.
8	500 ml/min	100 bar	Stainless steel	A29501

2. Simulated Moving Bed principle

2.1 Introduction

Due to the fast development of new compounds and the more precise knowledge about them, there is an increasing demand for purified products. In most cases, chemical syntheses produce a lot of side components. Therefore, different separation steps have to be involved into production processes.

The most established and theoretically described thermal separation processes (e.g. distillation, extraction, crystallization etc.) can not be applied to all separation tasks. Among new separation methods, the adsorption chromatography is a very effective but expensive process. The principle of adsorption chromatography was discovered in the last century. The separation is caused by different affinities of the compounds (dissolved in a liquid) to a useful solid phase.

Applications of adsorption chromatography were limited to analytical scale for a long time. In the analytical scale, the mixtures are very diluted and the distribution of a component between liquid and the solid phase can be described by linear relations.

In the last few decades, adsorption chromatography is more and more applied to the preparative process scale, where higher concentrations are used and the liquid-solid distribution of a component becomes non-linear and concentrationdependent. The peak profile and the retention times are changing, so that modeling is more complicated.

The most established chromatographic process mode is elution chromatography. Due to the theoretical understanding of this mode, it is possible to use it in a very large scale. The regime of elution chromatography is a discontinuous one, i.e. only discrete amounts of a mixture can be separated. For higher amounts, a continuous mode should be favored. For a binary (or pseudo-binary) mixture, the SMB process (Simulated Moving Bed) with conventional chromatographic columns is successful.

Only the general principles of SMB will be explained in these instructions.

2.2 Elution chromatography

Among the established industrial scale separation processes preparative adsorption chromatography is more and more used. Some separation task (e.g. the separation of enantiomers) can be solved only by chromatography. The basic principle of elution chromatography is shown in Fig. 4.

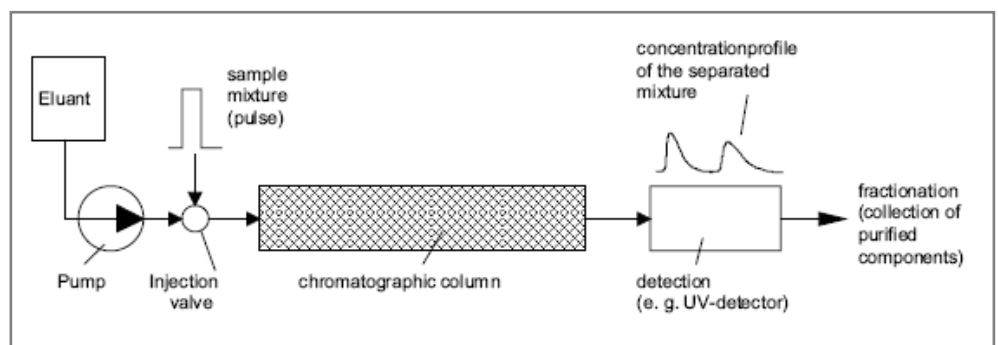


Fig. 4 Principle of elution chromatography

The chromatographic column packed with a solid phase (adsorbent) will be permanently penetrated by a fluid phase (eluent) with a well defined

interstitial velocity. At the time t_0 a well defined amount of the sample mixture is injected into the column. Due to different interaction forces of the mixture components and the adsorbent (different migration velocities), a separation of the mixture into the pure components will take place. At the column outlet the purified components can be detected and collected. This is the general principle of chromatography.

In preparative chromatography, the column is highly overloaded and is not sufficient to completely separate the mixture into the pure components. The peaks are overlapping. This is shown in Fig. 5. An unspecific detector would show only the sum of both components, while the single components are detectable in combination with a specific detector.

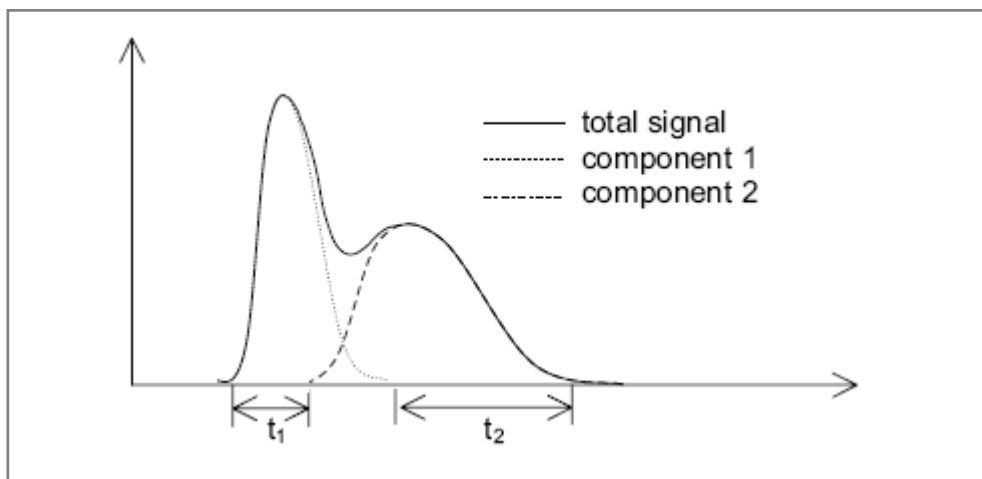


Fig. 5 Overlapping of peaks in preparative-scale chromatography

The reason for overloading the column is, that there are time sections (t_1 , t_2), where the components elute in the required purity with a high concentration. Because of the higher concentrations smaller liquid volumes are necessary to separate equal amounts of the sample, such that the separation of the value added components from the eluent (e.g. in vacuum evaporators) is much simpler and cheaper.

The overlapping part of the concentration profile shown in Fig. 5 with a lower purity than required can be recycled to the column inlet.

In elution chromatography pulses are injected into the column, which are separated in time (left side of Fig. 6).

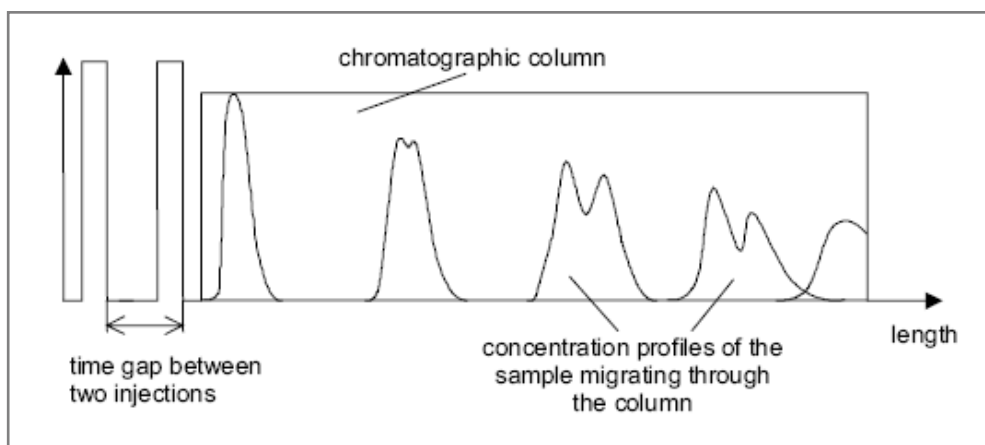


Fig. 6 Process mode of elution chromatography

While migrating through the column, these pulses are broadened, due to axial dispersion and different migration velocities and they are separated

into the pure components due to different interaction forces to the adsorbent.

The time between two pulses i and $i+1$ have to be chosen in such a case, that the slowest component of pulse i elutes before the fastest component of pulse $i+1$ or just before they overlap too much, where pulse i is injected before pulse $i+1$ (Fig. 6).

If you would separate the column into small sections, there are parts with two or more components within. Here and only here, the separation process takes place. In other sections there is only pure eluent and the separation capacity of the solid is not used. At the transients between these and the sections containing the components dilution and mixing are taking place. If two peaks are overlapping, because of different migration velocities, there is also mixing.

In the last described sections no separation takes place. These parts causes a lower use of the adsorbent capacity, but can not be avoided in elution chromatography. This is because of the discontinuous or batch regime of the process, i.e. only discrete amounts of the mixture can be injected. Increasing the scale of the column, increases the total amount, but it does not change the discontinuous character, connected with the lower use of the adsorbent capacity and dilution effects.

To separate larger amounts with smaller columns, other process modes than elution have to be used either a true counter-current process or a simulated one.

2.3 True counter-current process

By contacting an up-flowing liquid with a down-flowing solid in a separation column, a true counter-current chromatographic process is realized, called True Moving Bed (TMB). The TMB was first commercialized in the hypersorption process of the Union Oil Company of California in the early 1950th.

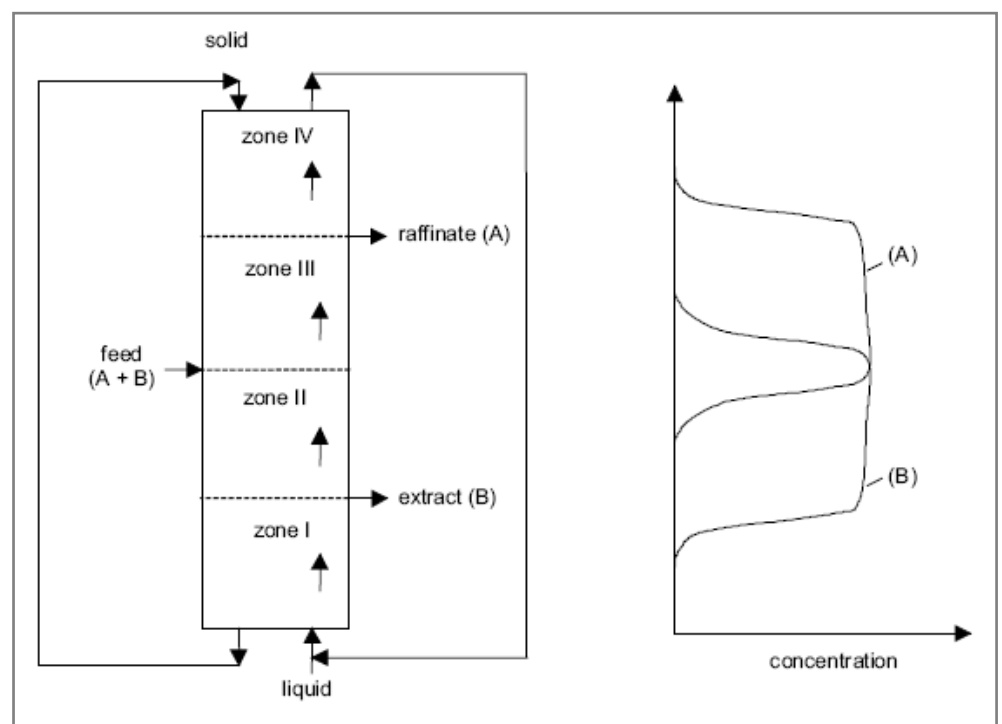


Fig. 7 Principle of a True Moving Bed (left) and typical concentration profile (right)

Unloaded solid (adsorbent) is continuously filled into the upper end of the column, while liquid (eluent) is pumped into the lower one. The binary (or pseudo-binary) feed-mixture is continuously injected in the middle of the column. To separate the components of the mixture, they have to be differently adsorbed on the solid.

The two feed components are called A and B, where A is the less and B the more adsorbed component, respectively. If there is a suitable ratio between the volumetric flow rates of the solid and the liquid phase, component B is adsorbed on the solid and is flowing downwards, while component A is displaced from the solid and is transported upward by the liquid. Pure component B can be withdrawn in the lower and pure A in the upper part of the column. The TMB process is able to separate binary or pseudo-binary mixtures into the two parts, where one part contains the less adsorbed compound, while the more adsorbed compound is in the other one. To achieve more than two fractions, difficult changes are necessary.

The separation column can be divided into four zones or sections shown in Fig. 7. These zones are fulfilling different functions.

Injecting a well defined volumetric flow rate with a well defined concentration of feed into an initially unloaded column at time t_0 , all species are transported into zone III. Following the laws of adsorption, the components of the mixture will be distributed between the liquid and the solid phase. The more adsorbed component will be enriched in the solid phase.

Since a high purity of component A is required in the raffinate stream, component B has to be adsorbed in section III in the volume between feed inlet and raffinate outlet. Therefore, the function of zone III is to adsorb component B or to separate it from A, respectively. The inverse problem exists in section II, where component A has to be desorbed from the solid phase and to be pushed back into zone III. Section II has the function to separate component A from B.

Zone I has to clean the solid. All components flowing into this section have to be desorbed, so that only the unloaded solid reaches the lower end of the column and is recycled to the upper end. In zone IV, vice versa, the liquid has to be cleaned. Recycling the pure liquid to the lower end of the column, decreases the desorbent or eluent consumption. Condensed, there are two separation sections (zone II and III) and two cleaning sections (zone I and IV).

If you realize a suitable ratio of the volumetric flow rates of the liquid and the solid phase in each zone, a concentration profile like that sketched on the right side in Fig. 7 is created. The raffinate stream contains nearly no B and the extract stream nearly no A. The most important parameters for properly designing a TMB unit are the volumetric flow rates of the liquid and the solid phase.

The true counter-current chromatographic process has the main drawback that the solid transport is difficult to realize due to back-mixing of the adsorbent particles. Another important point is, that only small flow rates are possible, since otherwise the solid would be pushed back by the liquid. So the separation performance of the process is decreased.

To avoid these problems, different attempts have been made, however they shall not be explained in detail here. The probably best possibility to avoid the TMB drawbacks is explained now.

2.4 Simulated counter-current process

The simulated counter-current process (Simulated Moving Bed - SMB) was developed in the early 60th by the Universal Oil Products Company. It was mainly applied to industrial-scale separations, like the xylene separation or the fructose-glucose separation. The process scheme is shown in Fig. 8.

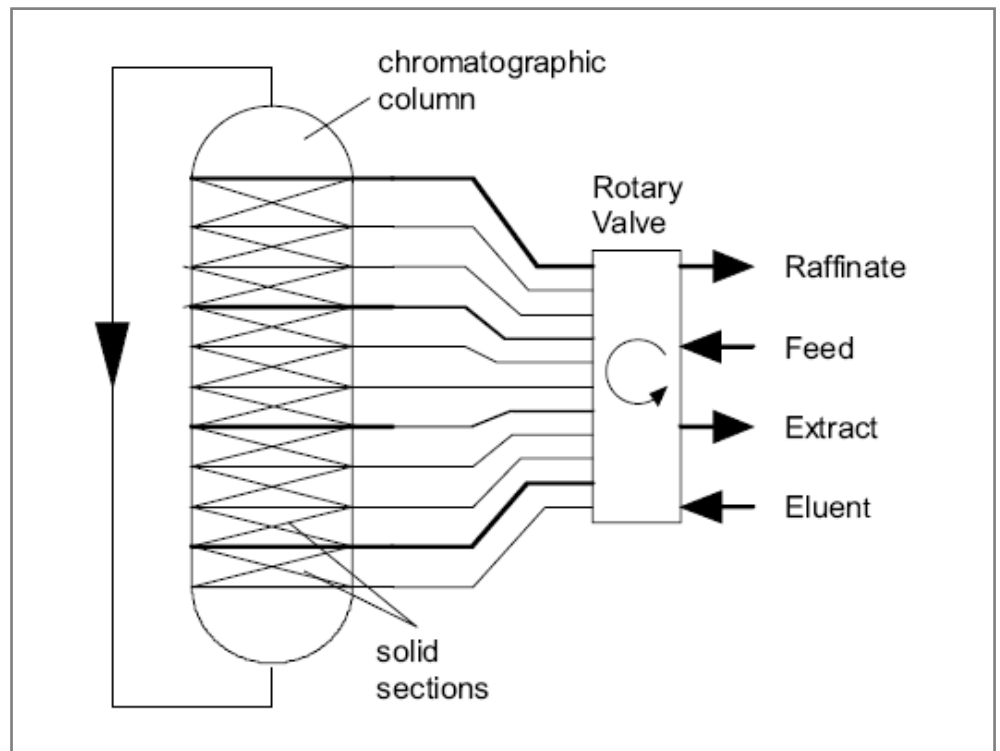


Fig. 8 Principle of a simulated counter-current process according to UOP

There is a strong analogy between the SMB and the TMB process. Under the use of a suitable system of adsorbent and eluent, a feed stream is separated into two withdrawal streams containing the pure components of a binary or pseudo-binary mixture.

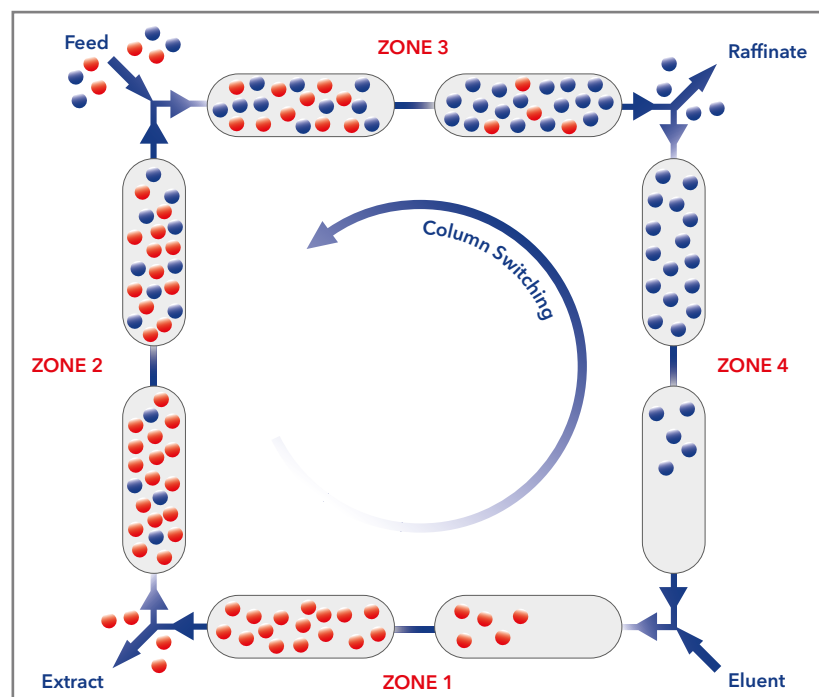


Fig. 9 SMB process scheme

For SMB applications the large column in Fig. 8 can also be substituted by a number of chromatographic columns. This is sketched for 8 columns in Fig. 9. Like in the TMB process there are feed, eluent, extract and raffinate inlets and outlets, dividing the circle of columns into four sections. In Fig. 9 the columns are distributed symmetrically, i.e. there are equal numbers of columns in each section.

Special valves allow the liquid to flow in only one direction. The inlets and outlets are arranged in a pre-defined manner and are switched in the same direction as the fluid flows.

The switching time T^{Shift} can be achieved from the volumetric flow rate of the solid and the solid volume.

$$T^{\text{shift}} = \frac{V_{\text{solid}}}{\dot{V}_s}$$

Where V_{Solid} is the solid volume and \dot{V}_s the volumetric flow rate of the phase respectively. The counter-current stream is only realized at the switching. Otherwise the columns are only a serial connection of conventional fixed beds. The necessary switches of a column to reach its initial position is called a cycle. Due to the discrete switching mode, there is no stationary but only a cyclic steady state in SMB technology.

The four sections of the SMB fulfil the same functions as in the TMB process, i.e. in the zones II and III the binary mixture is separated, and in the zones I and IV the solid and the liquid are cleaned, respectively. There are the same requirements in length or number of columns as in TMB.

Because of the desorption the extract-concentration is decreased and the breakthrough causes an increasing concentration in the raffinate at each switching interval. Typical concentration profiles of both outlets for a 4-column SMB configuration are shown in Fig. 10.

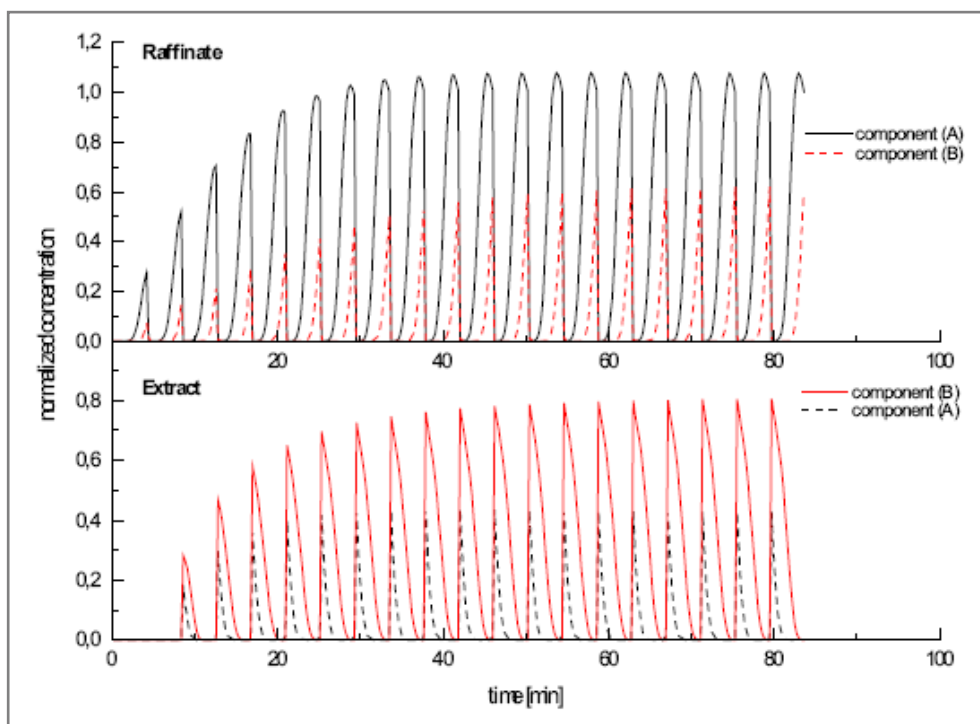


Fig. 10 Concentration profiles at extract and raffinate outlet (dashed lines: impurities)

In Fig. 10 the concentrations are normalized on the feed concentration of each component. These normalized concentrations are plotted versus the time coordinate. There are typical zigzag profiles, where a triangle symbolizes one switching interval or tact. After 12 of these intervals (i.e. 3 full cycles for a four column SMB process) the cyclic steady state is reached (two proceeding triangles are almost identical). By integrating the profiles over the time range, the averaged concentration can be calculated, i.e. the concentration measured in a vessel.

Since only four columns (minimal number) are supposed in simulating Fig. 10 the separation performance is low, i.e. there are impurities of the other component in each outlet. Increasing the number or the length of the columns would increase the separation performance.

2.5 Efficiency and economic aspects

Chromatographic separation processes belong to the expensive ones, compared with other thermal separation processes (distillation, crystallization, etc.). This is caused by high prices for the adsorbents (e.g. spherical silica gel in a high quality, chemically modified) and by the pure solvents needed.

Nevertheless, chromatographic processes are very effective. They are suitable for difficult separation problems, where the mixture compounds have very similar physico-chemical properties as well as for the separation of very expensive products. This is fulfilled for many products of the pharmaceutical and fine chemical industry or the food and drug industry. In these industries some compounds are produced, which can only be separated by chromatography.

In the previous chapters, three chromatographic modes were described (elution, TMB, SMB). Out of these modes preparative elution chromatography is theoretically as well as practically the best known one. It has been applied for a long time and creates the standard, at that new methods are measured.

The modes are suitable for preparative scale processes. These scales can be very different ones: While in petrol-chemistry 100,000 metric tons per annum are produced, in the pharmaceutical industry only a few kilograms are produced. From an industrial point of view only elution chromatography and the SMB process are attractive out of the three modes, due to the difficulties connected with the solid movement in TMB.

To compare the two important modes (elution, SMB) in a fair way, the plant scale has to be considered. After optimization of the two modes different optimal points in the multidimensional space of process relevant parameters are reached.

Such parameters are in the elution mode the volumetric flow rate of the eluent, the injected amount of the mixture per pulse, the time gap between two pulses, the produced amounts of purified compound, the column geometry, the properties of the column packing, the required purity, the temperature, etc. In SMB the volumetric flow rates (solid and liquid) in each zone, the number of columns and the column geometry, the produced amounts of purified compounds, the feed and withdrawal concentrations, the properties of the column packing, the required purity, the temperature etc. are relevant. Both modes are suitable for various separation tasks. The specific problem, hence, has to be known for a fair comparison.

The SMB process enables the separation of binary mixtures by means of a simulated countercurrent between the solid and liquid phases. This is accomplished with a series of chromatography columns arranged in a ring. An eluent flow circulates through this ring. Two inlets (for feed and eluent) and two outlets (extract/red and raffinate/blue) define four separation zones. By continuously feeding sample and synchronously switching the columns against the eluent flow direction, a countercurrent is achieved between the solid and liquid phases, leading to high purity of both target fractions. The movement of the solid phase is realized by simultaneously switching seven multi-position valves (AZURA® SMB).

The SMB saves up to 90% of the eluent in comparison to a batch process. Due to the simulated countercurrent, the stationary phase is significantly better utilized with the SMB technique as compared to the batch process technique. The number of theoretical plates might be also less important, making it possible to use cost-effective larger particle size for the stationary phases.

3. Scope of delivery



Note: Only use original parts and accessories made by KNAUER or a company authorized by KNAUER.

3.1 AZURA SMB Lab System

- 1 x AZURA® Assistant ASM 2.1 L with 1 x AZURA® Pump P4.1S (10 ml pump head) and 1 x 8-Multiposition-valve
- 3 x AZURA® Assistant ASM 2.1 L with 1 x AZURA® Pump P4.1S (50 ml pump head) and 2 x 8-Multiposition-valve
- SMB-Set with 1/16" capillaries, screw connections, T-pieces and connections for the assembly of the system
- Open/Closed Loop Kit
- PurityChrom® MCC control software
- Eluent tray AZURA® E2.1 L
- Chromatography Work station with Mini-PC and 24" monitor
- WLAN Router

Stainless Steel version:

- Pump heads (stainless steel)
- Multiposition-valve-head (stainless steel)
- Capillaries (stainless steel)

Biocompatible version:

- Pump heads (ceramic)
- Multiposition valve head (PAEK)
- Capillaries (PEEK)

Optional:

- Flowmeter
- Oven
- Detectors

Valid documents:

- Instructions for AZURA SMB system (document no. V6775)
- Declarations of conformity for single devices

3.2 AZURA SMB Pilot System

- 1 x AZURA® Assistant ASM 2.1 L with 1 x 8-Multiposition-valve
- 3 x AZURA® Assistant ASM 2.1 L with 2 x 8-Multiposition-valves
- 4 x AZURA® Pump P 2.1 L
- SMB-set with 1/8" capillaries, screw connections, T-pieces and connections for the assembly of the system
- Open/Closed Loop Kit
- PurityChrom® MCC control software
- Chromatography Work station with Mini-PC and 24" monitor
- WLAN Router

Stainless Steel version:

- Pump heads (stainless steel)
- Multiposition-valve-head (stainless steel)
- Capillaries (stainless steel)

Optional:

- Flowmeter
- Oven
- Detectors

Valid documents:

- Instructions for AZURA SMB system (document no. V6775)
- Declarations of conformity for single devices

4. Basic safety instructions

4.1 Target group

This document address persons who are qualified as chemical laboratory technicians or have completed comparable vocational training.

The following knowledge is required:

- Fundamental knowledge of liquid chromatography
- Knowledge regarding substances that are suitable only to a limited extent for use in liquid chromatography
- Knowledge regarding the health risks of chemicals
- Participation during an installation of a device or a training by the company KNAUER or an authorized company.

If you do not belong to this or a comparable professional group, you may not perform the work described in these instructions under any circumstances. In this case, please contact your superior.

4.2 Safety equipment

When working with the device, take measures according to lab regulations and wear protective clothing:

- Safety glasses with side protection
- Protective gloves
- Lab coat

4.3 What must the user take into account?

- All safety instructions in this document
- The environmental, installation, and connection specifications in this document
- National and international regulations pertaining to laboratory work
- Original spare parts, tools, and solvents made or recommended by KNAUER
- Good Laboratory Practice (GLP)
- Accident prevention regulations published by the accident insurance companies for laboratory work
- Filtration of substances under analysis
- Use of inline filters
- Once the capillaries have been used, never re-use them in other areas of the HPLC system.
- Only use a given PEEK fitting for one specific port and never re-use it for other ports. Always install new PEEK fittings on each separate port.
- Follow KNAUER or manufacturer's instructions on caring for the columns.

More safety-relevant information is listed below:

- flammability: Organic solvents are highly flammable. Since capillaries can detach from their screw fittings and allow solvent to escape, it is prohibited to have any open flames near the analytical system.
- solvent tray: Risk of electrical shock or short circuit if liquids get into the device's interior. For this reason, place all bottles in a solvent tray.
- solvent lines: Install capillaries and tubing in such a way that liquids cannot get into the interior in case of a leak.
- leaks: Regularly check if any system components are leaking.
- power cable: Defective power cables are not to be used to connect the device and the power supply system.
- self-ignition point: Only use eluents that have a self-ignition point higher than 150 °C under normal ambient conditions.
- power strip: If several devices are connected to one power strip, always consider the maximum power consumption of each device.
- power supply: Only connect devices to voltage sources, whose voltage equals the device's voltage.
- toxicity: Organic eluents are toxic above a certain concentration. Ensure that work areas are always well-ventilated! Wear protective gloves and safety glasses when working on the device!

Where is use of the device prohibited?

Never use the system in potentially explosive atmospheres without appropriate protective equipment. For further information, contact the Technical Support of KNAUER.

Secure decommissioning





Take the device completely out of operation by disconnecting the power plug from the power supply (wall socket or power strip).

Opening the device

The device may be opened by the KNAUER Technical Support or any company authorized by KNAUER only.

4.4 Warning notifications

Possible dangers related to the device are divided into personal and material damage in these instructions.

Sign	Meaning
	DANGER (red) indicates a hazardous situation which, if not avoided, will result in death or serious injury.
	WARNING (orange) indicates a hazardous situation which, if not avoided, could result in death or serious injury.
	CAUTION (yellow) indicates a hazardous situation which, if not avoided, could result in minor or moderate injury.
	NOTICE (blue) is used to address practices not related to physical injury.

4.5 Decontamination

Contamination of devices with toxic, infectious or radioactive substances poses a hazard for all persons during operation, repair, sale, and disposal of a device.

⚠ DANGER

Life-threatening injuries
Health danger if getting in contact with toxic, infectious or radio-active substances.

➔ Before disposing of the device or sending it away for repair, you are required to decontaminate the device in a technically correct manner.







All contaminated devices must be properly decontaminated by a specialist company or the operating company before they can be recommissioned, repaired, sold, or disposed of. All materials or fluids used for decontamination must be collected separately and disposed of properly.

Decontamination Report

Devices without a completed Decontamination Report will not be repaired. If you would like to return a device to KNAUER, make sure to enclose a completed Decontamination Report with the device:
<https://www.knauer.net/decontamination-report>.

5. Symbols and signs

The following symbols and signs can be found on the devices:

Symbol	Meaning
	Electric shock hazard
	Electrostatic discharge hazard, damages to system, device, or components can occur.
 0.5 kg	Obey maximum load for leak tray during transportation, installation and operation.
	A device or system marked with CE fulfills the product specific requirements of European directives. This is confirmed in a Declaration of Conformity.
	Testing seals in Canada and the USA at nationally recognized testing centers (NRTL). The certified device or system has successfully passed the quality and security tests.
	On some devices, a warranty seal is attached. For more information see paragraph 14.3 on page 52.

6. Unpacking and setup

The HPLC system will be set up, installed and commissioned by KNAUER or a company authorized and contracted by KNAUER.



Note: KNAUER recommends inviting future users to be present while setting up and commissioning the module so that they can become familiar with the analyzer and how to handle it.

6.1 Operating environment

Only if the requirements for ambient conditions of the operating environment are met, can the intended use be ensured. Details on the operating conditions can be found in the chapter „Technical Data“.

6.1.1 Space requirements

- At least 5 cm space if another device is set up on one side
- At least 10 cm space if further devices are set up on both sides
- At least 15 cm space on the rear panel for the fan.
- Make sure that the power plug on the power supply (wall mounted socket or power strip) is always accessible, so that the device can be disconnected from the power supply.

6.1.2 General requirements

NOTICE

Device defect

The device overheats at exposure to sunlight and insufficient air circulation. Device failures are very likely.

- ➔ Set up the device in such a way that it is protected against exposure to direct sunlight.
- ➔ Leave room for air circulation: See paragraph „space requirements“.

- Position the device on a level and even surface.
- Protect the device against direct exposure to sunlight.
- Set up the device at a location not exposed to air drafts (A/C systems).
- Do not set up the device near other machines that cause floor vibrations.
- Keep the device away from high-frequency sources. High frequencies may compromise measuring values.
- Avoid sources of high frequencies near the device. High-frequency sources may compromise measuring values.

6.1.3 Earthquake areas

If you are located in an earthquake area, use the bore holes ① in the side panels to secure the device. The bore holes are located on either right or left side panel.



6.2 Storing unopened shipping boxes

In your planning, include following information for immediate storage.

- sufficient space
- storage temperatures must be in the temperature range 4-40 °C, 39-104 °F

6.3 Unpacking

Prerequisite

- Check packaging for damage caused during transportation. If necessary, put forward any claim for damages to the carrier.

Tool

- Utility knife

⚠ CAUTION

Bruising danger

Damage to the device by carrying or lifting it on protruding housing parts. The device may fall and thus cause injuries.

➔ Lift the device only centrally on the side of the housing.

Process

Steps

1. Set up the package in such a way that you can read the label. Using the utility knife, cut the adhesive tape and open the packaging.
2. Remove the foam insert. Take out the accessory kit and the instructions.
3. Open the accessory kits and check the scope of delivery. In case any parts are missing, contact the Technical Support.
4. Clasp the device from below, lift it out of the packaging and place it on its feet. Do not hold onto the front cover.
5. Check the device for signs of damage that occurred during transport. In case you notice any damage, contact the Technical Support.
6. Place the device in its site of operation and remove protective foil.

Next step(s)

Store packaging and keep the included packing list for repeat orders.

6.4 Power supply

Prerequisites

- The electrical power supply at the installation site must be connected directly to the nearest main power line.
- The power must be free from ripple, residual current, voltage peaks and electromagnetic interference.
- The connectors for the mains voltage are grounded accordingly.
- The device receives sufficient power with reserve capacity.

Power cable

- Use only the enclosed power cable to connect the device to the power supply to make sure that the specifications are met which are described in the chapter "Technical Data".
- Beforehand, make sure to use power cables which are admitted for use in your country.
- Replace defective power cables only with accessories from KNAUER.
- Do not replace detachable power cables with different cable types.

NOTICE

Electronic defect

Electronic hazard when using an identically constructed power adapter from another manufacturer.

→ Only use spare parts and accessories from KNAUER or a company authorized by KNAUER.

Power plug

- The device is intended for use with AC power networks of 100-240 V.
- Make sure that the power plug on the power supply (wall mounted socket or power strip) is always accessible, so that the device can be disconnected from the power supply.

6.5 Connecting the capillaries

NOTICE

Component defect

Damage to components due to excessive tightening possible.

Observe the torque of the screw connection

→ Use 5 Nm torque for stainless steel fittings.

→ Use 1 Nm torque for PEEK fittings.

6.6 Flow charts

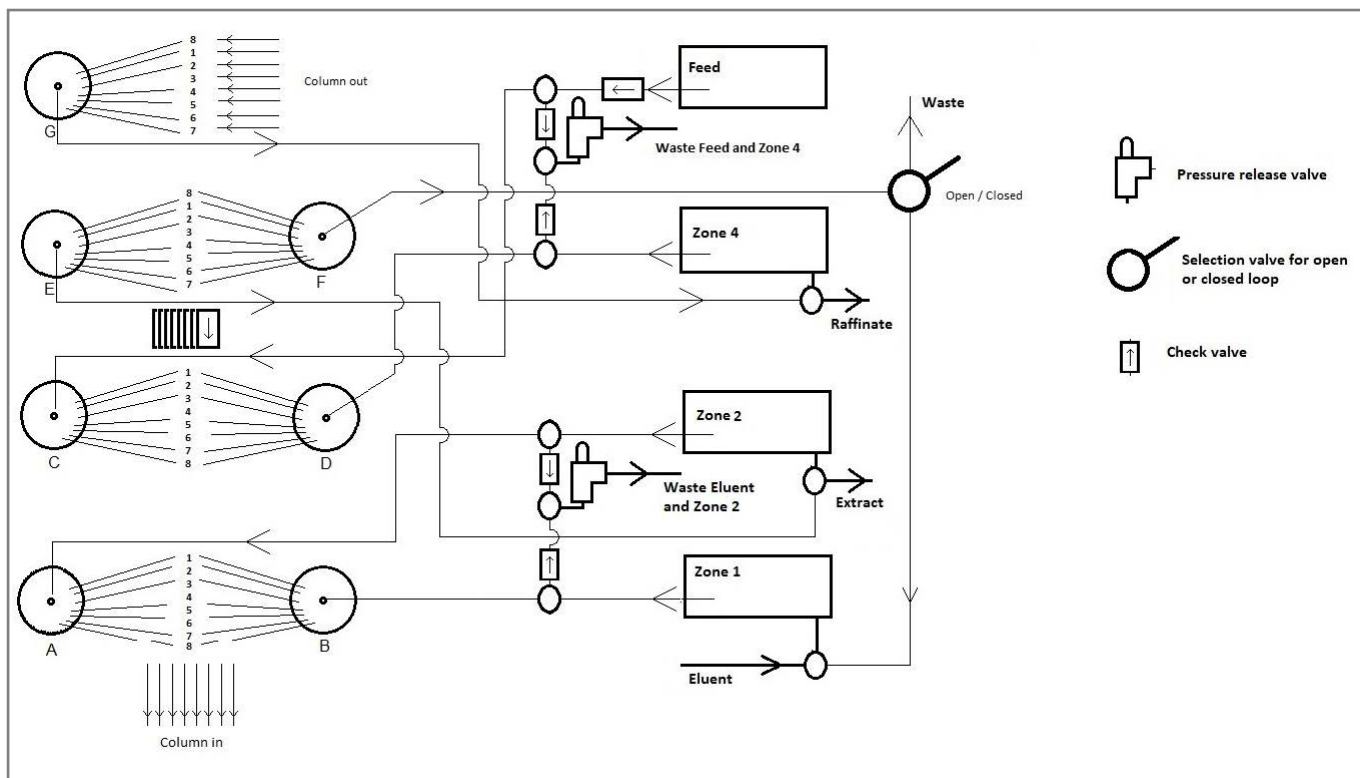


Fig. 11 Flow chart for AZURA® Lab system

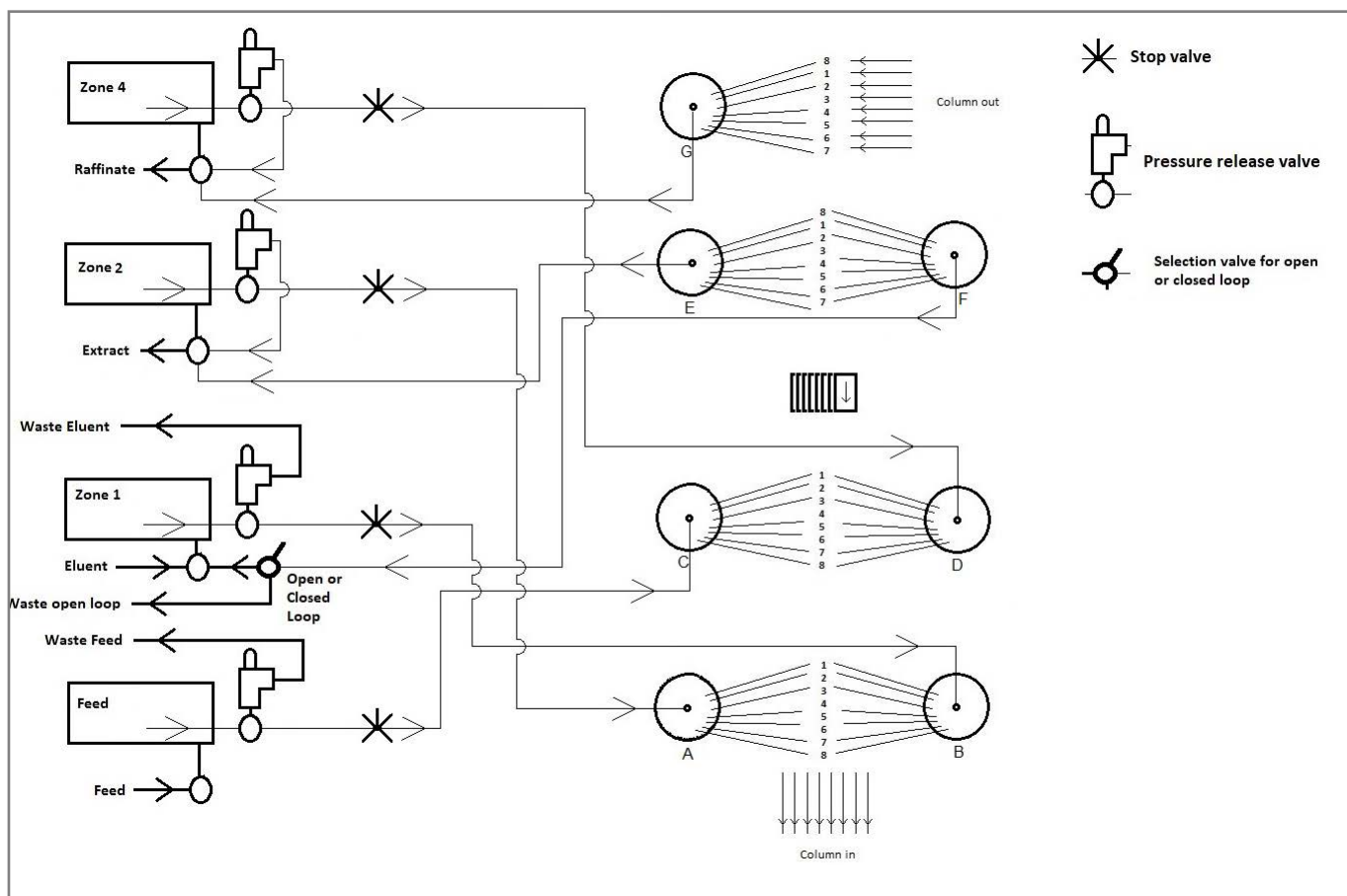


Fig. 12 Flow chart for AZURA® Pilot system

6.7 Connecting system to computer via LAN

The system can be operated in two ways:

- via remote connector
- as part of a LAN, via the LAN connector of the router

All connectors for external control are located on the back side of the single devices.

6.7.1 Connecting the single devices to the computer



Note: HPLC devices made by KNAUER work only with IP addresses which are assigned via IPv4. IPv6 is not supported.

This section describes how to set up an HPLC system in a local area network (LAN) and how a network administrator can integrate this LAN into your company network. The description applies to the operating system Windows and all conventional routers.

To set up a LAN, we recommend to use a router. That means the following steps are required:

6.7.2 Configuring the LAN settings

The LAN uses only one server (which is normally the router) from that the devices automatically receive their IP address.

Prerequisites

- In Windows, power saving, hibernation, standby, and screen saver must be deactivated.
- In case you use an USB-to-COM box, the option „Allow the computer to turn off this device to save power“ in the devicemanager must be deactivated for all USB hosts.
- For all LAN devices: For the network adapter, the following option in the Device Manager must be deactivated: „Allow the computer to turn off this device to save power“.

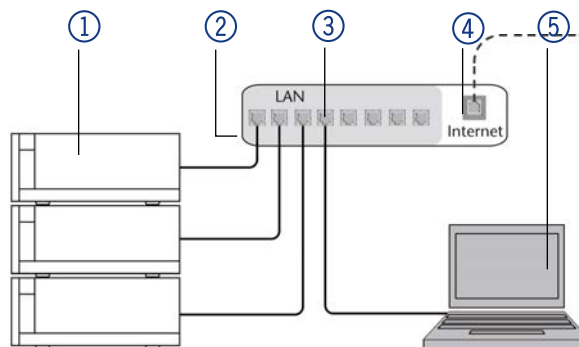
Process

Steps

1. In Windows open the Network and Sharing Center.
2. Double-click on LAN Connection.
3. Click on the button Properties.
4. Select Internet Protocol version 4 (TCP/IPv4).
5. Click on the button Properties.
6. Check the settings in the tab General. The correct settings for the DHCP client are:
 - a) Obtain IP address automatically
 - b) Obtain DNS server address automatically
7. Click on the button OK.

6.7.3 Connecting the cables

A router ② has several LAN ports ③ and one WAN port ④ that can be used to integrate the LAN into a wide area network (WAN), e.g. a company network or the Internet. In contrast, the LAN ports serve to set up a network from devices ① and a computer ⑤. To avoid interference, we recommend operating the HPLC system separately from the company network.



You will find a RJ45 patch cable for each device and the router in the accessories kit. To connect the router to a WAN, an additional patch cable is required, which is not supplied within the scope of delivery.

- Prerequisites**
- The computer is switched off.
 - There is a patch cable for each device and the computer.

Process

Steps

1. Use the patch cable to connect the router and the computer. Repeat this step to connect all devices.
2. Use the power supply to connect the router to the mains power system.

6.7.4 Configuring the router

The router is preset at the factory. You find information about IP address, user name and password in the router instructions:
www.knauer.net/router

Process

Steps

1. To open the router configuration, start your Internet browser and enter the IP address (not valid for all routers).
2. Enter user name and password.
3. Configure the router as DHCP server.
4. In the router configuration, check the IP address range and make changes if necessary.



Note: If the IP address range has been changed, it is necessary to note it down.

Result

Once the router has assigned IP addresses to all devices, the chromatography software can be used to remotely control the system.

6.7.5 Integrating the LAN into a company network

A network administrator can integrate the LAN into your company network. In this case you use the WAN port of the router.

- Prerequisite**
- There is a patch cable.

Process

Steps

1. Check that the IP address range of the router and of the company network do not overlap.
2. In case of an overlap, change the IP address range of the router.
3. Use the patch cable to connect the router WAN port to the company network.
4. Restart all devices, including the computer.

6.7.6 Controlling several systems separately in a LAN

Devices connected to a LAN communicate through ports, which are part of the IP address. If more than one HPLC system is connected to the same LAN and you plan on controlling them separately, you can use different ports to avoid interference. Therefore, the port number for each device must be changed and this same number must be entered into the device configuration of the chromatography software. We recommend to use the same port number for all devices in the same system.



Note: The port is set to 10001 at the factory. You must use the same numbers in the device configuration of the chromatography software as in the device, otherwise the connection fails.

Process

Steps

1. Find out port number and change it on the device.
2. Enter the port number in the chromatography software.

Result

The connection is established.

6.7.7 Remote control

On the rear panel of the single devices an electrical connector socket is located which serves to send or receive signals from other devices. For example start signals from an injection valve or an autosampler can be put to the START input. All voltages have to be mounted between GROUND and the corresponding event.

NOTICE

Electronic defect

Electrostatic discharge can destroy the electronics.

→ Wear a protective bracelet against electrostatic discharge and ground.

For test purposes or in some other cases, it can make sense to manually enter these signals.

- sending control signals (Events) to external devices
- opening and closing contacts
- activating 500 ms pulses

The following remote signals can be received and sent:

- for receiving start, control, and error signals from external devices
- for sending start, control and error signals to external devices



Note: For the specific relation between display and terminal strip, refer to the instructions of the device.

Connecting cables to the terminal strip

To control one device through another, you use the multi-pin connector. To use remote control, you have to connect cables to the terminal strip (both included with delivery). The single ports are used to exchange control signals.

Prerequisite

- The device is turned off.
- The power plug is pulled.

Tools

Operating tool

NOTICE**Electronic defect**

Connecting cables to the multi-pin connector of a switched on device causes a short circuit.

- ➔ Turn off the device before connecting cables.
- ➔ Pull the power plug.

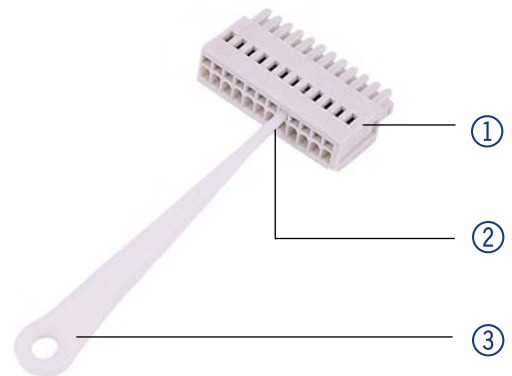
NOTICE**Electronic defect**

Electrostatic discharge can destroy the electronics.

- ➔ Wear a protective bracelet against electrostatic discharge and ground.

Process**Steps****Figure**

1. Push the operating tool ③ into an upper small opening on the front of the terminal strip ①.
2. Lead the cable into the opening ② below the inserted operating tool.
3. Remove the operating tool.

**Next steps**

- Check if the cables are firmly attached.
- Push the terminal strip onto the multi-pin connector.
- Finish the installation.
- Put the device into operation.

7. Start-up

7.1 Initial start-up checklist

NOTICE

Device defect

Changes of the environmental temperature cause condensation inside the device.

→ Allow device to acclimate for 3 h before connecting to power supply and taking into operation.

Use this checklist to determine if the system is ready for initial start-up:

- Devices are positioned in the correct location.
- The power plugs are connected.
- The network connection to the router is established
- The chromatography software has been installed by KNAUER or a company authorized by KNAUER.
- The capillaries are connected.

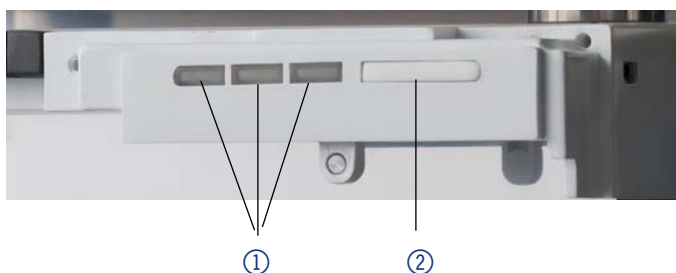
7.2 SOP: SMB power-up and shut-down

7.2.1 Power-up

If the assistants are connected via Error in/out, follow this procedure to power up the AZURA® SMB system.

Step	Task
1	Power up the router and wait until the self-test is performed.
2	Power up all instruments and the PC.
3	Shortly press the switch (see picture below no. ②) to deactivate the error message (red flashing of the left LED see picture no. ①) of the assistants.

Note: The red flashing is a normal behaviour and a safety feature, that indicates if one assistant has no power, the other assistants show an error warning.



4	If a user management is used on the PC, login with the correct credentials.
5	Start the PurityChrom® software with double click on the desktop icon.

If the assistants are NOT connected via Error in/out, follow this procedure to power up the SMB.

Step	Task
1	Power up the router and wait until the self-test is performed.
2	Power up all instruments and the PC.
3	If a user management is used on the PC, login with the correct credentials.
4	Start the PurityChrom® software with double click on the desktop icon.

7.2.2 Shut down

Follow this procedure to perform an AZURA® SMB shut down.

Step	Task
1	Close the software PurityChrom®.
2	Power up the PC.
3	Turn off all instruments.
<p>Note: If the assistants are connected via Error in/out, after shutting one assistant down, the other assistants will show an error message by a red flashing of the left LED. This behaviour is normal and a security feature.</p>	
4	Turn off the router.

7.3 SOP: SMB start-up

Follow this procedure to perform an AZURA® SMB system start-up.

Prerequisite Ensure that the eluents are HPLC quality and are filtered by 0.45 µm filter.

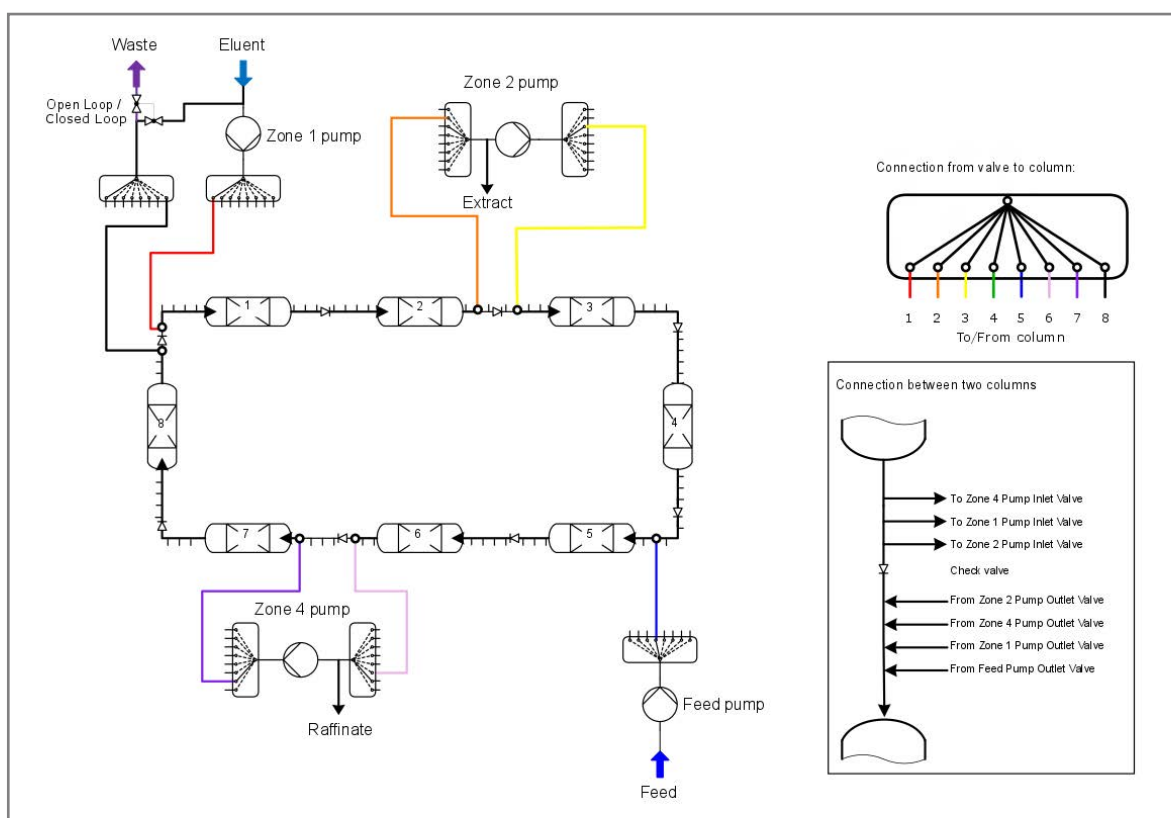


Fig. 13 In the main flow scheme, only active lines in start position between column loop and multi-position lines are shown. Each connection on the multi-port valves up- and downstream of the pumps is connected to the related connection port between the columns as shown in the figure.

7.3.1 Removing air, flushing/cleaning the SMB and cleaning columns

To remove air from the SMB system, flush/clean the SMB or clean the columns, the system must be switched into the "Open Loop" mode. Afterwards the pumps will be started step by step and the air or the contamination will be removed from the system.

The system must be flushed / cleaned stepwise from Zone 1 to Zone 2, from Zone 2 and 3 to Zone 4 and from Zone 4 to waste.



Note: High flow rates will generate high backpressure. Do not try to flush the system with the highest flow rate possible.

7.3.2 Preparation

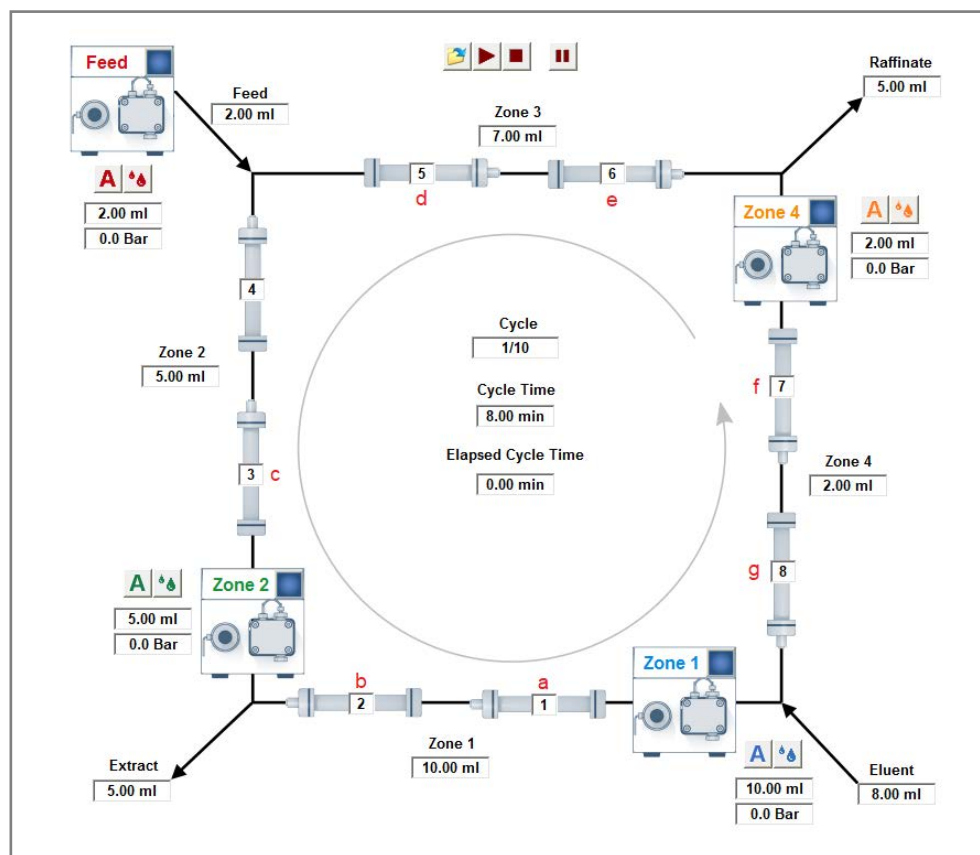


Fig. 14 Visualization of SMB system with initial valve position in Puritychrom® MCC


Step	Task
1	"Loop valve" is in position "Open Loop". Otherwise the air/contamination will be transferred continuously through the SMB.
2	Set the valves (default values): <ol style="list-style-type: none"> pump "Zone 1 out" to 1 pump "Zone 2 in" to 2 pump "Zone 2 out" to 3 pump "Feed out" to 5 pump "Zone 4 in" to 6 pump "Zone 4 out" to 7 pump "Zone 1 in" to 8
3	Open the purge valve of Zone 1 pump and connect it to a syringe.


Step	Task
4	Set the flow rate at 10 % of the pump heads max. flow.
5	Suck eluent through the pump.
6	When no more air bubbles are visible in the inlet tubing, stop the pump and close the purge valve.

7.3.3 Flushing the system



Note: For flushing the system without column, proceed here. For cleaning the whole system including columns, go to chapter "Flushing all columns".

Step	Task
1	For the Zone 1 pump, set the flow rate at 25 % of the pump heads max. flow.
2	Wait until there is a stable flow rate exiting the extract line and no more air bubbles are visible in the extract outlet. A slow dripping out of the raffinate line can occur. <div data-bbox="619 817 687 891" style="float: left; margin-right: 10px;"> </div> <div data-bbox="713 817 1439 990" style="float: right;"> <p>Note: If the eluent is mainly exiting the raffinate or waste line, proceed to step 3 and 4. Afterwards continue with step 2. If the eluent is still mainly exiting the raffinate or waste line, check if valve positions, flow rates, purge valves and external check valves.</p> </div>
3	Switch all valves to the next position (Valve Control: All Valves, Next Position -> Set)
	
	Wait until no air bubbles leave the extract.
4	Repeat step 3 until you reach the initial valve position.
5	Connect a syringe to the purge valve of the Zone 2 pump and open the purge valve.
6	For the Zone 2 pump, set the flow rate at 15 % of the pump heads max. flow.
7	Suck the eluent through the pump and wait until no more air bubbles leave the pump.
8	Close the purge valve.
9	Wait until there is a stable flow rate exiting the raffinate line and no more air bubbles are visible in the raffinate outlet. A slow dripping out of the waste line can occur. <div data-bbox="619 1758 687 1832" style="float: left; margin-right: 10px;"> </div> <div data-bbox="713 1758 1439 1854" style="float: right;"> <p>Note: The flow rate in raffinate should be higher than in extract, otherwise please check the valve positions, flow rates, purge valves and external check valves.</p> </div>
	Please ensure that the pumps were flushed sufficiently. If not, repeat the procedure from step 1.
10	Connect a syringe to the purge valve of the Feed pump and open the purge valve.
11	For the Feed pump, set the flow rate at 25 % of the pump heads max. flow.

Step	Task
12	Suck the eluent through the pump and wait until no more air bubbles leave the pump.
13	Close the purge valve.
14	Wait until there is a stable flow rate exiting the raffinate line and no more air bubbles are visible in the raffinate outlet. A slow dripping out of the waste line can occur. <div style="display: flex; align-items: flex-start;"> <div style="border: 1px solid blue; border-radius: 50%; width: 30px; height: 30px; display: flex; align-items: center; justify-content: center; margin-right: 10px;">i</div> <div> <p>Note: The flow rate in raffinate should be higher than in extract, otherwise please check the valve positions, flow rates, purge valves and external check valves. Please ensure that the pumps were flushed sufficiently. If not, repeat the procedure from step 10.</p> </div> </div>
15	Connect a syringe to the purge valve of the "Zone 4 pump" and open the purge valve.
16	For the Zone 4 pump, set the flow rate at 10 % of the pump heads max. flow.
17	Suck the eluent through the pump and wait until no more air bubbles leave the pump.
18	Close the purge valve.
19	The "extract" flow should now be equal to the "raffinate" flow. Otherwise, check the valve positions, flow rates, all purge valves and external check valves.
20	Wait until there is a stable flow rate exiting the waste line and until no more air exits the system through the waste tubing plus additional 30 min.
21	Switch all valves to the next position (Valve Control: All Valves, Next Position -> Set)
	
	Wait until no air bubbles leave the extract, raffinate and waste outlet.
22	Repeat step 21 until you reach the initial valve position.

7.3.4 Washing one single column



Prerequisite

Check if the correct washing solvent is connected to the eluent inlet.



Note: The back pressure caused by the specific columns may be too high with the recommended flow rates in the SOP. Reduce the flow rates for each pump in the same ratios to reach moderate pressure values (~ < 50 % of p max.).

Step	Task
1	Set the "Loop valve" in position "Open Loop". Otherwise the air/contamination will be transferred continuously through the SMB.

Step	Task
2	Set the valves "pump Zone 1 out" and "pump Zone 1 in" to the number of the column you want to clean up. Set every other column to +4 positions in comparison of the "washing column". e.g.: washing of column 1 a. pump "Zone 1 out" to 1 b. pump "Zone 2 in" to 5 c. pump "Zone 2 out" to 5 d. pump "Feed out" to 5 e. pump "Zone 4 in" to 5 f. pump "Zone 4 out" to 5 g. pump "Zone 1 in" to 1
3	Open the purge valve of Zone 1 pump and connect it to a syringe.
4	For Zone 1 pump, set the flow rate at 10 % of the pump heads max. flow.
5	Suck eluent through the pump.
6	When no more air bubbles are visible in the inlet tubing, stop the pump and close the purge valve.
7	For Zone 1 pump, set the flow rate at 20 % of the pump heads max. flow.
8	Wait until a stable flow in the waste channel.
9	Wash the column with at least ten column volumes.  Note: It might be necessary to use more eluent to clean the columns or to change the eluent, to get the impurities removed.
10	Stop the pump.  Note: Skip the next two steps, if the columns have been washed with the SMB eluent.
11	Change the eluent to the eluent used in the SMB process.
12	Start the pump and flush the column with at least 5 column volumes.

7.3.5 Flushing all columns



Note: If you want to flush your columns with a new solvent, please run the procedure "Flushing the system" with five column volumes (whole column volume!).



Note: The back pressure caused by the specific columns may be too high with the recommended flow rates in the SOP. Reduce the flow rates for each pump in the same ratios to reach moderate pressure values (~ < 50 % of p max.).

7.3.6 Washing all columns



Note: If you want to wash your columns, please run the procedure "Flushing the system" with ten column volumes (whole column volume!).



Note: The back pressure caused by the specific columns may be too high with the recommended flow rates in the SOP. Reduce the flow rates for each pump in the same ratios to reach moderate pressure values (~ < 50 % of p max.).

8. Operation

8.1 Software operation

To operate the system with software, you have to establish a connection between the LAN port and a computer. The system can be controlled with Puritychrom® MCC. You find a detailed description on chromatography software in the corresponding software instructions (document no. [V2660](#)).

8.2 Meaning of the LEDs

There are three LEDs and a switch on the front of the devices.

Legend

- ① Left LED
- ② Center LED
- ③ Right LED
- ④ Switch/Standby button

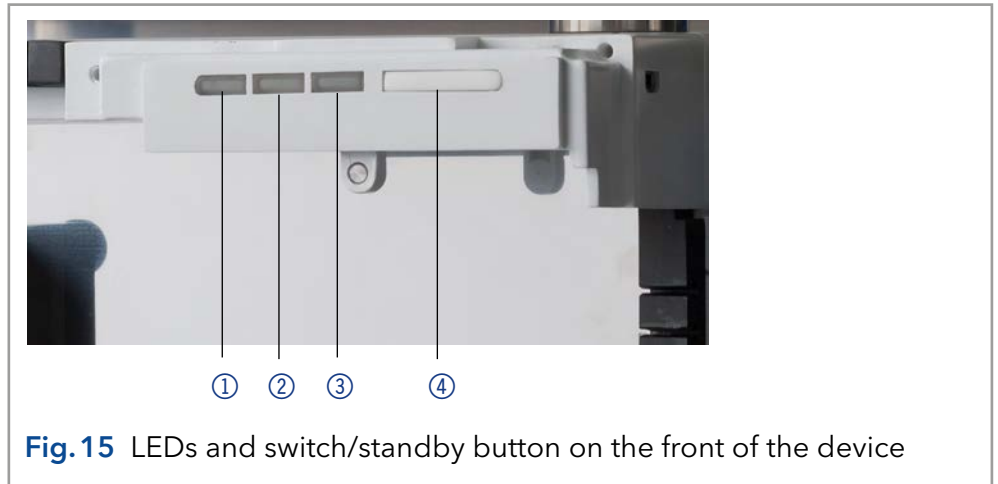


Fig. 15 LEDs and switch/standby button on the front of the device

The LEDs can have different colors depending on the operating conditions.

Standby To start the standby, keep the standby button pressed for 5 seconds.



Note: Malfunctioning system after repeated standby possible. After repeatedly using standby, restart the device using the power switch to reset the device's data storage.

	Color	Status	Operation
Left LED	red	Error	<ul style="list-style-type: none"> ▪ Check the system. ▪ Shortly press the switch to deactivate the error message.
	green	3D data are acquired.	
Center LED	does not light	The lamp has been switched off.	
	flashes green	The lamp/lamps are initializing or the validation is progressing.	<ul style="list-style-type: none"> ▪ Wait until the lamp is running or the validation is finished.
	green	The deuterium lamp is active.	
Right LED	green	The device has been switched on.	




Color	Status	Operation
flashes green	The device not ready for operation.	<ul style="list-style-type: none"> Wait until the device is ready for operation.
blue	The device is in standby	<ul style="list-style-type: none"> Press the standby button to end the standby.

8.3 SOP: AZURA® SMB separation

For an overview of the flow scheme, refer to Fig. 9 on page 26.

- Prerequisites**
- Ensure that the eluents are HPLC quality.
 - Ensure that the feed is filtered by 0.45 µm filter.

8.3.1 Preparation



Step	Task
1	<p>Perform a preparative separation to determine the SMB parameters. Use the same column type as for the SMB process (dimension and packing material). The columns must be as similar as possible regarding the test parameters of the quality test (RT, asymmetry, theoretical plates) to guarantee proper results.</p> <p>A separation with an analytical column is also possible. Therefore an analytical column must be used with the same physical properties of the material as the columns used in the SMB process.</p> <p> Note: We recommend to use a preparative separation with a separation factor between 1.2 and 2 as basis for the SMB process. Values below 1.2 and above 2.0 will decrease the efficiency enormously.</p>
2	Determine the adsorption type (i.e. linear isotherm) and the adsorption parameters to calculate the SMB parameters.
3	Transfer the parameters to the current SMB scale, if necessary.
4	<p>Flush the system first without columns, install the columns and equilibrate the columns inside the system (see section 7.3.3).</p> <p> Note: In case of massive contamination inside the system, the system has to be washed without columns. It may be necessary to use back pressure capillaries.</p>
5	<p>Insert the SMB parameter into the software.</p> <p> Note: In case of a linear adsorption, you can calculate the SMB parameter directly in the software (some system information are required).</p>

8.3.2 Starting the system

Step	Task
1	Connect the feed pump with the eluent tray. Ensure there is no contamination of the eluent with remaining feed at the tubing. "Purge" the pump with eluent by using a syringe.
2	Wait until the temperature is already equilibrated.
3	Start the system with the calculated parameters.

Step	Task
4	Check for leakage and proper work of pumps and flowmeter.
5	Check dripping at the Extract and Raffinate outlets.

8.3.3 Pausing the separation

Step	Task
1	If required, the SMB system can be paused at every time by clicking the pause button ().
2	The time will be paused, and all pumps will stop. The valves do not switch anymore.
3	To restart the separation, click the play button ().

Improving the productivity

The productivity can be improved using the following methods:

- Increasing the feed concentration



Note: May cause impurity in raffinate or extract, see below "Contamination of raffinate in extract".

- Increasing the feed flow



Note: May cause impurity in raffinate or extract, see below "Contamination of raffinate in extract". Can destabilize the SMB process, new process optimization might be necessary.

- Optimizing the SMB parameter

Contamination

Contamination of raffinate in extract

Contamination due to zone IV:

- Open the eluent recycling valve and check on an analytical instrument if there is any contamination of raffinate in the waste.
- If there is a contamination, decrease the flowrate in zone IV, otherwise go ahead.

Contamination due to zone II:

- Increase the flow rate in zone II to eliminate the raffinate component from this zone.

Contamination of extract in raffinate

Contamination due to zone IV:

- Open the eluent recycling valve and check if there is any contamination of extract in the waste.
- If there is a contamination, increase the flowrate in zone I.

Contamination due to zone II:

- Decrease the flow rate in zone II to prevent that the extract will contaminate the raffinate component here.



Note: After changing parameters, the effect will be shown up to three cycles later.

- Analyze after each cycle.

8.3.4 Stopping the separation

Step	Task
1	Stop the data collecting.
2	Clean the system: Use the SOP "SMB start-up" (see section 7.3 on page 25).
3	Stop the heater (if possible/necessary).

8.4 Change Zone configurations

8.4.1 From 2:2:2:2 to 1:3:3:1

This section describes the change of the zone configuration to 1:3:3:1 (see Tab.1).

Zone	Columns per zone
1	1
2	3
3	3
4	1

Tab. 1 Zone configuration 1:3:3:1



Note: For a customized SMB set-up with i.e. flowmeters and detectors the visualisation must be adapted.

Preparation

Step	Task
1	Please ensure that the SMB is switched off and the software PurityChrom® MCC is closed.
2	Insert the following files into the described subdirectories : a. KNAUER-SMB-1-3-3-1.vis → C:\PurityChrom\Visualisation\Visualisation Files b. KNAUER SMB 1-3-3-1.bmp → C:\PurityChrom\Visualisation\Visualisation Backgrounds
3	Start the PurityChrom® MCC Software.
4	Change the start position of the valves according to Tab. 2.

Valve	Valve positions
1	1
2	1
3	2
4	5
5	7
6	8
7	8

Tab. 2 Start position of the valves for zone configuration 1:3:3:1

Procedure

Step Task

- 1 Open the PurityChrom® MCC Setup here:



PurityChrom-MMC Setup

File

User Defined Channel	Dead Time / Volume	Valve Locking	Alarm Outputs	Program Colors	
Communication	Presets	Limiter	Annotation	Descriptions	
Device	Addr.	Port	Baudrate	RTS	Driver
<input checked="" type="checkbox"/> Eluent pump	1	Winsock 3	9600	<input type="checkbox"/>	Knauer Pump 100/10P/20P/P2.1S/P4.1
<input checked="" type="checkbox"/> Zone 2 pump	2	Winsock 6	9600	<input type="checkbox"/>	Knauer Pump 100/10P/20P/P2.1S/P4.1
<input checked="" type="checkbox"/> Feed pump	3	Winsock 11	9600	<input type="checkbox"/>	Knauer Pump 100/10P/20P/P2.1S/P4.1
<input checked="" type="checkbox"/> Zone 4 pump	4	Winsock 9	9600	<input type="checkbox"/>	Knauer Pump 100/10P/20P/P2.1S/P4.1
<input type="checkbox"/> Collector	7	Com 3	9600	<input type="checkbox"/>	CAT ThirdArm
<input type="checkbox"/> Heater Control	6	Com 1	9600	<input type="checkbox"/>	MultiCom Interface (Serial Control)
<input type="checkbox"/> Serial Event Box	7	Com 1	9600	<input type="checkbox"/>	MultiCom Interface (Serial Control)

Number of Valves: 7

Valve 1	Valve 2	Valve 3	Valve 4	Val
Address	Positions	Startposition	Port	
*	8	1	Winsock 2	
Type	Vici Valve			

Control Inputs

Stop all: Disabled

Time Control Start: Event Box Input 1

Time Control Hold/Continue : Disabled

- 2 Change the Startposition of Valve 1 to 1.

Number of Valves: 7

Valve 1 | Valve 2 | Valve 3 | Valve 4 | Val

Address	Positions	Startposition	Port
*	8	1	Winsock 2
Type	Vici Valve		

- 3 Change the Startposition of Valve 2 to 1.

Number of Valves: 7

Valve 1 | Valve 2 | Valve 3 | Valve 4 | Val

Address	Positions	Startposition	Port
*	8	1	Winsock 7
Type	Vici Valve		

- 4 Change the Startposition of Valve 3 to 2.

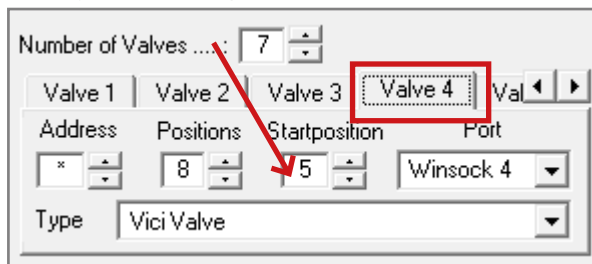
Number of Valves: 7

Valve 1 | Valve 2 | Valve 3 | Valve 4 | Val

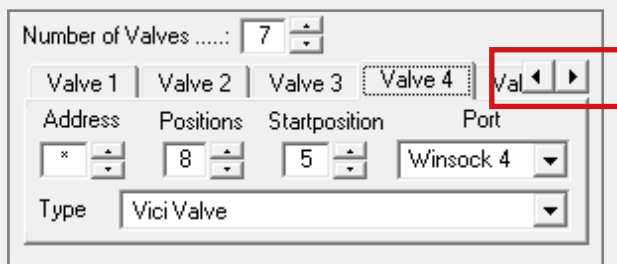
Address	Positions	Startposition	Port
*	8	2	Winsock 1
Type	Vici Valve		

Step Task

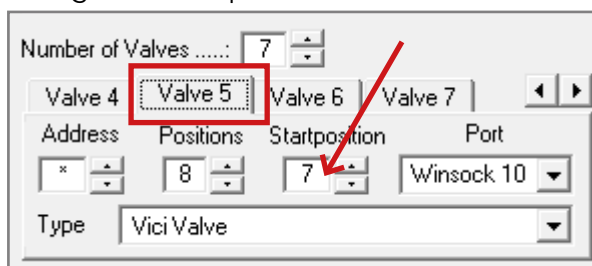
5 Change the Startposition of Valve 4 to 5.



6 Use the arrow buttons to switch to the Valves 5 to 8.



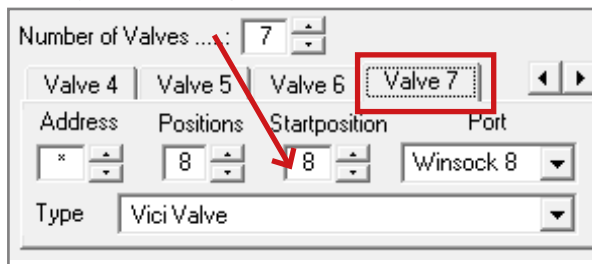
7 Change the Startposition of Valve 5 to 7.



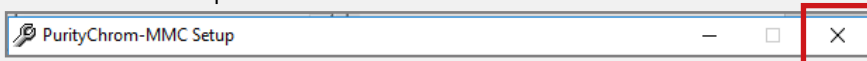
8 Change the Startposition of Valve 6 to 8.



9 Change the Startposition of Valve 7 to 8.

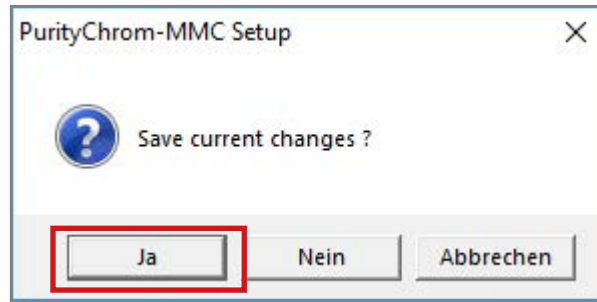


10 Close the Setup here:



Step	Task
------	------

- 11 Save the current changes with <Ja>.



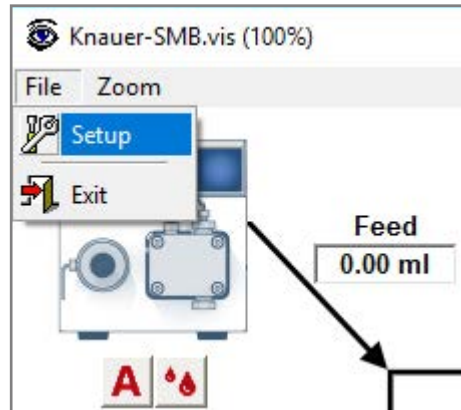
- 12 Close the PurityChrom® MCC Software.

- 13 Start the PurityChrom® MCC Software.

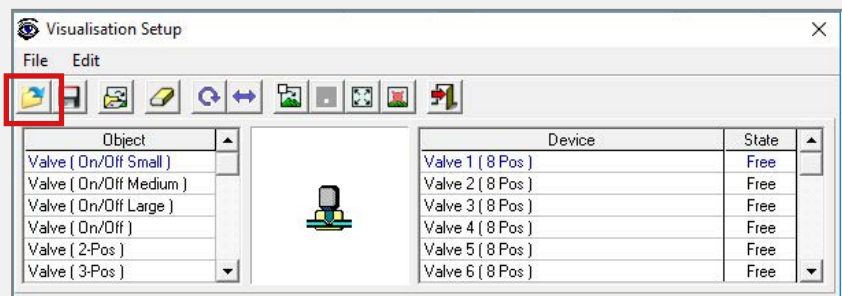
- 14 Open the Visualisation here:



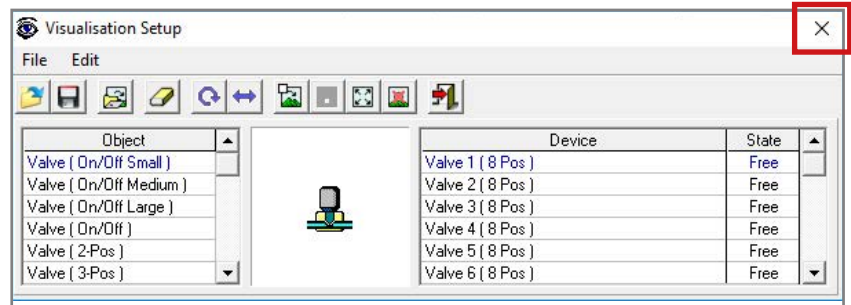
- 15 Open the Visualisation Setup here:



- 16 Load the Visualisation "KNAUER SMB 1-3-3-1".

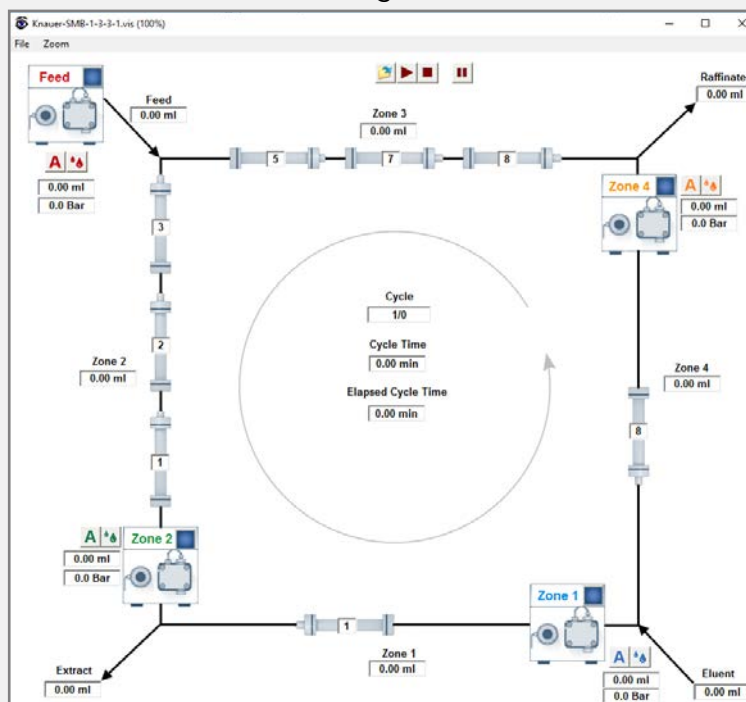


- 17 Close the Visualisation Setup.



Step	Task
------	------

- | | |
|----|--|
| 18 | The change to the zone configuration 1:3:3:1 is completed. You should see the following visualisation: |
|----|--|



8.4.2 From 2:2:2:2 to 1:1:1:1

This section describes the change of the zone configuration to 1:1:1:1.

Step	Task
------	------

- | | |
|---|---|
| 1 | Install one column on every second position: <ol style="list-style-type: none"> Column 1 at Position 1 Column 2 at Position 3 Column 3 at Position 5 Column 4 at Position 7 |
| 2 | Install a coupling instead of a column at the position 2, 4, 6, 8. |
| 3 | Calculate the SMB Parameters with the SMB Operating Point Calculator. |
| 4 | Divide the Switching Time in half and run the system with the resulting switching time. |

9. Functionality tests



Note: Standard processes in single devices may be handled differently in individual cases.



Note: Functionality tests can be requested individually for the single devices. The tests for the entire system is made by a Performance Verification without the columns. The flow rate accuracy is tested with a different document. Please contact the Technical Support for more details.

9.1 Installation Qualification (IQ)

The customer may request the IQ of KNAUER devices which is free of charge. In case of a request, the Technical Support of KNAUER or from a provider authorized by KNAUER performs this functionality test during the installation.

The IQ is a standardized document includes the following:

- Confirmation of flawless condition at delivery
- Check if the delivery is complete
- Certification on the functionality of the device.

9.2 Operation Qualification (OQ)

The OQ of KNAUER devices includes an extensive functionality test according to KNAUER standard OQ documents. The OQ is a standardized document and free of charge. It is not part of the delivery, please contact the Technical Support in case of request.

The OQ includes the following:

- Definition of customer requirements and acceptance terms
- Documentation on device specifications
- Device functionality check at installation site

Test intervals

To make sure that the device operates within the specified range, you should test the device regularly. The test intervals are dependent on the usage of the device.

Execution

The test can be carried out either by the Technical Support of KNAUER or from a provider authorized by KNAUER (for a fee).

10. Troubleshooting

10.1 First measures

1. Check all cabling.
2. Check all screw fittings.
3. Check whether air has gotten into the supply lines.
4. Check device for leaks.
5. Pay attention to system messages.

10.2 LAN

Go through the following steps, in case no connection between the computer and the devices can be established. Check after each step if the problem is solved. If the problem cannot be located, contact the Technical Support.

Steps

1. Check the status of the LAN connection in the Windows task bar:



Connected



Not connected

If no connection was established, test the following:

- Is the router switched on?
- Is the patch cable connected correctly to the router and the computer?

2. Check the router settings:

- Is the router set to DHCP server?
- Is the IP address range sufficient for all the connected devices?

3. Check all connections:

- Are the patch cable connected to the LAN ports and not the WAN port?
- Are all cable connections between devices and router correct?
- Are the cables plugged in tightly?

4. If the router is integrated into a company network, pull out the patch cable from the WAN port.

- Can the devices communicate with the computer, even though the router is disconnected from the company network?

5. Restart the system in the following order:

- Turn off all devices, router, and computer.
- Switch on the router and wait until its self-test is finished.
- Switch on the devices and the computer.

6. Replace the patch cable to the device with that no connection could be established.

7. Make sure that the IP port of the device matches the port in the chromatography software.

10.3 Possible problems and solutions



Note: If you have problems with the handling of a single device, please refer to the troubleshooting section of the respective instructions.

Device/system	Problem	Solution
SMB system	During the procedure of the sop start up, the eluent exits the wrong outlet.	<ul style="list-style-type: none"> ▪ Switch the valve position until the initial valve position and flush the system step by step according to SOP start up. ▪ Check the valve positions. ▪ Purge the pumps. ▪ Check the external check valves.
	Pump sucks air through the outlets (extract, raffinate, waste).	<ul style="list-style-type: none"> ▪ Check the valve position. ▪ Check the external check valves .
	Pumps sucks air through the inlet.	Check the filling level of the solvent supply, afterwards proceed SOP start-up.
	Air bubbles in the tubes.	Carefully degass and filter all solvents and feeds before use, afterwards proceed the SOP start up.
Leaksensor oven	Leaksensor shows constant Leaksignal (LED Out is orange).	<ul style="list-style-type: none"> ▪ Check for leakage. ▪ Check if the sensor has direct contact to the surface. ▪ Reduce the sensitivity of the leak-sensor by adjusting the ADJ screw counter-clockwise.
Oven	Actual temperature is over the set temperature.	Turn off the internal light of the oven, the light bulb heats fast up.
	Oven does not heat.	Check if the timer of the oven is set to 00:00 → turn off the timer.
Valve	Leakage of the valve.	Check the rotor seal of the valve according to technical note 801 and 703.

11. Maintenance and care

11.1 SOP: Cleaning procedure for pressure release valves and connected tubings for AZURA® Lab SMB



Note: This procedure is valid for AZURA® Lab SMB A29001 only.

11.1.1 Zone 1 pump & zone 2 pump

Follow this procedure to clean the pressure release valves and the connected tubings of zone 1 pump and zone 2 pump.

Proceed step 1 till 3 for each pump. Afterwards continue with step 4. Refer to the scheme (Fig. 13).

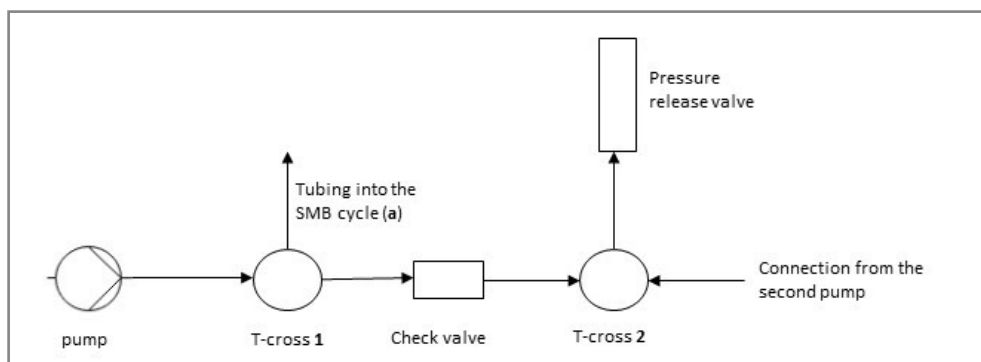


Fig. 16 Flow scheme of installed pressure release valves

Step	Task
1	Disconnect the tubing a on the t-cross 1 of each pump (see Fig. 13).
2	Connect the tubing a with a coupling and close the open side of the coupling (1/16") with a blind fitting.
3	Close the open position on the t-cross 1 with a blind fitting
4	Turn on zone 1 pump and set the flow rate to 16 % max. flow of the pump heads. The pressure rises up and the eluent exits the outlet tube of the pressure release valve.
5	Turn on zone 2 pump and set the flow rate to 8 % max. flow of the pump heads. The pressure rises up and the eluent exits the outlet tube of the pressure release valve.
6	Let the two pumps run for at least 5 min.
7	Turn off zone 2 pump.
8	Turn off zone 1 pump.
9	Rebuild all to the initial state.

11.1.2 Feed pump & zone 4 pump

Follow this procedure to clean the pressure release valves and the connected tubings of feed pump and zone 4 pump.

Proceed step 1 till 3 for each pump. Afterwards continue with step 4. Refer to the scheme (Fig. 13).

Step	Task
1	Disconnect the tubing a on the t-cross 1 of each pump (see Fig. 13).
2	Connect the tubing a with a coupling and close the open side of the coupling (1/16") with a blind fitting.
3	Close the open position on the t-cross 1 with a blind fitting
4	Turn on the feed pump and set the flow rate to 80 % max. flow of the pump heads. The pressure rises up and the eluent exits the outlet tube of the pressure release valve.
5	Turn on zone 4 pump and set the flow rate to 8 % max. flow of the pump heads. The pressure rises up and the eluent exits the outlet tube of the pressure release valve.
6	Let the two pumps run for at least 5 min.
7	Turn off zone 4 pump.
8	Turn off feed pump.
9	Rebuild all to the initial state.

11.2 SOP: Cleaning procedure for pressure release valves and connected tubings for AZURA® Pilot SMB

Follow this procedure to clean all pressure release valves and the connected tubings.

Step	Task
1	Turn on zone 1 pump and set the flow rate at 25 % of the pump heads max. flow.
2	Close the valve "eluent" and wait 5 min.
3	Open the valve "eluent".
4	Turn on zone 2 pump and set the flow rate at 15 % of the pump heads max. flow.
5	Close the valve "extract" and wait 5 min.
6	Open the valve "extract".
7	Turn on feed pump and set the flow rate at 25 % of the pump heads max. flow.
8	Close the valve "feed" and wait 5 min.
9	Open the valve "feed".
10	Turn on zone 4 pump and set the flow rate at 10 % of the pump heads max. flow
11	Close the valve "raffinate" and wait 5 min.
12	Open the valve "raffinate".

11.3 Maintaining the AZURA® Lab System

11.3.1 Pumps

Maintenance by customer (please refer to the AZURA Pump P 4.1S manual, document no. [V6870](#)):

- Replacing the pump head AHB40 (feed pump) or AHC20 (zone 1, 2 and 4 pump)
- Exchanging the ball valves A06841 (feed pump) or A06842 (zone 1, 2 and 4 pump)

Suggest maintenance interval pumps:

Please refer to the Service Manual of the pump (document no. [V6875A](#), available on Partner Area of KNAUER website, only for trained service technicians).

Spare parts for pump heads:

Please refer to the Service Manual for pump heads (document no. [VSM-001](#), available on Partner Area of KNAUER website).

11.3.2 VICI valves

No suggestion for preventive maintenance available.

Replacement parts:

Valve: M6035 (see Technical Notes 801 in the attachment)

Rotor Seal: A205121

Stator: A205122

External check valves: AZZ00FC

11.4 Maintaining the AZURA® Pilot system

11.4.1 Pumps

Maintenance by customer (please refer to the pump manual):

- Replacing the pump head A4029-1 (feed pump) or A4038-1 (zone 1, 2 and 4 pump)
- Exchanging the ball valves A1122 (feed pump) or A1080 (zone 1, 2 and 4 pump)

Suggest maintenance interval pumps:

Please refer to the Service Manual of the pump (document no. [V6875A](#), available on Partner Area of KNAUER website, only for trained service technicians).

Spare parts for pump heads:

Please refer to the Service Manual for pump heads (document no. [VSM-001](#), available on Partner Area of KNAUER website).

11.4.2 VICI valves

No suggestion for preventive maintenance available

Replacement parts:

Valve: M6036 (see Technical Notes 703 in the attachment)

Rotor Seal: A205123

External check valves: M1077-1

11.5 Decommissioning

Auxiliary material The system is designed for the use of different solvents. In case the system has not been used for several weeks, solvent residues may cause damage. We, therefore, recommend to:

- Flush the capillaries.
- Completely remove used solvents.
- Fill the capillaries with isopropanol.

Prerequisites The system has been flushed.

Hole plugs and/or cap fittings

Process

Steps

1. Unscrew the eluent supply lines and close the open connectors with hole plugs.
2. Disconnect the system.

Next step(s) Select a storage location according to the requirements (see chapter „12. Technical data“ on page 46).

11.6 Transport

Carefully prepare the device for transport. If you want to return your device to KNAUER for repairs, enclose the Service Request Form which can be downloaded from our website.

For a secure transport, note the weight and dimensions of the device (see chapter „Technical data“).

CAUTION

Bruising danger

Damage to the device by carrying or lifting it on protruding housing parts. The device may fall and thus cause injuries.

→ Lift the device only centrally on the side of the housing.

11.7 Storage

Pay attention that all tubes and capillaries have been emptied or filled with flushing solution (e. g. isopropanol) before storage. To prevent algae formation, do not use pure water. Close all inputs and outputs with cap fittings.



Note: Pay attention to the ambient conditions for storage (see chapter „Technical data“).

11.7.1 Disconnecting the power supply

Prerequisites The devices are switched off.

Process

Steps

1. Pull the power plug out of the socket and afterwards out of the devices.
2. Pack the power cable together with the devices.

Next step(s) Disconnect further electrical connections. Remove all accessories and pack the devices for transport or storage.

12. Technical data

12.1 General system parameters

Parameter	AZURA® SMB Lab	AZURA® SMB Pilot
Max. flow rate *	50 ml/min	500 ml/min
Recommended flow rate *	2-28 ml/min	2-200 ml/min
Max. delivery pressure *	1880 psi / 130 bar / 13 MPa	1450 psi / 100 bar / 10 MPa
Max. temperature	60 °C / 140 °F	60 °C / 140 °F
Max. number of columns	8	8
Column dimensions	up to 30 mm ID	up to 50 mm ID
Column configuration	2:2:2:2	2:2:2:2
	1:3:3:1	1:3:3:1
	1:1:1:1	1:1:1:1

* The maximum operating parameters for flow and pressure depend on the specific columns and customer application. Is not recommended to operate the system close to both maximum values for pressure and flow rate, due to increased wear of consumables.

12.2 Technical parameters

Parameter	AZURA® SMB Lab	AZURA® SMB Pilot
Supported software package	PurityChrom® MCC	PurityChrom® MCC
Ambient conditions	Temperature range 4-40 °C, 39.2-104 °F	Temperature range 4-40 °C, 39.2-104 °F
	Humidity: below 90 %, non-condensing	Humidity: below 90 %, non-condensing
Power supply	100-240 V; 50- 60 Hz; max. 100 W for ASM 2.1L	100-240 V; 50- 60 Hz; maximum 100 W for ASM 2.1L; max. 320 W for P 2.1L
Dimensions (Width × Height × Depth)	560 × 740 × 640 mm	950 × 800 × 660 mm
Weight	Approx. 56 kg	Approx. 160 kg

12.3 Wetted materials

Valid for AZURA® SMB Lab system (Art. Nr. A29000 and A29001) and AZURA® SMB Pilot system (Art. Nr. A29501).



Note: For chemical compatibility of wetted materials, see chapter 13 on page 48.

The components of the SMB system contain the following wetted parts:

Pumps

Material	Description
Stainless Steel	AISI 316
Titanium	Ti ₆ Al ₄ V
Ceramics	Al ₂ O ₃
Fluorinated rubber	FPM (FKM)
Graphite fiber reinforced PTFE	GFP55-27
Polyetheretherketone	PEEK
Ruby	Al ₂ O ₃
Sapphire	Al ₂ O ₃

Valves

Material	Description
Nitronic 60 (Stator) *	similar to 316 stainless steel
PAEK (Stator) **	Polyaryletherketone
Valcon E (Rotor)	PEEK/PTFE composite

*only for A29001 and A29501

**only for A29000

Capillaries & fittings

Material	Description
Stainless Steel*	AISI 316
PEEK**	Polyetheretherketone
Fluorinated Ethylene Propylene	FEP
Polyethylene terephthalate	PETP
Fluorinated rubber	FPM (FKM)
Polytetrafluoroethylene	PTFE

*only for A29001 and A29501

**only for A29000

13. Chemical compatibility of wetted materials



Note: The user is responsible for using fluids and chemicals in an appropriate and safe way. If there is any doubt, please contact the Technical Support.

13.1 General

The device is very resistant against a variety of commonly used eluents. However, make sure that no eluents or water come in contact with the device or enter into the device. Some organic solvents (such as chlorinated hydrocarbons, ether) may cause coating damage or loosen glued components by improper handling. Even small quantities of other substances, such as additives, modifiers, or salts can influence the durability of the materials. Exposure time and concentration have a high impact on the resistance.

The following list contains information about the chemical compatibility of all wetted materials which are used in devices made by KNAUER. The data bases on a literature research on the manufacturer specifications of the materials. The wetted materials of the present device are listed in chapter "Technical data".

All resistances listed here refer to an operation at temperatures up to 40 °C, unless stated otherwise. Note that higher temperatures may have a significant impact on the stability of several materials.

13.2 Plastics

Polyetheretherketone (PEEK)

PEEK is a durable and resistant plastic and, apart from stainless steel, the standard material in HPLC. It can be used at temperatures up to 100 °C and is highly chemical resistant against almost all commonly used solvents in a pH range of 1-12,5. PEEK is potentially moderate resistant against oxidizing and reducing solvents.

Therefore, following solvents should not be used: Concentrated and oxidizing acids (such as nitric acid solution, sulfuric acid), halogenated acids (such as hydrofluoric acid, hydrobromic acid) and gaseous halogens. Hydrochloric acid is approved for most applications.

In addition, following solvents can have a swelling effect and may have an impact on the functionality of the built-in components: Methylene chloride, THF and DMSO in any concentration such as acetonitrile in higher concentrations.

Polyethylene terephthalate (PET, outdated PETP)

PET is a thermoplastic and semi-crystalline material with high wear resistance. It is resistant against diluted acids, aliphatic and aromatic hydrocarbons, oils, fats and alcohols, but not against halogenated hydrocarbons and ketones. Since PET belongs chemically to esters, it is not compatible with inorganic acids, hot water and alkalis. Maximum operating Temperature: up to 120 °C.

Polyimide (Vespel®)

This material is wear-resistant and permanent resilient thermally (up to 200 °C) as well as mechanically. It is chemically broadly inert (pH range 1-10) and is especially resistant against acidic to neutral and organic solvents, but vulnerable to pH strong chemical or oxidizing environments: It is incompatible with concentrated mineral acids (such as sulfuric acid), glacial acetic acid, DMSO and THF. In addition, it will be disintegrated by nucleophilic substances like ammonia (such as ammonium salts under alkaline conditions) or acetate.

Ethylene-tetrafluorethylene copolymer (ETFC, Tefzel®)

This fluorinated polymer is highly resistant against neutral and alkaline solvents. Some chlorinated chemicals in connection with this material should be handled with care. Maximum operating Temperature is 80 °C.

Perfluorethylenpropylene copolymer (FEP), perfluoroalkoxy copolymer (PFA)

These fluorinated polymers hold similar features as PTFE, but with a lower operation temperature (up to 205 °C). PFA is suitable for ultrapure applications, FEP can be used universally. They are resistant against almost all organic and inorganic chemicals, except elemental fluorine under pressure or at high temperatures and fluorine-halogen compounds.

Systec AF™

This amorphous perfluorinated copolymer is inert against all commonly used solvents. However, it is soluble in perfluorinated solvents like Fluorinert® FC-75 and FC-40, and Fomblin perfluor-polyether solvents from Ausimont. In addition, it is affected by Freon® solvents.

Polychlorotrifluorethylene (PCTFE, Kel-F®)

The semi-crystalline thermoplastic material is plasticizer-free and dimensionally stable, even in a wide temperature range (-240 °C to +205 °C). It is moderately resistant against ether, halogenated solvents and toluene. Halogenated solvents over +60 °C and chlorine gas should not be used.

Fluorinated rubber (FKM)

The elastomer consisting of fluorinated hydrocarbon stands out due to a high resistance against mineral oils, synthetic hydraulic fluids, fuels, aromatics, and many organic solvents and chemicals. However, it is not compatible with strong alkaline solvents (pH > 13) like ammonia, and acidic solvents (pH value < 1), pyrrole and THF. Operating temperature: Between -40 °C and +200 °C.

Perfluorinated rubber (FFKM)

This perfluoro elastomer has a higher fluorine content as fluorinated rubber and is therefore chemically more resistant. It can be employed at higher temperatures (up to 275 °C). It is not compatible with Pyrrole.

13.3 Non-metals

Diamond-like carbon (DLC)

This material stands out due to its high hardness, low friction coefficient and thus minimum wear. In addition, it is highly biocompatible. DLC is inert against all acids, alkalis and solvents commonly used in HPLC.

Ceramic

Ceramic is resistant against corrosion and wear and is fully biocompatible. An incompatibility against acids, alkalis and solvents commonly used in HPLC is not known.

Aluminium oxide (Al₂O₃)

Due to their high resistance to wear and corrosion, aluminium oxide ceramics are used as a coating for mechanically stressed surfaces. They are a biocompatible material with low thermal conductivity and low thermal expansion.

Zirconium oxide (ZrO₂)

Zirconium oxide ceramics are characterized by their high mechanical resistance, which makes them particularly resistant to wear and corrosion. They are also biocompatible, have low thermal conductivity and are resistant to high pressures.

Sapphire

Synthetic sapphire is virtually pure monocrystalline aluminium oxide. It is biocompatible and very resistant to corrosion and wear. The material is characterized by a high hardness and a high thermal conductivity.

Ruby

Synthetic ruby is monocrystalline aluminium oxide and has a red coloration by the addition of some chromium oxide. It is biocompatible and very resistant to corrosion and wear. The material is characterized by a high hardness and a high thermal conductivity.

Mineral wool

This insulating material consists of glass or stone wool fibers and isolates in high oxidizing conditions and at high temperatures. Mineral wool is valid as commonly inert against organic solvents and acids.

Glass, glass fiber, quartz, quartz glass

These mineral materials are resistant against corrosion and wear and are mostly chemical inert. They are compatible with oils, fats and solvents and show a high resistance against acids and lyes up to pH values of 3–9. Concentrated acids (especially hydrofluoric acid) may embrittle and corrode the minerals. Lyes may ablate the surfaces slowly.

13.4 Metals

Stainless steel

Stainless steel is, apart from PEEK, the standard material in HPLC. Steels with WNr. 1.4404 (316L) are used, or a mixture with higher compatibility.

They are inert against almost all solvents. Exceptions are biological applications which are metal ion sensible, and applications with extreme corrosive conditions. These steels, in comparison to commonly used steels, are increasingly resistant against hydrochloric acid, cyanides and other halogen acids, chlorides and chlorinated solvents.

The application in ion chromatography is not recommended. In case of electrochemical applications, a passivation must be executed first.

Hastelloy®-C

This nickel-chrome-molybdenum alloy is extremely resistant to corrosion, especially against oxidizing, reducing and mixed solvents, even at high temperatures. This alloy may be used in combination with chlorine, formic acid, acetic acid and saline solutions.

Titanium, titanium alloy (TiA16V4)

Titanium has a low weight and a high hardness and stability. It stands out due to its very high chemical compatibility and biocompatibility. Titan is applied when neither stainless steel nor PEEK are usable.

14. Legal information

14.1 Transport damage

The packaging of our devices provides the best possible protection against transport damage. Check the devices for signs of transport damage. In case you notice damages, contact the Technical Support and the forwarder company within three workdays.

14.2 Warranty conditions

The factory warranty for the device is stipulated by contract. During the warranty period, any components with material or design-related defects will be replaced or repaired by the manufacturer free of charge. Please connect to our website for further information on terms and conditions.

All warranty claims shall expire in the event that any unauthorized changes are made to the device. This warranty also excludes the following:

- accidental or willful damage
- damage or errors caused by third parties that are not contractually related to the manufacturer at the time the damage occurs
- wear parts, fuses, glass parts, columns, light sources, cuvettes and other optical components
- damage caused by negligence or improper operation of the device and damage caused by clogged capillary
- packaging and transport damage

In the event of device malfunctions, directly contact the manufacturer.

KNAUER Wissenschaftliche Geräte GmbH
Hegauer Weg 38
14163 Berlin, Germany

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Fax: +49 30 8015010
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Internet: www.knauer.net

14.3 Warranty seal

A warranty seal is attached on some devices. The warranty seal is color-coded. A blue seal is used by the assembly or technical support of KNAUER for devices to be sold. After repair, service technicians stick an orange seal in identical position. If unauthorized persons interfere with the device or the seal is damaged, the warranty claim becomes void.



14.4 Declaration of Conformity

The Declaration of Conformity accompanies the product as a separate document and is available online:

<https://www.knauer.net/de/Support/Declarations-of-conformity>

14.5 Disposal

Hand in old devices or disassembled old components at a certified waste facility, where they will be disposed of properly.

AVV marking in Germany According to the German „Abfallverzeichnisverordnung“ (AVV) (January, 2001), old devices manufactured by KNAUER are marked as waste electrical and electronic equipment: 160214.

WEEE registration KNAUER as a company is registered by the WEEE number DE 34642789 in the German „Elektroaltgerätereister“ (EAR). The number belongs to category 8 and 9, which, among others, comprise laboratory equipment.

All distributors and importers are responsible for the disposal of old devices, as defined by the WEEE directive. End-users can send their old devices manufactured by KNAUER back to the distributor, the importer, or the company free of charge, but would be charged for the disposal.

Solvents and other operating materials All solvents and other operating materials must be collected separately and disposed of properly.

All wetted components of a device, e. g. flow cells of detectors or pump heads and pressure sensors for pumps, have to be flushed first with isopropanol and then with water before being maintained, disassembled or disposed.

15. Checklist

Transportation and storage

- There must be enough space available for storing the shipping pallet.

HPLC System set-up

- A lab table with adequate carrying capacity and dimensions is available.
- The power supply and cable are available.
- The power supply and cable comply with the requirements.

Ambient conditions at installation site

The installation site complies with the requirements with respect to:

- equipment
- temperature
- humidity
- vibration
- high frequency emissions

Computer and Operating System

- The computer complies with the requirements or is delivered by KNAUER.

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Science Together



Latest KNAUER instructions online:
<https://www.knauer.net/en/Support/User-manuals>

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