

Operating Manual

multi N/C 3100 (duo, pharma)
TOC/TNb Analyzers



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For a proper and safe use of this product follow the instructions. Keep the operating manual for future reference.

General Information <http://www.analytik-jena.com>

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1 Basic information

1.1 About this user manual

Content

The operating manual describes the following device model(s):

- multi N/C 3100
- multi N/C 3100 duo
- multi N/C 3100 pharma

In this manual, these models are collectively referred to as the multi N/C 3100. Any differences between the models are explained in the relevant section.

The device is intended to be operated by qualified specialist personnel under observance of the operating manual.

The operating manual provides information about the design and operation of the device and provides operating personnel with the necessary know-how for safe handling of the device and its components. Furthermore, the operating manual includes information on the maintenance and servicing of the device as well as information on potential causes of malfunctions and their correction.

The multi N/C 3100 duo modular measuring system enables automated analysis of liquid and solid samples. The layout, installation and operation of the modular measuring system are described in the user manual for the HT 1300 solids module. Pay particular attention to the information on switching between liquid and solid operation given there.

The multi N/C 3100 pharma model is a special model for the pharmaceutical industry. No solids modules and no ChD detectors are available for the pharma model.

Conventions

Instructions for actions occurring in chronological order are numbered and combined into action units.

Warnings are indicated by a warning triangle and a signal word. The type, source and consequences of the hazard are stated together with notes on preventing the hazard.

Elements of the control and analysis program are indicated as follows:

- Program terms are in bold (e.g., the **System** menu).
- Menu items are separated by vertical lines (e.g., **System | Device**).

Symbols and signal words used in this manual

The user manual uses the following symbols and signal words to indicate hazards or instructions. These warnings are always placed before an action.



WARNING

Indicates a potentially hazardous situation which can cause death or very serious (possibly permanent) injury.



CAUTION

Indicates a potentially hazardous situation which can cause slight or minor injuries.



NOTICE

Provides information on potential material or environmental damage.

1.2 Analyzer area of application

- multi N/C 3100 pharma: Special model for use in the pharmaceutical, medical and biotechnology industries

This model was developed especially for TOC and TN_b detection for ultrapure water. It is suitable, for example, for use in cleaning validation, as well as for the analysis of water for injection purposes. Extractable organic compounds in pharmaceutical plastic packaging can also be summarily analyzed. The control and analysis software provides complete data integrity and conforms to the pharmaceutical guidelines in 21 CFR Part 11 and EudraLex Volume 4 Annex 11.

- Use in water treatment

The device can be used for both drinking water and waste water analysis in communal and industrial treatment systems. Complex water bodies containing particles or salt can also be safely analyzed.

- Use in environmental monitoring

Surface waters such as seawater often have low TOC content with high TIC concentrations and high salinity. These difficult samples can be analyzed thanks to special analysis modes (NPOC plus).

- Use in power plants and laboratories

With its dynamic measurement range, the analyzer provides TOC detection for power plants and in industrial steam production.

- Analysis of waste and soil samples

Carbon detection (TC/TOC detection) in solid samples is possible by adding a solids module to the device. Additionally, eluates can be analyzed. In these and other liquid samples, TC and TN_b can be determined simultaneously.

- Use in research and education

Due to its many configuration options, the analyzer is suitable for research and education. TC and TOC can be determined in solids in conjunction with a solids module.

1.3 Intended use

The device and its components may only be used for the analyses listed in the user manual. Only this specified use is regarded as the intended use, ensuring the safety of the user and the device.

The analyzer may only be used to determine the total carbon content and the concentration of organic and inorganic bound carbon in aqueous samples.

The analyzer is particularly suited for detection of the listed parameters in drinking water, ground water, surface water, ultrapure water and water for pharmaceutical purposes.

When equipped with a nitrogen detector, the analyzer can be used to examine the nitrogen content in aqueous samples.

In conjunction with an optional solids module, the total carbon content in solids can be determined.

No flammable liquids or substances that can form explosive mixtures may be analyzed with the analyzer. Do not analyze concentrated acids with the analyzer!

Only use the following carrier gases with the device: Oxygen, synthetic air or purified compressed air.

2 Security

For your own safety and to ensure error-free and safe operation of the device, please read this chapter carefully before commissioning.

Observe all safety instructions listed in this user manual and all messages and information displayed on the monitor by the control and analysis software.

2.1 Safety labeling on the device

Warning and mandatory action labels have been attached to the device and must always be observed.

Damaged or missing warning and mandatory action labels can cause incorrect actions leading to personal injury or material damage. The labels must not be removed. Damaged warning and mandatory action labels must be replaced immediately!

The following warning and mandatory action labels have been attached to the device:

Warning symbol	Meaning	Comment
	Warning against hot surface	- On the furnace, on the furnace cover: - On the left side wall: Risk of burns from the hot furnace
	Warning against corrosive substances	On the front side, next to the phosphoric acid bottle: Warning against phosphoric acid
	Warning against harmful or irritating substances	On the front side: Warning against phosphoric acid
	Warning against crushing	On the autosampler: There is a risk of injury in the movement range of the autosampler.

Hazardous substances are used during operation:

GHS labeling	Meaning	Comment
	Corrosivity warning	On the phosphoric acid bottle: Phosphoric acid is corrosive
Mandatory action labels/information symbols	Meaning	Comment
	Disconnect the power supply before opening the device cover.	On the side parts and the rear of the device: Before opening the device cover, switch off the device and disconnect the mains plug from the mains socket.

Mandatory action labels/information symbols	Meaning	Comment
	Observe the operating manual	On the side parts and the rear of the device: Before starting work, read the user manual.
	For People's Republic of China only	The device contains controlled substances. Analytik Jena GmbH+Co. KG warrants that these substances will not be released from the device within the next 25 years provided the device is used as intended.

2.2 Requirements for the operating personnel

The device must only be operated by qualified specialist personnel instructed in the use of the device. This instruction also include teaching the contents of this user manual and of the user manuals of the connected system components. We recommend training by qualified employees of Analytik Jena or its representatives.

In addition to the safety instructions in this user manual, the general applicable safety and accident prevention regulations of the respective country the device is operated in must be observed and adhered to. The operator must ensure the latest version of these regulations.

The user manual must be accessible to the operating and service personnel.

2.3 Safety instructions, transport and commissioning

Incorrect installation can create serious hazards. This may result in electric shock and explosion if the gases are not connected correctly.

- Only the Analytik Jena customer service or specialist personnel trained and authorized by them is allowed to install and commission the device and its system components.
- Unauthorized assembly and installation is not permitted.

Insufficiently secured components pose a risk of injury.

- During transport, secure the device components as specified in these operating instructions.
- Loose parts must be removed from the system components and packed separately.

To prevent health damage, the following must be observed when moving the device in the laboratory (lifting and carrying):

- For safety reasons, two persons are required to transport the device who must hold the unit by either side of the equipment.
- The device does not have any carrying handles. Therefore the device must be gripped firmly with both hands at the lower end.
- Risk of damage to health due to improper decontamination! Perform a professional and documented decontamination of the device before returning it to Analytik Jena. The decontamination report is available from Service when registering the return. Without a completed decontamination report, the acceptance of the device will be refused. The sender may be liable for damage caused by inadequate decontamination of the device.

2.4 Safety instructions: during operation

2.4.1 Summary of safety instructions

The operator must make sure that the device and its safety equipment is in sound condition each time before starting up the device. This applies in particular after each modification or extension of the device or its repair.

Observe the following:

- The device may only be operated if all items of protective equipment (e.g. covers in front of electronic components) are in place, properly installed and fully operational.
- The sound condition of the protection and safety equipment must be checked regularly. Any defects must be corrected as soon as they occur.
- Protective and safety equipment must never be removed, modified or switched off during operation.
- Always ensure free access to the main switch and to the emergency shutdown switches and locks during operation.
- The ventilation equipment on the device must be in good working condition. Covered ventilation grilles or slots etc. may cause the device to break down or may cause damage to it.
- Modifications, conversions and extensions to the device are only permitted after consultation with Analytik Jena. Unauthorized modifications can jeopardize the device's operational safety and may lead to limitations regarding the warranty and access to customer service.
- Keep all combustible materials away from the device.
- The furnace operates at temperatures of 700 to 950 °C. Do not touch the hot components (furnace, condensation coil) during or directly after operation.
- Caution when handling glass components. Risk of broken glass and therefore risk of injury!
- Ensure that no liquid enters the interior of the device, for example at cable connections. There is a danger of electric shock.
- There is a risk of injury in the movement range of the autosampler. For example, hands or fingers may be crushed. Maintain a safety distance from the autosampler during operation.
- The optional chemiluminescence detector (CLD) contains an ozone generator which produces ozone (O₃). When used in accordance with the intended use, the downstream ozone decomposer destroys the toxic gas. Various safety measures result in the automatic shut-down of the ozone generator. However, the following still applies: if you notice a strong smell of ozone, switch the device off immediately and inform customer service. In order to guarantee perfect and safe operation, Analytik Jena recommends an annual inspection and maintenance of the detector by customer service.

2.4.2 Safety instructions – protection against explosion and fire

The device may not be operated in an explosive environment.

Smoking or handling open flames are prohibited in the room in which the device is operated!

2.4.3 Electrical system safety instructions

Life-threatening electrical voltages occur in the device in the area of the right side component! Contact with live components may cause death, serious injury or painful electrical shock.

- The power plug must be connected to a proper power outlet to ensure that the device meets protection class I (ground connector). The device may only be connected to power sources whose nominal voltage is the same as that on the rating plate of the equipment. Do not replace the removable power cable of the device with a power cable that does not meet the specifications (with no protective ground conductor). Extensions of the supply cable are not permitted!
- Work on the electronics may only be carried out by the customer service of Analytik Jena and specially authorized technicians.
- The electrical components must be checked regularly by a qualified electrician. Any defects such as loose connections or faulty or damaged cables must be repaired without delay.
- Before opening the device, the device must be switched off via the main switch and the power plug must be disconnected from the power outlet!
- The basic module and the system components may only be connected to the mains when they are switched off.
- Electrical connection cables between the basic module and the system components may only be connected or disconnected when the device is switched off.
- Switch off the analyzer immediately using the main switch on the rear of the housing if there is any malfunction of the electrical components. Disconnect the power plug from the power socket.

2.4.4 Safety instructions for the operation of compressed gas containers and compressed gas systems

- The operating gases are taken from compressed gas containers or local compressed gas systems. The operating gases must have the required purity.
- Work on compressed gas containers and systems may only be carried out by individuals with specialist knowledge and experience in compressed gas systems.
- Compressed air hoses and pressure reducers may only be used for the assigned gases.
- Pipes, hoses, screw connections and pressure reducers for oxygen must be kept free from grease.
- Check all pipes, hoses and screw connections regularly for leaks and externally visible damage. Repair leaks and damage without delay.
- Shut off the gas supply to the device prior to any maintenance and repair work on the compressed gas containers.
- After successful repair and maintenance of the components of the compressed gas containers or system, the device must be checked for proper operation prior to recommissioning.
- Unauthorized assembly and installation are not permitted!

2.4.5 Handling of auxiliary and operating materials

The operator is responsible for the selection of substances used in the process as well as for their safe handling. This is particularly important for radioactive, infectious, poisonous, corrosive, combustible, explosive and otherwise dangerous substances.

When handling hazardous substances, the locally applicable safety instructions and instructions in the safety data sheets from the manufacturers of the auxiliary and operating materials must be complied with.

- Special care must be taken when handling concentrated acids. The regulations and notes in the safety data sheets for the handling of orthophosphoric acid (H_3PO_4) or hydrochloric acid (HCl) must be observed.
- The catalyst supplied by the manufacturer should be handled with the usual caution when handling chemicals.

Observe the following instructions when working with quartz wool:

- Only store quartz wool in closed containers.
- When working with quartz wool, avoid the formation of dust! Inhaled dust may cause irritation to respiratory pathways.
- Wear personal protective equipment (laboratory coat, protective gloves, safety goggles, respiratory mask) when replacing the quartz wool and when cleaning the combustion tube.
- Collect used quartz wool in suitable, sealed containers and dispose of the material in accordance with applicable legal regulations. Contact the responsible waste disposal company to organize the disposal of the waste.

Observe the following:

- The operator is responsible for carrying out suitable decontamination should the device become contaminated externally or internally with dangerous substances.
- Splashes, drops or larger liquid spillages should be removed using an absorbent material such as cotton wool, laboratory wipes or cellulose.
- For biological contamination, wipe the affected area with a suitable disinfectant, such as an Incidin Plus solution. Then wipe the cleaned areas so that they are dry.
- The only suitable cleaning method for the housing is wipe disinfection. If the disinfectant has a spray nozzle, apply disinfectant to a suitable cloth before using it on the device.
Work particularly carefully and cleanly with infectious material because the device cannot be decontaminated as a whole.
- Before using a cleaning or decontamination procedure other than that prescribed by the manufacturer, the user is required to check with the manufacturer that the intended procedure will not damage the device. Safety labels attached to the device must not have methanol applied.

2.4.6 Safety instructions – maintenance and repair

The device is generally maintained by the customer service department of Analytik Jena or specialist personnel trained and authorized by them.

Unauthorized maintenance can damage the device. For this reason, only the activities described in the user manual in the "Maintenance and care" chapter may be performed by the operator.

- Only clean the exterior of the device with a slightly moistened, non-dripping cloth. Use only water and, if required, customary surfactants.
- All maintenance and repair work on the device must only be carried out when the device is switched off (unless specified otherwise).
- The gas supply must be shut off before performing any maintenance or repair work (unless specified otherwise).
- Allow the device to cool down before any maintenance work or replacement of system components.
- Use only original spare parts, wear parts and consumables. They have been tested and ensure safe operation. Glass parts are wear parts and are not subject to the warranty.
- All protective equipment must be reinstalled and checked for proper function when the maintenance or repair work is complete.

See also

 Maintenance and care [► 86]

2.5 Behavior during emergencies

- If there is no immediate risk of injury, switch off the device and the connected system components immediately in hazardous situations or in the event of an accident and/or disconnect the power plugs from the power outlets.
- Close the gas supply as soon as possible after switching off the devices.

3 Function and design

3.1 Layout

The analyzer is a compact table-top device with permanently installed main components. Further accessories and reagents are required for the measurement process.

Control of the analyzer and analysis of the measurement data is performed via the multiWin software.

All components of the analyzer operated or serviced by the user can be accessed via the two doors on the front, the left-hand removable side wall or the top cover.

The analyzer consists of the following main components:

- Sample supply system
- Gas box and hose system
- Combustion system
- Measuring gas drying and cleaning
- Detector
- Indicator and control elements, connections
- Electronics
- Accessories

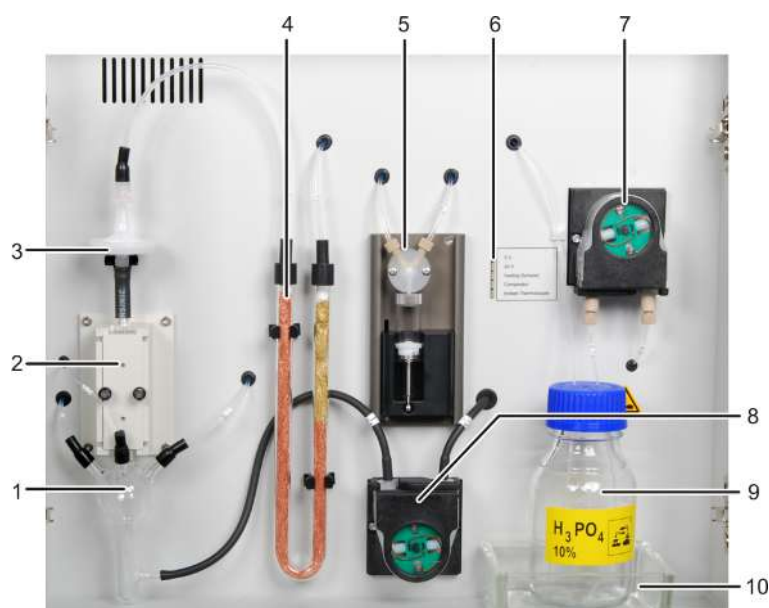


Fig. 1 Analyzer, front doors opened

- | | |
|--------------------------------------|-------------------|
| 1 TIC condensate container | 2 Cooling block |
| 3 Water traps | 4 Halogen trap |
| 5 Syringe pump with 2-port valve | 6 LED displays |
| 7 Phosphoric acid pump | 8 Condensate pump |
| 9 Reagent bottle for phosphoric acid | 10 Drip tray |

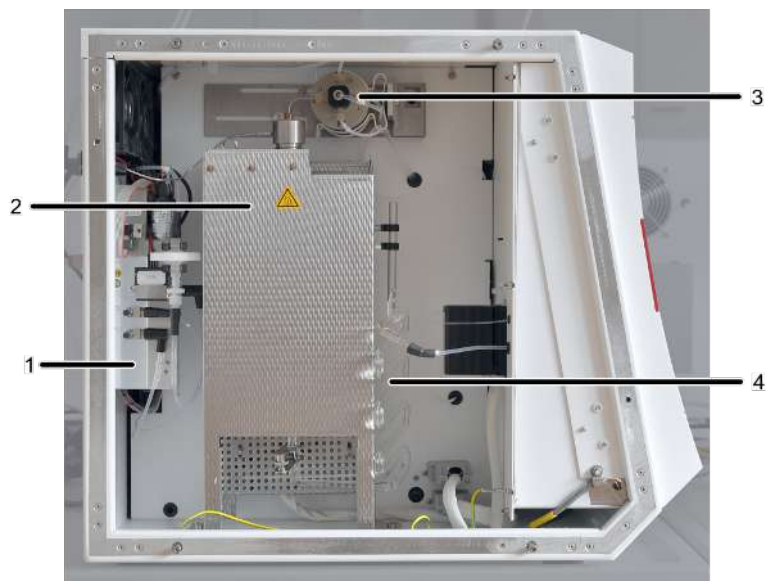


Fig. 2 Analyzer, left side wall opened

- | | |
|---------------|---------------------|
| 1 Gas box | 2 Combustion system |
| 3 5-way valve | 4 Condensation coil |

3.1.1 Sample supply system

Sample supply is carried out as flow injection via syringe pump with 2-port valve. The injection volume is 100 to 1000 μl .

The hose connections are attached to the 2-port valve using Fingertight screw connections. The syringe body is made of glass and replaceable.

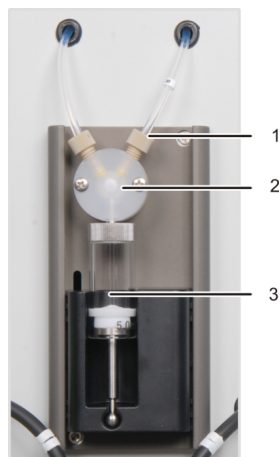


Fig. 3 Syringe pump

- | | |
|--------------------------|----------------|
| 1 Fingertight connection | 2 2-port valve |
| 3 Dosing syringe | |

The hoses on the 2-port valve are connected to the following components:

- Hose 8 to the ultrapure water bottle
- Hose AB to the change-over valve

3.1.2 Hose system

Hose diagram

The connection between the individual components is made with labeled hoses. The numbers and letters circled in the hose diagram correspond to the labels on the hoses in the analyzer.

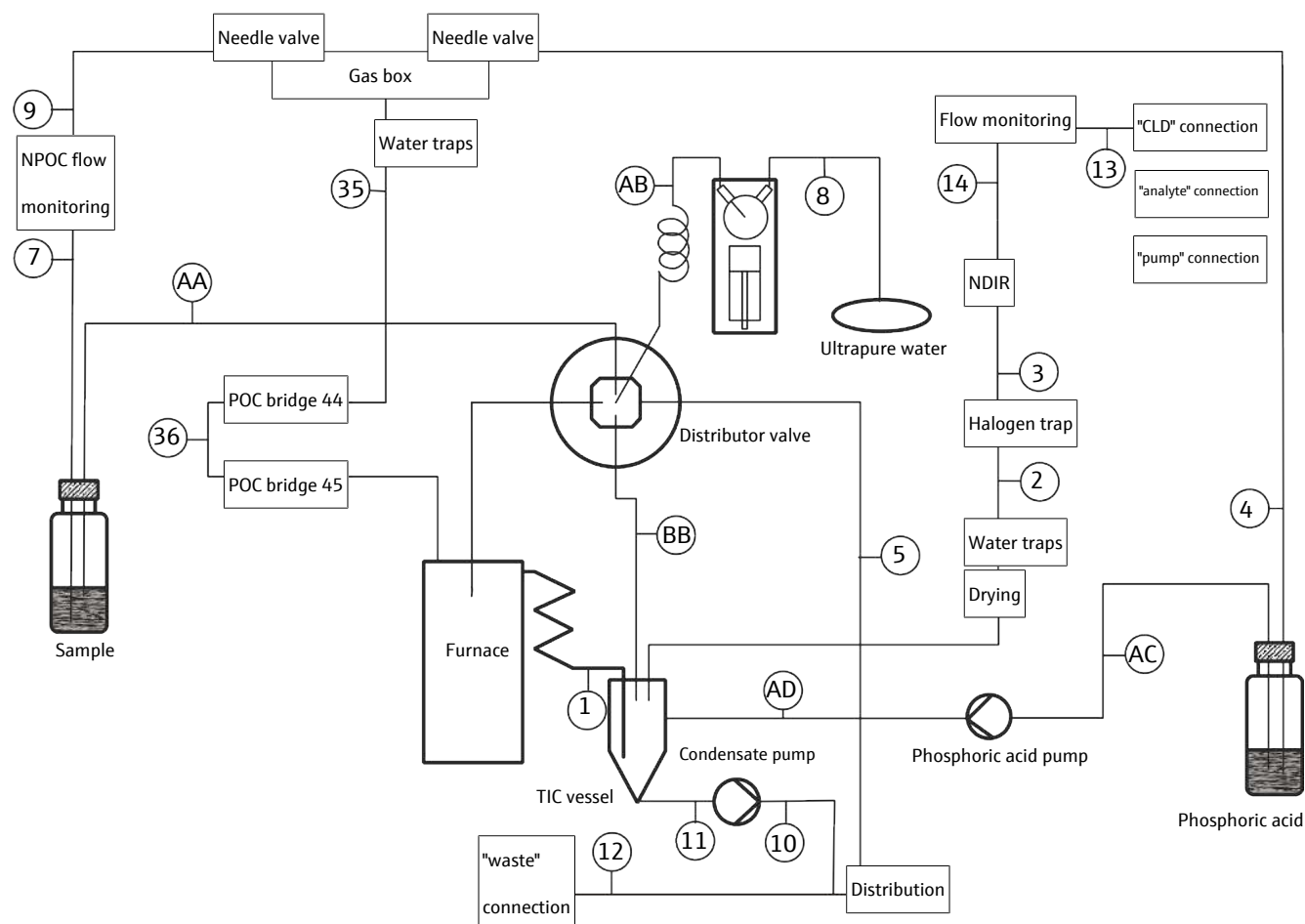


Fig. 4 Hose diagram

Components for flow adjustment

The analyzer automatically sets the carrier gas flow and controls the inlet flow via an MFC (Mass Flow Controller). An MFM (Mass Flow Meter) measures the carrier gas flow at the device outlet. This automatically checks for leaks. The results are displayed in the multiWin software in the **System state** window. A water trap protects the gas box from the return of wet combustion gases.

The NPOC purge flow can be set via the needle valve on the gas box. The needle valve can only be accessed when the left side wall has been removed. The NPOC purge flow is measured with an MFM and displayed in the **System state** window.

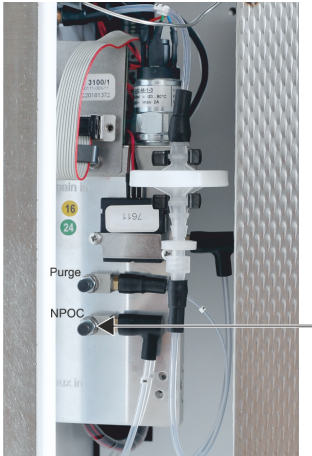


Fig. 5 Setting the NPOC purge flow

Condensate pump

The condensate pump pumps the condensate or the waste solution from TIC determination out automatically after each measurement. The condensate pump is located behind the front doors next to the halogen trap.



Fig. 6 Condensate pump

Phosphoric acid pump

The phosphoric acid pump transports phosphoric acid (10 %) to the TIC condensate container. During this process, the phosphoric acid is permanently outgassed.



Fig. 7 Phosphoric acid pump

Connection method

Inside the device, most gas connections have been implemented via FAST connectors (FAST – Fast, Safe, Tight). These connectors provide a tight transition between the hoses and connections with different diameters. The soft sleeves prevent the risk of glass breakage in comparison to rigid screw connections. There are different connector versions.



Fig. 8 FAST connector

So-called Fingertight screw connections are also used. These flangeless fittings consist of a conical nipple and a banjo bolt. These hose connections seal purely by tightening the plastic banjo bolt finger-tight.

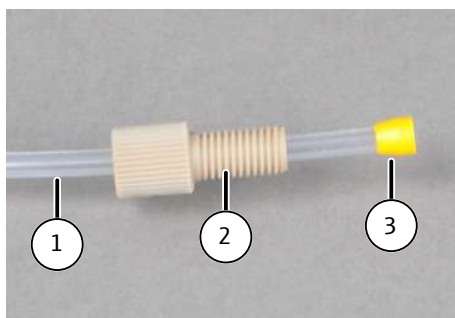


Fig. 9 Fingertight screw connection

1 Hose

2 Banjo bolt

3 Conical nipple

3.1.3 Combustion system

The combustion system is behind the left side wall of the analyzer.

The combustion furnace is a resistor-heated vertical furnace for digestion temperatures of up to 950 °C.

The combustion tube (reactor) consists of quartz glass. It is filled with catalyst and auxiliary material. If the effectiveness of the catalyst decreases, the combustion tube must be filled again.

The furnace head is fitted to the top opening of the combustion tube. At the bottom end, the combustion tube is connected to the condensation coil via a fork clamp.

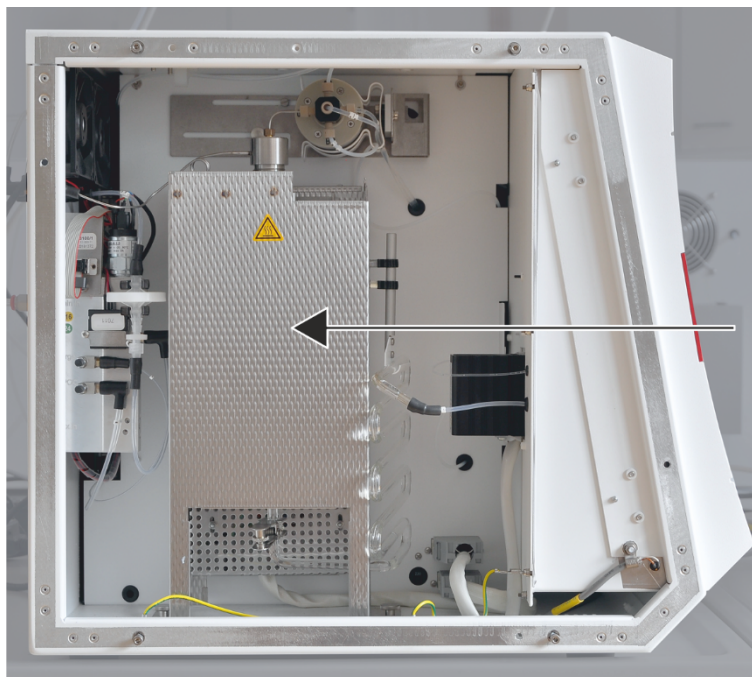


Fig. 10 Combustion furnace

3.1.4 Measuring gas drying and cleaning

Condensation coil

The glass condensation coil is located to the right of the furnace and is attached to the outlet of the combustion tube.

The condensation coil quickly cools the measuring gas. The water vapor contained in the measuring gas condenses. The measuring gas and water mixture is routed to the TIC condensate container via a hose.



Fig. 11 Condensation coil

1 Condensation coil

2 Hose 1 to the TIC condensate container

TIC condensation module

The TIC condensation module consists of a TIC condensate container and a cooling block. The TIC reactor and the gas/liquid separator are combined in the TIC condensate container. The cooling block dries the measuring gas at the same time.

The TIC condensation module is located on the left front side. The measuring gas/water mixture is supplied via the top left connection via hose 1.

The top middle connection on the glass container is connected to the phosphoric acid pump. The phosphoric acid pump pumps phosphoric acid (10 %) into the TIC reactor for each TIC determination.

The cooling block dries the measuring gas by freezing out the water vapor. The dry measuring gas is routed via the top connection out of the TIC condensate container. The measuring gas drying is maintenance-free.

The condensate pump pumps the condensate or waste solution from the TIC determination out via the bottom side outlet on the glass container after each measurement

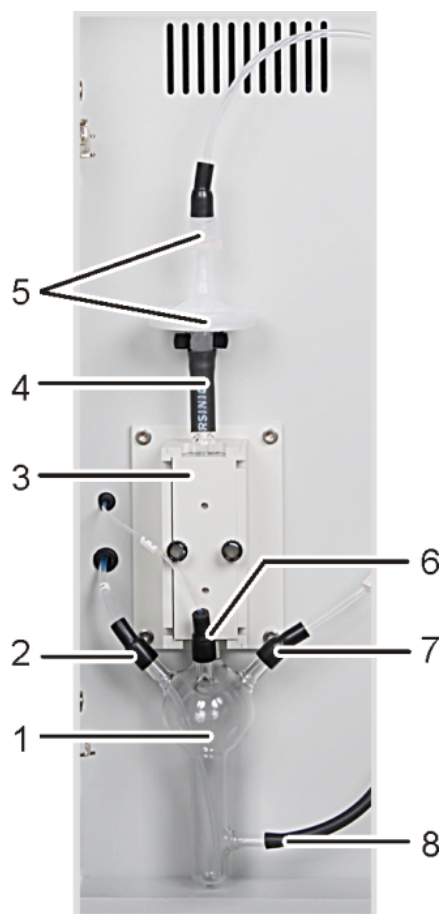
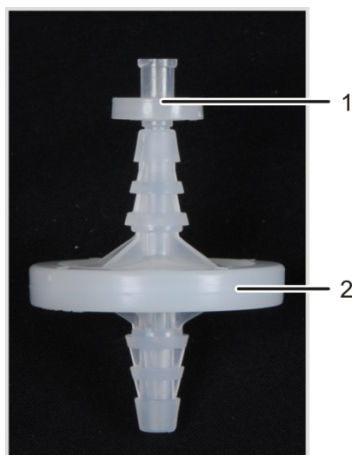


Fig. 12 TIC condensate module

- | | |
|---|--|
| 1 TIC condensate container | 2 Connection for hose 1/condensation coil measuring gas feed |
| 3 Cooling block | 4 Connection to the water traps |
| 5 Water traps | 6 Connection for hose AD/phosphoric acid feed |
| 7 Connection for hose BB/direct sample feed for TIC detection | 8 Connection to condensate pump (waste hose 11) |

Water traps

The water traps remove interfering components from the measuring gas and protect the detector and the gas box. The water traps are mounted in the gas path behind the cooling block or behind the gas box. The water traps each consist of a larger and a smaller water trap. The larger water trap (TC prefilter) retains aerosols during operation. The smaller water trap (disposable retention filter) retains rising water.

**Fig. 13 Water traps**

1 Disposable retention filter

2 TC prefilter

Halogen trap

The halogen trap removes interfering components (halogens, halogen-hydrogen compounds) from the measuring gas. It also protects the detectors and the flowmeter in this manner. The halogen trap is installed in the gas path behind the TIC condensate container and the water traps.

The halogen trap consists of a U-shaped tube. It is filled with special copper wool and brass wool. The filling of the halogen trap has to be replaced once half of the copper wool has changed color to black or when the brass wool has changed color at the latest.

**Fig. 14 Halogen trap**

3.1.5 Detection

NDIR detector

The NDIR detector (non-dispersive infrared absorption detector) is behind the right side wall of the analyzer.

Gases with molecules from different atoms have specific absorption bands in the infrared wavelength range. When a light beam is sent through an arrangement of cells which contains IR-active gases, these gas components absorb a proportional share of the total radiation on their characteristic wavelengths according to their concentration in the gas mixture.

The radiation receiver used in the NDIR detector is selective for CO₂.

Measurements using the VITA method

The CO₂ molecules are detected metrologically as long as they remain in the cell of the NDIR detector. The measuring gas flow can fluctuate during CO₂ measurement, because, for example, liquid samples evaporate or condense during dosing. For this reason, the CO₂ molecules are sometimes detected spectrometrically for a longer time (at lower gas flows) or a shorter time (at higher gas flows).

The VITA method is formally the residence-time-coupled integration for TOC analyses. The measuring gas flow is determined in parallel with the NDIR signal in the VITA method. The NDIR signal is normalized via computer control. This compensates for occurring flow fluctuations, ensuring constant gas flow. Integration is only carried out after this.

A highly precise digital flowmeter detects the gas flow in the immediate area of the NDIR detector.

Electrochemical NO detector (ChD, optional)

For TN_b detection, the electrochemical NO detector can be used. The NO detector is behind the right side wall of the analyzer. It analyzes the nitrogen oxide (NO) content in the measuring gas.

After thermal oxidation of the sample, the measuring gas enters the detector. In the detector, the nitrogen oxides diffuse via a highly selective membrane in the electrochemical measuring cell.

The nitrogen oxides are oxidized at the anode. This alters the current flow between the electrodes in proportion to the concentration of nitrogen oxide. The change of the current flow is evaluated as a signal and the nitrogen content of the analyzed sample is determined from this. The electrolyte in the measuring cell only serves as catalyst and is not used up.

A supply voltage is required for the operation of the electrochemical NO detector (ChD). Even if the analyzer is switched off, a support voltage must maintain the electrochemical equilibrium in the ChD. A battery (U9VL) is installed in the right side part of the analyzer for this.

The optional ChD is not provided for the multi N/C 3100 pharma model.

Chemiluminescence detector CLD (optional)

Optional addition of a chemiluminescence detector to the analyzer enables TN_b determination. The CLD must be positioned next to the analyzer as an external device.

The measuring gas formed by the thermal oxidation of the sample is dried and then enters the reaction chamber of the chemiluminescence detector. There, the nitrogen monoxide present in the measuring gas is oxidized with ozone into activated nitrogen dioxide. Emission of light photons (luminescence) returns the molecules of the nitrogen dioxide to their original state. The luminescence is recorded. The signal is proportional to the nitrogen monoxide concentration. The total nitrogen content of the sample can be determined in this manner.

Sample digestion for TN_o detection cannot result in 100 % NO recovery. While the combustion gases are cooled and condensed, nitrogen oxides also form at higher oxidation levels.

3.1.6 Indicator and control elements, connections

LED display

A green LED is installed on the left door of the analyzer. The LED is lit when the analyzer is switched on, indicating operational readiness.



Fig. 15 Status LED

The LED strip behind the right door indicates different operating states of the analyzer.

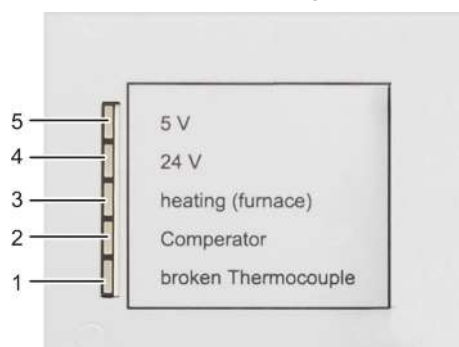


Fig. 16 LED strip (right door open)

- | | |
|---|---|
| 1 Broken thermocouple (is lit when a thermocouple is defective) | 2 Furnace comperator (is lit at excessive temperatures) |
| 3 Furnace heating on/off | 4 Device voltage |
| 5 Voltage of the internal firmware controller | |

Main switch and connections

The main switch and the following connections are located on the rear of the analyzer:

- Mains power connection with device fuse
- Media connections for gases and waste
- Interfaces for PC and accessory connection

A diagram in the center details the different connections.



Fig. 17 Device rear

- | | |
|---|---|
| 1 "Power switch" main switch | 2 "FUSE" mains fuse holder |
| 3 "Main plug" mains connection | 4 "analyte" gas connection |
| 5 "pump" gas connection | 6 "CLD" gas connection |
| 7 Bridge for the gas connection of the POC module | 8 "O ₂ /Air" carrier gas connection |
| 9 POC module connection (optional) | 10 Connection of the neutral conductor on the autosampler |
| 11 "waste" connection | 12 RS 232 interface for "sampler" autosampler |
| 13 RS 232 interface for "CLD/HT" CLD and solids modules | 14 USB 2.0 "PC" interface |

Type plate

The type plate is attached to the device rear.

The type plate contains the following information:

- manufacturer address, trademark
- Designation of the device, serial number
- Electrical connection data
- Conformity markings
- WEEE marking

3.1.7 Accessories

The following accessories are required for measurements with the analyzer:

- Connection cables, connection hoses
- Suitable waste container or drainage
- Reagent bottle with drip tray for phosphoric acid (250 ml)
- Ultrapure water bottle (2.5 l)

The reagent bottle must be positioned in the drip tray behind the right door. The reagent bottle is labeled with a safety symbol and the name of the contents and must be filled with phosphoric acid (10 %) by the user.

3.2 Additional options for the analyzer

Autosampler	The following autosamplers are available for the analyzer: ■ AS vario with various tray sizes ■ AS vario ER with various tray sizes and canula flushing ■ AS 10e for 10 samples ■ AS 21hp for 21 samples ■ POC Sampler for POC measurement ■ EPA Sampler with piercing function
POC module	The addition of a POC module to the analyzer enables direct detection of POC in aqueous samples.
External solids module	The addition of the external HT 1300 solids module to the analyzer enables the catalyst-free digestion of solid samples at temperatures of up to 1300 °C in the ceramic combustion furnace. The ceramic boats allow input of large sample sizes (up to 3000 mg). This can compensate for sample inhomogeneities.
Manual TIC solids module	The TIC determination in solid samples can be performed by equipping the analyzer with a TIC solids module. Large sample amounts can be weighed in an Erlenmeyer flask. The sample is acidified and magnetically stirred on a heating plate to digest carbonates and hydrogen carbonates to CO₂. No solids modules are available for the multi N/C 3100 pharma model.

3.3 Function and measuring principle

The analyzer is a compact, high-performance device for determination of the content of organic bound carbon and/or the total nitrogen content in aqueous samples.

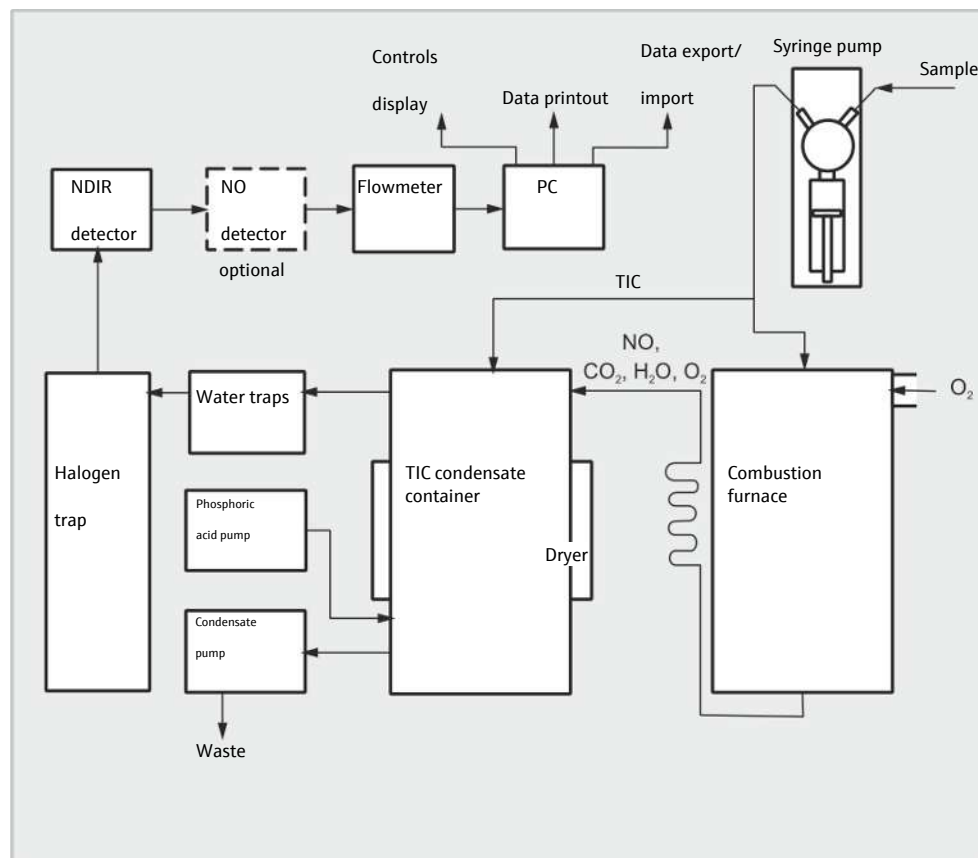
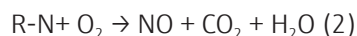


Fig. 18 Principle of operation

The samples are digested at high temperatures in the presence of special catalysts. This allows even very stable and complex carbon and nitrogen compounds to be converted quantitatively.

The sample aliquot is dosed directly into the hot zone of the filled reactor (combustion tube). Here, the pyrolysis and oxidation of the sample in the carrier gas flow is performed with the aid of the catalyst. The carrier gas is also used as an oxidizing agent.



R - carbonic substance

The measuring gas is cooled in the condensation coil and condensed water is separated from the measuring gas in the subsequent TIC condensate container. After further drying and removal of corrosive gases, the CO₂ measuring gas is added to the NDIR detector or NO detector.

Inorganic carbon is detected by injecting a sample aliquot into the acidic TIC reactor and driving out the formed CO₂ via the NDIR detector.

The CO₂ or NO concentration is detected several times every second. An integral over time is calculated from this signal sequence. The integral is proportional to the concentration of the carbon or nitrogen in the measurement solution. Afterwards, the calculation of the carbon or nitrogen content in the sample is performed via a previously determined calibration function.

3.4 Measuring methods

The detection of several parameters can be combined in the multiWin control and analysis software.

3.4.1 TC analysis

TC: Total Carbon

In TC analysis, the total carbon contained in the sample, i.e. the organic and inorganic bound carbon, as well as elemental carbon, is detected.

The sample is dosed automatically into the combustion tube and digested, and the generated carbon dioxide is detected.

TN_b detection is possible in parallel to TC detection.

3.4.2 TOC analysis

TOC: Total Organic Carbon

In TOC analysis, the total organic bound carbon contained in the sample is detected.

TOC determination is carried out in the analyzer using the differential method which can be described with the following formula.

$$\text{TOC} = \text{TC} - \text{TIC}$$

TOC - total organic carbon

TC - total carbon

TIC - total inorganic carbon

Two sequential measurements are used on one sample to determine TIC and TC. The calculated difference is given as TOC. The differential method detects volatile as well as non-volatile organic carbon compounds.

TOC analysis can be used when the sample contains easily purgeable organic substances such as benzol, cyclohexane, chloroform, etc. If the TIC content of the sample is significantly above the TOC content, TOC analysis should not be applied.

TN_b detection is possible in parallel to TOC detection.

3.4.3 TIC analysis

TIC: Total Inorganic Carbon

In TIC analysis, the total inorganic carbon from carbonates and hydrocarbonates, as well as dissolved CO₂, is detected.

Cyanides, cyanates, isocyanates and carbon particles are not detected.

An aliquot of the sample is dosed directly into the TIC reactor to determine the inorganic carbon (TIC). The CO₂ is purged and detected.

3.4.4 NPOC analysis

NPOC: Non-purgeable Organic Carbon

During the NPOC analysis, the total non-purgeable organic carbon content of a sample is detected.

The sample is acidified to $\text{pH} < 2$ outside of the analyzer with acid (HCl (2 mol/l)). The generated CO_2 is purged externally, e.g., in the autosampler. The analyzer then determines the remaining organic carbon in the sample.

Other highly volatile organic compounds are purged with the CO_2 . The NPOC analysis should not be used when the sample contains easily purged organic substances.

NPOC analysis according to the NPOC plus method

This method was developed especially for the detection of low TOC content in samples with high TIC content or a high level of dissolved CO_2 . The NPOC method is generally recommended for the analysis of such samples. For high and, in particular, unknown TIC content, very long time periods ($t > 10$ min) may, however, be required for complete purging of the CO_2 . This is why the inorganic bound carbon is purged externally with this method.

The NPOC plus method process is a combination of the NPOC and the differential method.

As with NPOC analysis, the sample is acidified ($\text{pH} < 2$) outside of the analyzer. Immediately before the analysis of the sample, the greater part of the carbon dioxide generated is purged externally. Afterwards, the remaining organic carbon (TOC) from the sample prepared in this manner is determined using the differential method.

The TIC value determined using this method is only a calculated variable and of no analytical relevance.

Highly volatile organic substances are also purged during the sample preparation and not detected for this reason.

TN_b detection is possible in parallel to the NPOC and NPOC plus detection.

3.4.5 DOC analysis

DOC: Dissolved Organic Carbon

In DOC analysis, the organic carbon remaining in the filtrate after the sample is filtered is determined. The filter typically has a pore size of $0.45 \mu\text{m}$.

The sample is filtered outside of the analyzer and then analyzed as a TOC sample.

3.4.6 POC analysis

POC: Purgeable Organic Carbon

During POC analysis, the total purgeable organic carbon is detected. At low pH values, parts of the inorganic carbon (carbonates, bicarbonates) are also converted to CO_2 . The CO_2 is purged from the sample with the volatile organic components.

To determine the purgeable organic carbon, an aliquot of the sample is transferred to the optional POC module. In the POC module, a carrier gas purges the volatile components out of the sample.

The purged components are fed through an adsorber tube. The adsorber tube binds the CO_2 in the gas mixture, separating the proportion of inorganic bound carbon. The volatile organic compounds pass through the tube. They are led to the analyzer and oxidized there. The resulting CO_2 is detected.

3.4.7 TN_b analysis

TN_b : Total Nitrogen bound

The content of nitrogen compounds in aqueous samples can be determined in the analyzer. In environmental samples, these can be ammonia salts, nitrites and nitrates, and in pharmaceutical samples, amino acids and proteins.

The thermocatalytic oxidation results in nitrogen oxides which can be detected using a chemiluminescence detector (CLD) or an electrochemical detector (ChD).

3.5 Catalysts

As an oxygen carrier, the catalyst supports combustion of the samples. Solids that are catalytically active in a temperature range of 700 to 950 °C can be used as catalysts.

The platinum catalyst can be used universally over the entire working range for carbon and nitrogen determination. Its optimal function is at a reaction temperature of 800 °C. Because of its very low individual blank value, it allows safe and precise analysis of low carbon and nitrogen contents. The catalyst also works effectively during analysis of highly contaminated waters.

To minimize wear, reducing the furnace temperature to temperatures below the melting point of the salts is recommended with high salt matrices (e.g., seawater).

Alternatively, a CeO_2 catalyst can be used at a reaction temperature of 850 °C.

3.6 Calibration

3.6.1 Calibration strategies

Multiple point calibration with constant sample volume

In many applications, multiple point calibration with a constant dosage volume and multiple standard solutions at different concentrations is suitable.

The calibration range can encompass a wide range of concentrations and must be defined in accordance with the expected sample concentrations. Multiple standard solutions are measured with the selected method.

Multiple point calibration with constant concentration

Additionally, a multiple point calibration with variable dosage volumes and constant concentration can be performed. This calibration strategy is particularly interesting and the norm in the pharmaceutical industry for measurements at very low concentrations (<1 mg/l).

Only create one standard solution for the calibration range. The analyzer then analyzes different volumes of this standard solution. Do not go below the lowest standard solution volume of 2 ml when doing this.

Check the calibration via a second independently made standard solution to exclude errors during standard solution creation.

Take the blank value of the preparation water into account for measurements in the range of low concentration (<10 mg/l).

Single point calibration

For low TOC concentrations such as those in the pharmaceutical industry, single point calibration is a very good solution. A big advantage is that the device blank value is low and that the NDIR detector performs linear measurement across a wide range of concentrations.

Proceed as follows to minimize errors during manual standard solution creation:

- Prepare 3 standard solutions at the same concentration.
- Measure the standard solutions.
- Determine the calibration curve from the average value in the results.

Take the blank value of the preparation water into account during single point calibration.

3.6.2 Daily factor

Calibration with a standard solution can be checked and corrected via the daily factor. The software multiplies all subsequent measurement results with this factor.

The daily factor F is calculated in accordance with the following formula:

$$F = c_{\text{target}} / c_{\text{actual}}$$

3.6.3 Calibration method

Each parameter (TC, TOC, TIC, etc.) of a method can be calibrated in the multiWin software. Not all parameters require calibration, however.

You can store up to three calibration functions for different concentration ranges for each parameter in a method. The software automatically assigns the measurement results to the correct calibration range.

The software determines the calibration function in relation to mass m per injected sample. It determines linear or quadratic calibration functions in accordance with the following equations via regressive calculation:

$$\text{Linear calibration function: } c = (k_1 \times I_{\text{Net}} + k_0) / V$$

$$\text{Quadratic calibration function: } c = (k_2 \times I_{\text{Net}}^2 + k_1 \times I_{\text{Net}} + k_0) / V$$

c : target concentration of the standards

V : Sample volume

I_{Net} : Net integral

k_0, k_1, k_2 : calibration coefficient

The net integral is the raw integral corrected by the blank value of the preparation water.

You can specify the regression type (linear or quadratic). Individual measuring points or measured values for the calculation of the current calibration (manual outlier selection) can be selected. If necessary, you can define individual standards again, or also add additional measurement points to the calibration.

Up to 20 calibration points can be used, with a tenfold determination per calibration point. The calibration function can be determined either from the average values of the repeat measurements or from all individual determinations.

TC/NPOC

The TC channel is calibrated directly for the TC parameter, and after sample purging for the NPOC parameter.

The concentration c_{TC} is proportional to integral I_{TC} : $c_{\text{TC}} = f(I_{\text{TC}})$.

TIC	The TIC channel is calibrated. The following applies: $c_{TIC} = f(I_{TIC})$ The calculated parameters appear in the method in the TIC analysis channel. The calculation of the analysis results is based on the calculated calibration function.
TOC	The TOC is determined with the differential method (TOC Diff). Generally, separate calibration functions are determined for the TC and TIC channels. The calculation of the analysis results is based on the calculated calibration functions for TC and TIC. The TOC content results from the following formula: $c_{TOC} = c_{TC} - c_{TIC}$ The parameters appear in the TIC and TC analysis channels in the method. The TC and TIC parameters can be calibrated simultaneously. The use of mixed standard solutions such as carbonate/hydrogen carbonate and potassium hydrogen phthalate or sucrose is recommended for this. The TIC and TC channels can also be calibrated consecutively with separate standard solutions. This is useful if different ranges are to be calibrated for the TC and TIC channels.
NPOC plus	The calibration of the NPOC plus method is the same as the calibration of the TOC (Diff) method. Before analysis, the TIC must be sufficiently purged for the use of the differential method to be practical. Method process: ■ Separate calibration of TIC and TC channels ■ Measurement of samples and calculation of the analysis results via the software – Purging of the acidified sample (3 to 5 min) – Determination of the remaining TIC with the calibration curve – Determination of the TC with the calibration curve – Calculation of the TOC from the difference of TC and TIC The matrix-dependent calibration is as close to real samples as possible. To do this, add carbonate to the standard solutions until you get a TIC content similar to that of the samples.
TNb	The TN channel is calibrated. The following applies for the determined calibration function: $c_{TN} = f(I_{TN})$. The calculated parameters appear in the method in the TN analysis channel.

3.6.4 Method characteristics

Remaining standard deviation	The remaining standard deviation (remaining variance) expresses the dispersion of the integral values around the regression function (regression precision).
Method standard deviation	The standard deviation of the method describes the quality of the calibration in a non-ambiguous and generally applicable way. For the non-ambiguous evaluation of the quality, the standard deviation of the method must be used.
Method variation coefficient	The variation coefficient of the method (relative standard deviation of the method) is used for the comparison of different calibrations with different calibration ranges.

Correlation coefficient	The correlation coefficient compares the dispersion of the calibration measuring points of the regression function with the total dispersion of the calibration. If all calibration measuring points are on the calculated regression function, the correlation coefficient is +1 or -1. With a positive correlation coefficient, the regression function is increasing, with a negative coefficient it is decreasing.
Coefficient of determination	The square of the correlation coefficient is called the coefficient of determination.
Limit of verification	The verification limit of the calibration specifies the lowest concentration that can be differentiated qualitatively from the zero point with a given probability. The verification limit should always be smaller than the lowest calibration measuring point.
Limit of detection	The detection limit of the calibration specifies the lowest concentration for which a verification is possible with a given probability.
Limit of determination	The determination limit of the calibration specifies the lowest concentration that can be differentiated quantitatively from the zero point with a given probability.

3.6.5 Other calculations

	For all measurements where multiple injections are carried out, the average value (AV), the standard deviation (SD) and the variation coefficient (VC) are calculated and displayed. For each sample, a tenfold determination can be carried out as a maximum.
Outlier selection	The control and analysis software can automatically select outliers. The user can specify a maximum limit for the variation coefficient or even for the standard deviation for this. The analyzer performs the minimum number of measurements specified in the method. If the distribution of the measured values is then above the specified maximum value (SD or VC), additional injections are carried out from the same sample until the specified maximum number of measurements has been reached. After each measurement, the software determines the variation coefficient and standard deviation for all combinations of the measured values. If the variation coefficient or the standard deviation of at least one combination is smaller than the specified maximum value, no further measurements are carried out. The software determines the analysis result from the combination of measured values with the smallest variation coefficient or the smallest standard deviation. The unused measurements are considered outliers and deleted. If carbon and nitrogen are detected in parallel, the outlier selection takes place separately for each parameter.
Average value	The average value of the final result is calculated from the concentrations determined for the individual detections after eliminating the outliers.

3.7 Blank values

3.7.1 Water blank values

Preparation water blank value	Especially for measurements with low TOC concentrations ($\mu\text{g/l}$ range), the TOC content of the water used to prepare the standard solutions must be taken into account. The concentration of the standard solution and the TOC blank value of the preparation water are often within the same range. This blank value can be taken into account during calibration. The TOC content of the preparation water is measured separately before the calibration. The software then subtracts the average integral determined for the preparation water for each measuring point of the calibration from the determined gross integral. $I_{\text{Net}} = I_{\text{Gross}} - I_{\text{Preparation water}}$ The software determines the calibration function from the net integrals. Mathematically, this corresponds to a parallel movement of the calibration line. The software also takes the preparation water blank value into account when determining the daily factor.
Diluent blank value	If the sample is diluted, the blank value for the diluent is of interest. This value can be determined separately or entered manually in the software. The software takes the diluent blank value into account when calculating the concentration of diluted samples. The diluent blank value can change over time and must therefore be determined again before beginning a measurement series. Otherwise, the software will use the last value. The diluent blank value is always indicated in the software normalized to a volume of 1 ml.
Diluent blank value use	The software calculates the actual diluent integral (I_{DiBV}) for each measurement based on the diluent blank value, the sample volume used and the dilution ratio. The software then subtracts the diluent integral (I_{DiBV}) from the experimentally determined raw integral (I_{Raw}). $I_{\text{DiBV}} = V_{\text{DiBV}} \times (V_{\text{Sample}} - N_{\text{p}}/N_{\text{D}} \times V_{\text{Sample}})$ $I_{\text{eff}} = I_{\text{Raw}} - I_{\text{DiBV}}$ V_{DiBV}: Diluent blank value V_{Sample}: Sample volume I_{eff}: Effective integral N_{p}: Number of units of the primary sample N_{D}: Number of units of the diluent I_{Raw}: Raw integral I_{DiBV}: Diluent integral
Diluent indication	Proportions of the primary probe: in total proportions (e.g., 10 parts in 100 parts) This means that 10 ml of the primary sample is filled to a total volume of 100 ml with dilution water. A 1:1 dilution ratio equals $I_{\text{DiBV}} = 0$.
Calculation of the sample concentration	To calculate the sample concentration c, the sample volume and the dilution ratio are used: $c = m/V_{\text{Sample}} \times N_{\text{D}}/N_{\text{p}}$

The following equation applies for the linear calibration function:

$$c = (k_1 \times I_{\text{eff}} + k_0) / V_{\text{Sample}} \times N_D / N_P$$

If the user dilutes a sample and enters the dilution ratio in the software, the software automatically calculates the concentration of the undiluted primary sample and outputs it to the analysis report.

3.7.2 Eluate blank value

The eluate blank value is a special blank value for samples from cleaning validation or eluate preparation. It corresponds to the TOC content of the ultrapure water used which has been used, e.g., to extract/eluate swabs.

The eluate blank value is a fixed method parameter. The user can activate or deactivate the eluate blank value in the method. The user can optionally determine the eluate blank value separately and enter it in the software manually.

The blank value can change over time and must therefore be determined again before beginning a measurement series. Otherwise, the software uses the last value.

The eluate blank value is always indicated normalized to 1 ml.

The eluate blank value is not taken into account when carrying out a calibration. The calibration is carried out with normal standard solutions in which only the preparation water blank value is taken into account.

If samples are measured with the so-called eluate method, the software automatically subtracts the integral of the blank value from the integral of the sample measurement.

$$I_{\text{eff}} = I_{\text{Raw}} - I_{\text{Eluate blank value}}$$

I_{eff} : Effective integral

I_{Raw} : Raw integral

$I_{\text{Eluate blank value}}$: Eluate blank value

3.7.3 Boat blank value

For solids methods, the user can determine the boat blank value. To do this, the user inserts a boat with sample additives in the combustion furnace and analyzes it.

The user can optionally determine the boat blank value separately and enter it in the control and analysis software.

The boat blank value can change over time and must therefore be determined again before beginning a measurement series. Otherwise, the software will use the last value.

3.8 System suitability test

System suitability tests are used in the pharmaceuticals industry to validate analytical methods and devices to document the suitability of the selected procedure.

For TOC analysis in the ultrapure water range for pharmaceutical purposes, such as e.g., WFI (Water For Injection), the recovery rate of a poorly oxidizable compound is determined in comparison with that of an easily oxidizable compound.

The standards solutions and their concentrations are defined in the respective pharmacopeia, e.g., in the European Pharmacopeia or in the USP (United States Pharmacopeia). These define sucrose as an easily oxidizable compound, and p-benzoquinone as a poorly

oxidizable one. The ratio of the recovery rate of p-benzoquinone to the recovery rate of sucrose must be within the range of 85 to 115 %. Only then is the selected method suitable.

Procedure:

- ▶ Create a reference solution of sucrose and TOC water with a concentration of 500 µg/l. This corresponds to a concentration of 1.19 mg/l sucrose.
- ▶ Prepare a solution of p-benzoquinone and TOC water that also has a concentration of 500 µg/l to check system suitability. This corresponds to a concentration of 0.75 mg/l p-benzoquinone.
- ▶ Determine the TOC concentrations of the reference solution, the system suitability solution and the TOC water in the selected mode (direct or differential method).

The effectiveness of the system in percent is calculated using the following formula:

$$E = (r_{ss} - r_w) / (r_s - r_w) \times 100$$

E: System efficiency in %

r_s : TOC of the reference solution (sucrose)

r_{ss} : TOC of the system suitability solution (p-benzoquinone)

r_w : TOC of the TOC water used (preparation water blank value)

4 Installation and commissioning

4.1 Installation conditions

4.1.1 Ambient conditions

- This laboratory device is designed for inside use.
- Avoid direct sunlight and radiation from heaters onto the device. If necessary, provide air conditioning.
- The installation site must be free of drafts, dust and caustic fumes.
- The room air must be as low in TOC and NO_x as possible.
- Avoid mechanical shocks and vibrations.
- Do not locate the device near sources of electromagnetic interference.
- Place the device on a heat-resistant and acid-resistant surface.
- The device must be positioned in such a way that allows easy access from all sides.
- Keep the ventilation slits free and do not obstruct them with other devices.

The following climate requirements apply in the room of operation:

Operating temperature	+10 to 35 °C (air-conditioning recommended)
Maximum humidity	90 % at 30 °C
Air pressure	0.7 to 1.06 bar
Storage temperature	5 to 55 °C
Humidity during storage	10 to 30 % (use desiccant)
Operating altitude (max.)	2000 m

4.1.2 Device layout and space requirements

The basic device and its modules were designed as table-top devices. The required space depends on all components that make up the measuring station.

Further components of the measuring station:

- The PC, monitor and printer may be placed on a separate side table.
- An acid-resistant waste container can be placed on or under the bench.
- The AS 10e and AS 21hp autosamplers are attached to the right side wall of the basic device. Alternatively, the autosamplers can be placed next to the device.
- The AS vario, AS vario ER and EPA Sampler autosamplers must be positioned to the left of the device.
With the multi N/C pharma HT model, these autosamplers must be placed next to the device on the right.
- The CLD nitrogen detector is set up to the right of the basic device.
With the multi N/C pharma HT model, the nitrogen detector must be placed next to the device on the left.
- The ChD (approx. 0.5 kg) nitrogen detector is installed in the basic device.
- The integrated solids module (Swab-Test Module) is attached to the left side panel of the basic device.
- The HT 1300 and the manual TIC solids module are placed to the right of the basic device. The HT 1300 solids module can be set up with the front side or with its left side facing forward.
- The FPG 48 solids sampler is placed in front of the HT 1300 solids module.

Component	Dimensions (width x height x depth)	Weight
Basic device	513 x 464 x 550 mm	30 kg
multi N/C 3100 duo modular measuring system (basic device + AS vario ER autosampler + HT 1300 solids module + FPG 48 autosampler)	2215 x 550 x 650 mm (minimum)	85 kg
AS 10e autosampler	260 x 390 x 320 mm	4.5 kg
AS 21hp autosampler	260 x 390 x 320 mm	4.5 kg
AS vario, AS vario ER autosamplers	350 x 470 x 400 mm	15 kg
EPA Sampler	500 x 560 x 400 mm	15 kg
CLD nitrogen detector	300 x 470 x 550 mm	12.5 kg
Swab-Test Module	300 x 80 x 80 mm	3 kg
HT 1300 solids module	510 x 470 x 550 mm	22 kg
FPG 48 autosampler	500 x 460 x 650 mm	20 kg
Manual TIC solids module	300 x 470 x 550 mm	10 kg

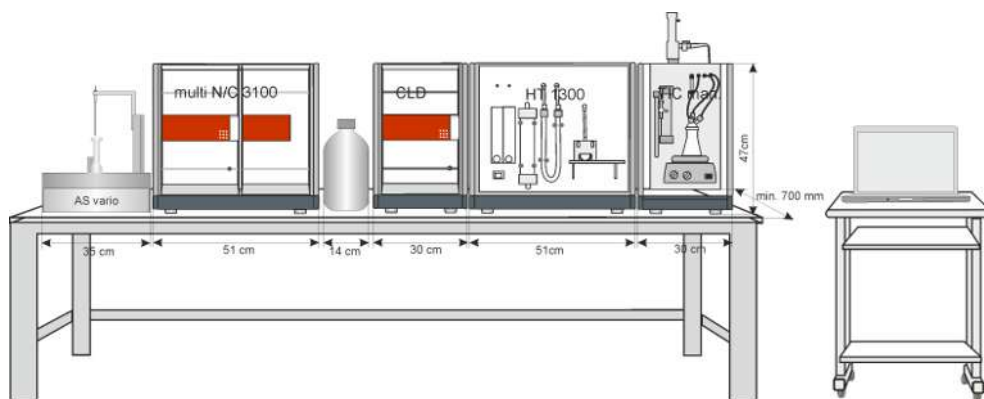


Fig. 19 Space required for multi N/C 3100 with modules

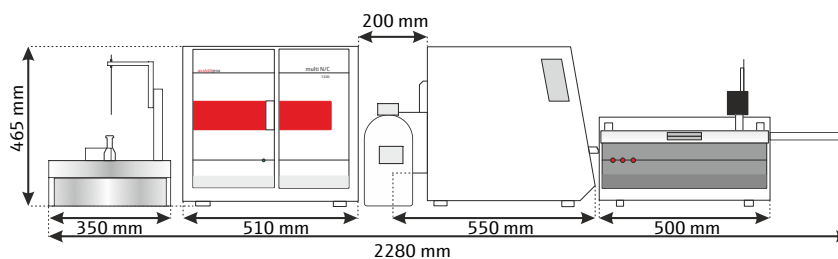


Fig. 20 Space required for multi N/C 3100 duo modular measuring system

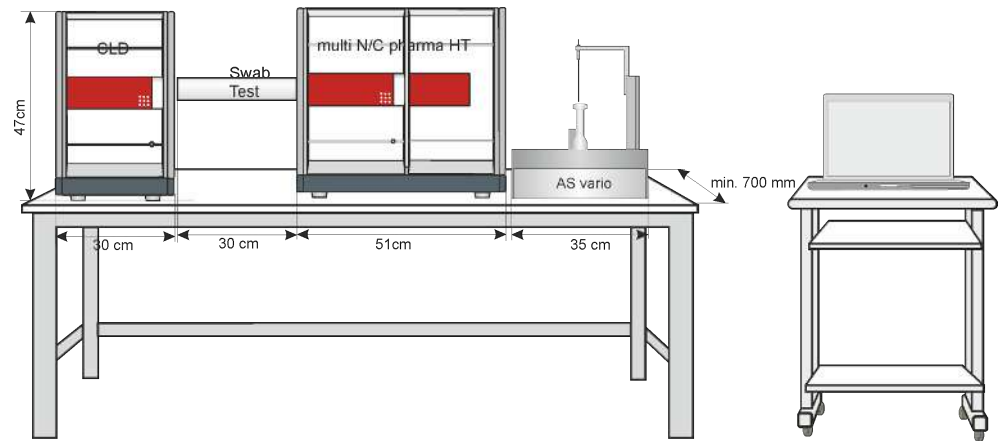


Fig. 21 Space required for multi N/C pharma HT with modules

4.1.3 Power supply



WARNING

Danger due to electrical voltage

- Only connect the device to a properly grounded socket which complies with the voltage indicated on the device's rating plate.
- Do not use an adapter in the feeder.

The device operates on single-phase alternating current.

The installation of the electrical equipment in the laboratory must comply with the DIN VDE 0100 standard. At the connection point, an electrical current in accordance with the standard IEC 60038 must be available.

4.1.4 Gas supply

The operator is responsible for the gas supply with connections and pressure reducers.

The connection hose is supplied:

- Outer diameter 6 mm
- Inner diameter 4 mm

4.2 Unpacking and setting up the device

The device will be delivered directly to the final device location by a transportation company. The delivery by this company requires the presence of a person responsible for device installation.

It is imperative that all persons designated to operate the device are present during the briefing given by the service technician.

The device may only be set up, installed and repaired by the customer service department of Analytik Jena or by persons authorized by Analytik Jena.

When installing and commissioning your device, observe the information in the "Safety instructions" section. Compliance with these safety instructions is a requirement for the error-free installation and the proper functioning of your measuring station. Observe all warnings and instructions that are attached to the device itself or displayed by the control and analysis program.

To ensure trouble-free operation, please make sure that the installation conditions are observed.

4.2.1 Installing and commissioning the analyzer

After initial commissioning, you may want to transport the device again, or store it. You can recommission the analyzer as described below. Analytik Jena always recommends installation via customer service.

- ▶ Carefully remove the basic device, the accessories and the supplementary device from the transport packaging. Retain the transport packaging for future transport.
- ▶ Place the analyzer at its intended location.
- ▶ Remove the adhesive tape from the doors and side walls.
- ▶ Remove the adhesive tape from the top cover. Remove the top cover.
- ▶ Open the left side wall:
 - Unscrew the four attachment screws. The screws are captive and remain attached to the wall.
 - Remove the protective grounding. Set the side wall aside safely.
- ▶ Remove all remaining adhesive tape and protective bags.
- ▶ Install the combustion furnace.
- ▶ Mount the condensation coil in the device interior.
- ▶ Fill the combustion tube. Insert the combustion tube into the combustion furnace.
- ▶ Close the left side wall of the analyzer again:
 - Attach the protective grounding to the side wall.
 - First screw in the screws on the bottom side and then on the top side. Tighten the screws in turns.
- ▶ Open the front doors.
- ▶ Install the halogen trap and the water traps.
- ▶ Mount the TIC condensate container on the front side.
- ▶ Connect the canulas with hoses AA and 7. Tighten the Fingertight connections finger-tight.
- ▶ Reapply the top furnace cover.
- ▶ Put the reagent bottle with the drip tray into the analyzer.
- ▶ Close the doors of the analyzer.
 - ✓ The device has been installed.

See also

📖 Maintenance and care [▶ 86]

4.2.1.1 Connecting the analyzer

The mains power connection and the media connections are located on the rear of the device.

A diagram in the center details the different connections.

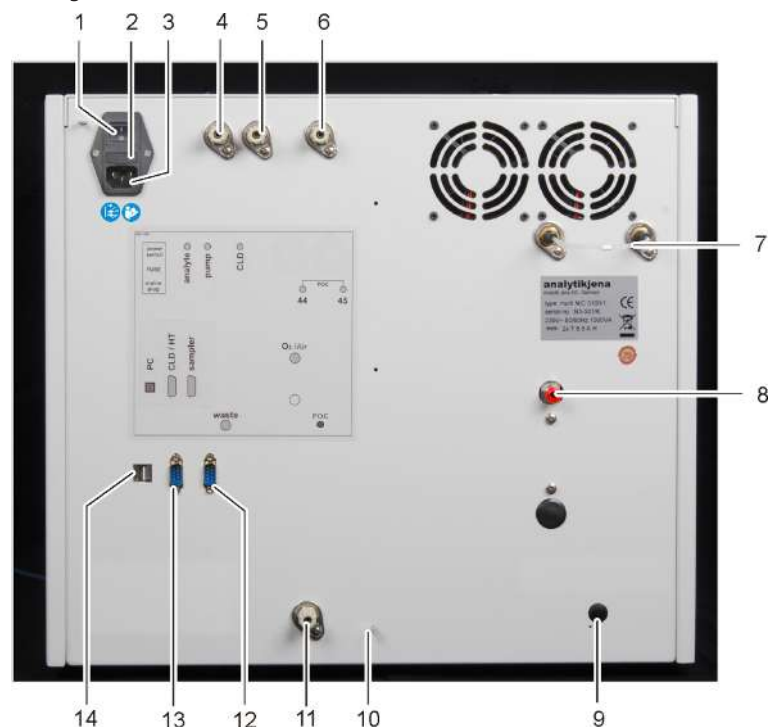


Fig. 22 Device rear

- | | |
|---|---|
| 1 "Power switch" main switch | 2 "FUSE" mains fuse holder |
| 3 "Main plug" mains connection | 4 "analyte" gas connection |
| 5 "pump" gas connection | 6 "CLD" gas connection |
| 7 Bridge for the gas connection of the POC module | 8 "O ₂ /Air" carrier gas connection |
| 9 POC module connection (optional) | 10 Connection of the neutral conductor on the autosampler |
| 11 "waste" connection | 12 RS 232 interface for "sampler" autosampler |
| 13 RS 232 interface for "CLD/HT" CLD and solids modules | 14 USB 2.0 "PC" interface |

Connecting the power



NOTICE

Risk of damage to the sensitive electronics

- Only connect the device and the other components to the power grid when they are switched off.
- Only connect and disconnect electrical connection cables between the system components when the system is switched off.



NOTICE

Damage to the electronics due to condensation

Significant temperature differences can lead to the formation of condensation which can damage the device's electronics.

- After long-term storage or transport in a colder environment, allow the device to acclimatize at room temperature for at least one hour before switching it on.

-
- ▶ Connect the connection cable to the mains power connection on the rear of the analyzer.
 - ▶ Connect the power plug to a grounded power outlet.
 - ▶ Do not switch the device on yet.

Connecting the gases

You are responsible for the gas supply in the laboratory. Ensure that the inlet pressure on the pressure reducer is set between 400 to 600 kPa.

- ▶ Connect the carrier gas. To do this, connect the supplied connection hose to the pressure reducer of the gas supply.
- ▶ Connect the carrier gas hose to the "O₂/Air" gas connection on the rear of the device.
 - To do this, insert the hose in the quick-release connector.
 - To release the hose again later, press the red ring back and pull the hose out of the connection.

Connecting accessories



WARNING

Risk of chemical burns from concentrated acids

Concentrated acids are highly corrosive and sometimes have an oxidizing effect.

- Wear safety goggles and protective clothing when handling concentrated acids. Work under an extractor.
- Observe all instructions and specifications in the safety data sheets.

Connect the reagent bottle and accessory components as follows:

- ▶ Connect the waste hose to the "waste" connection on the rear of the analyzer. Put the free hose end in a suitable waste container.
- ▶ Open the front doors on the analyzer.
- ▶ Fill the reagent bottle with phosphoric acid (10 %). Put the bottle with the drip tray into the analyzer.
- ▶ Connect hoses 4 and AC to the reagent bottle with phosphoric acid.
 - ✓ The analyzer has been commissioned.

4.3 Connecting accessories



NOTICE

Risk of damage to the sensitive electronics

- Only connect the device and the other components to the power grid when they are switched off.
- Only connect and disconnect electrical connection cables between the system components when the system is switched off.

4.3.1 The AS 10e and AS 21hp autosamplers

AS 10e autosampler

The autosampler is equipped with a rotatable sample tray for 10 sample vessels with a volume of 50 ml. Optionally, sample values with a volume of 40 ml can be used.



Fig. 23 AS 10e autosampler

The autosampler can be equipped with two canulas. This allows the autosampler to automatically purge samples for NPOC analysis.

During **NPOC analysis**, the sample is acidified outside the analyzer with diluted acid to a pH value of <2 . The autosampler purges volatile organic compounds and the produced CO_2 from the sample by means of the carrier gas. The analyzer then determines the remaining organic carbon.

During NPOC analysis, the autosampler works **sequentially**:

- First, the autosampler purges volatile organic compounds and CO_2 from the sample.
- In a second step, the autosampler picks up the prepared sample and transfers it via the intake hose to the analyzer.

AS 21hp autosampler

The autosampler is equipped with a rotatable sample tray for 21 sample vessels with a volume of 50 ml. Optionally, sample values with a volume of 40 ml can be used.

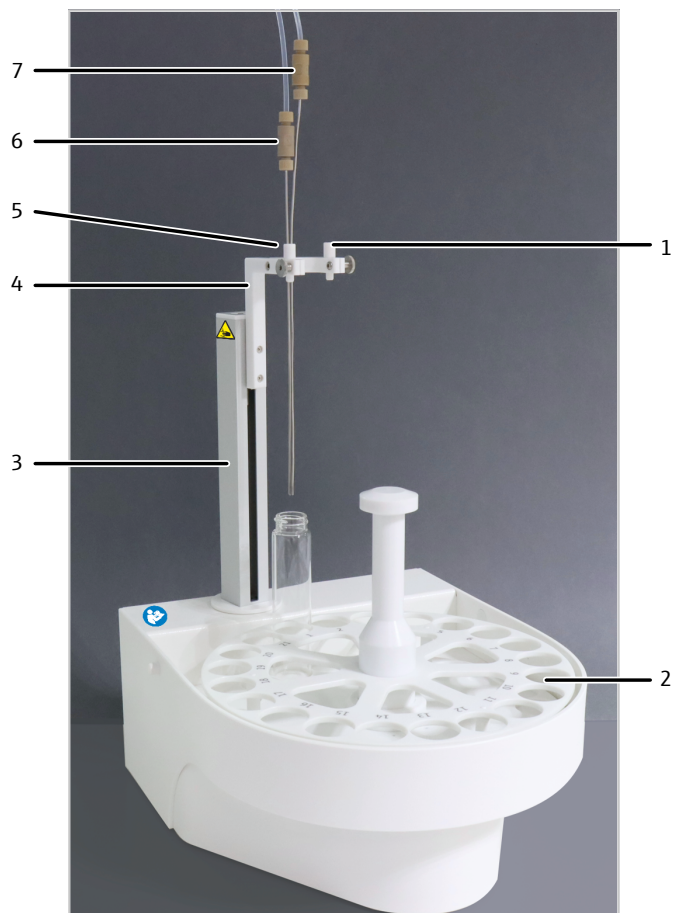


Fig. 24 AS 21hp autosampler

- | | |
|---|--|
| 1 Sleeve (with 1 bore) as a purging canula holder | 2 Sample tray (rotatable, 21 samples) |
| 3 Autosampler arm with Z-drive | 4 Canula holder |
| 5 Sleeve (with 2 bores) | 6 Sample intake canula with screw connection |
| 7 Purging canula with screw connection | |

The autosampler can be equipped with two canulas. This allows the autosampler to automatically purge samples for NPOC analysis.

The autosampler comes with a canula holder for two canulas. The holder keeps the two canulas at a distance. This allows the autosampler to aspirate a sample and to purge a second sample in parallel (**parallel purging**). During NPOC analysis, the autosampler can also work sequentially (option).

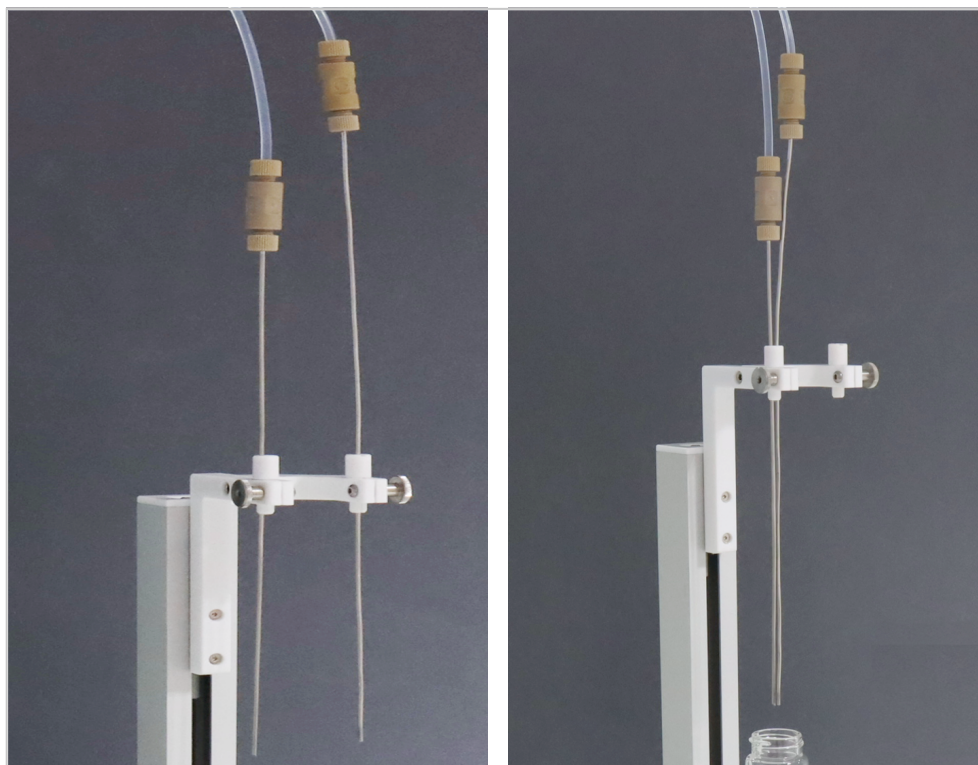


Fig. 25 Parallel purging (left) and sequential purging (right)

The autosampler has an integrated magnetic stirrer. The magnetic stirrer automatically homogenizes samples containing particles prior to sampling. You can define the stirring speed in the multiWin software in the method under the process parameters.

Autosampler in operation

Both autosamplers can be attached to the right-hand side of the analyzer by means of the supplied holder. Alternatively, the autosamplers can be placed next to the analyzer.



Fig. 26 Autosampler attached to the analyzer by means of the holder

The external power supply unit supplies the autosampler with operating voltage (24 V DC). The autosamplers do not have a mains switch. The analyzer is connected to the bottom of the autosampler via the RS 232 interface.

Cover (optional)

A cover is provided as an optional accessory for both autosamplers. The cover protects the sample chamber against environmental influences from the laboratory atmosphere.

4.3.1.1 Installing and commissioning the sampler



CAUTION

Risk of injury from moving parts

There is a risk of injury in the movement range of the sampler arm. For example, hands or fingers might be crushed.

- Maintain a safety distance from the sampler during operation.



NOTICE

Risk of device damage

If the sampler arm is obstructed during operation, the drives can be destroyed.

- Do not touch the sampler arm during operation.
 - Only carry out manual adjustment when the device is switched off.
-
- ▶ Switch off the analyzer before installing the autosampler.
 - ▶ Plug the grounding conductor into the connection on the rear of the analyzer. Connect the grounding conductor to the connection on the bottom of the sampler.
 - ▶ Plug the cable on the low voltage side of the external power supply unit into the connection on the bottom of the sampler. Do not connect the power supply unit to the mains power supply yet.
 - ▶ Connect the autosampler to the analyzer with the interface cable (interface on the bottom of the sampler and "sampler" interface on the rear of the analyzer).



Fig. 27 Connections on the bottom of the autosampler

- | | |
|--|--------------------------|
| 1 Connection for equipotential bonding cable (grounding cable) | 2 Power cable connection |
| 3 Analyzer interface | |

- ▶ Attach the autosampler to the side of the analyzer with the holder.
 - Screw the holder to the right side of the analyzer with the two knurled head screws.
 - Insert the autosampler into the holder. To do this, insert the two knurled head screws on the rear of the sampler into the slots of the holder.

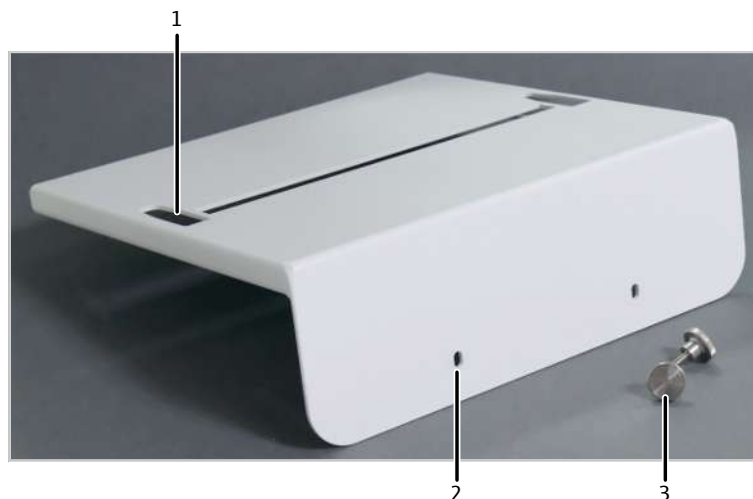


Fig. 28 Holder

- | | |
|--------------------------------------|-----------------------------------|
| 1 Slot for inserting the autosampler | 2 Bore for attaching the analyzer |
| 3 Knurled head screw | |

- ▶ **Alternatively:** Place the autosampler to the left of the analyzer.
- ▶ Place the sample tray on the autosampler. Ensure that it clicks into place.
- ▶ Place a sample vessel in position 1 of the sample tray.
For AS 21hp autosamplers only: Place a magnetic stirring rod in the sample vessel.
- ▶ Insert the canulas in the canula holder. To do this, guide the two canulas through the sleeve with two holes (for sequential purging).
- ▶ Manually adjust the height of the canulas so that the canula tips protrude 1 to 2 cm over the edge of the vessel at the highest position of the autosampler arm and do not touch the vessels when the sample tray rotates.
- ▶ Secure the canulas by slightly tightening the knurled head screws.
- ▶ Connect the hoses from the analyzer to the canulas using Fingertight connections:
 - Hose AA – sample intake hose
 - Hose 7 – purging hose for NPOC measurement
 - To do this, guide the hose through the banjo bolt (see image).
 - Slide the conical nipple onto the hose with the conical side towards the banjo bolt. The conical nipple and hose must be flush.
 - Retighten the Fingertight connections.

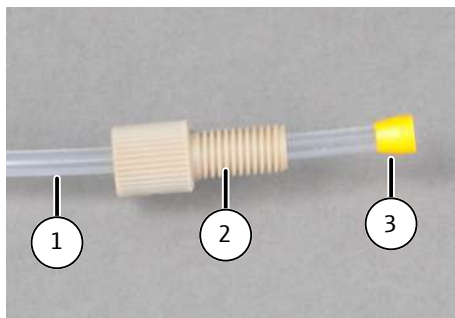


Fig. 29 Fingertight connection

- | | |
|------------------|--------------|
| 1 Hose | 2 Banjo bolt |
| 3 Conical nipple | |

Checking and changing the configuration

- ▶ Connect the power supply unit to the mains network.
- ▶ Switch on the analyzer. Start the multiWin program and initialize the analysis system.
- ▶ Check the device configuration with the **Instrument | System-Information** menu option in the **multiWin set-up info** window.
- ▶ If necessary, change the configuration:
 - Exit the multiWin program.
 - Start the setup tool in the Windows interface with **Start | Program files | multiWin | multiWin setup tool**.
 - Select the autosampler type in the **Sampler** list.
 - Exit the **multiWin set-up tool** window with **[Create]**.
 - Start the multiWin program and initialize the analysis system.
 - Open the **Options** window with menu option **Configuration | Edit options**. Go to the **Analyzer components** tab.
 - In the **Sampler** group, select the appropriate tablet and vessel size.
- ▶ Exit the window with **[OK]**.

Adjusting the autosampler

During adjustment, the canulas are adjusted to the sample tray so that they are optimally immersed into the sample vessels. Adjustment must be carried out during commissioning and after conversion, transport or storage.

- ▶ Adjust the immersion depth of the canulas in the vessel (z-axis direction) in the software.
 - Open the window of the same name with the **Instrument | Sampler Alignment** menu option.

multiWin Demo-Version - Alignment - sampler

Please loosen blocking screw on needle holder!

Rack size: 21

Go to position

Select position: 1

Please select position needing adjustment

Position 1

Position 1 adjust (old: z=0)

z [0 ... 116mm]: 0 + lower - higher

Fig. 30 Autosampler adjustment window Alignment - sampler

- Under **Please select position needing adjustment**, select **Position 1**.
- Click on **[Position 1 adjust]**. The autosampler arm lowers the canulas into the vessel in position 1.
- If necessary, increase or decrease the z-axis values. After each change, click **[Position 1 adjust]** to check the adjustment.
- With the AS 21hp autosampler, maintain a distance of about 0.5 cm from the magnetic stirring rod so that the rod can move freely and does not damage the canulas.
- When adjustment is complete, exit the window with **[Save]**.
- ✓ The autosampler is ready for operation.

4.3.1.2 Conversion for parallel purging (AS 21hp)

The AS 21hp autosampler is equipped with a canula holder which can accommodate two canulas and keep them at a distance. By repositioning the canulas, the autosampler can be easily converted to "parallel purging".

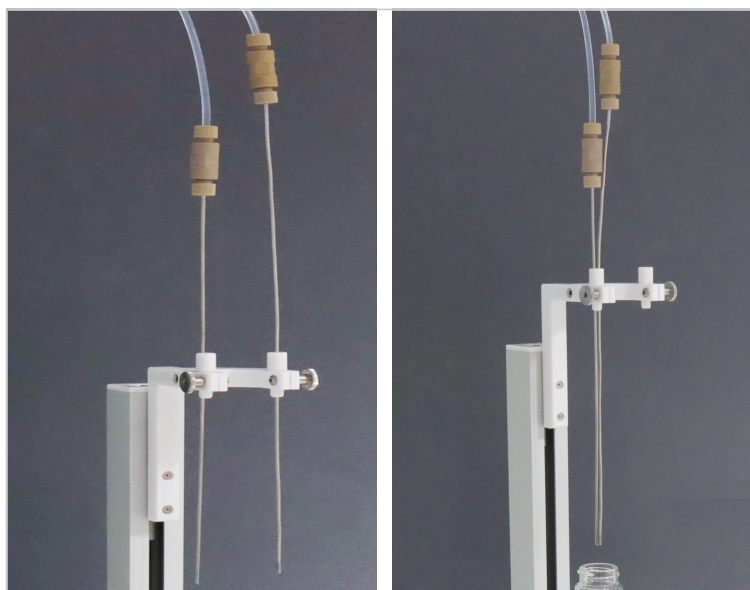


Fig. 31 Parallel purging (left) and sequential purging (right)

- Insert the canulas in the two positions of the canula holder in accordance with the image (left). Secure the canulas only slightly with the knurled head screws.

- ▶ Place two sample vessels in positions 1 and 2 of the sample tray under the two canulas.
- ▶ Place magnetic stirring rods in the vessels.
- ▶ Manually adjust the height of the canulas so that the canula tips protrude 1 to 2 cm over the edge of the vessel at the highest position of the autosampler arm and do not touch the vessels when the sample tray rotates.
- ▶ Secure the canulas by slightly tightening the knurled head screws.
- ▶ Connect the hoses to the canulas using Fingertight connections:
Sample intake hose AA – connection to the canula above position 1
Purging hose for NPOC measurement 7 – connection to the canula above position 2
- ▶ Check the configuration and adjust the autosampler. Installing and commissioning the autosampler

See also

 Installing and commissioning the sampler [▶ 47]

4.3.2 AS vario autosampler



CAUTION

Risk of injury from moving parts

There is a risk of injury in the movement range of the sampler arm. For example, hands or fingers might be crushed.

- Maintain a safety distance from the sampler during operation.



NOTICE

Risk of device damage by commissioning with transport locks

If you commission the device with the transport locks still in place, the drives may be damaged.

- Remove the transport locks before commissioning.



NOTICE

Risk of device damage

If the sampler arm is obstructed during operation, the drives can be destroyed.

- Do not touch the sampler arm during operation.
- Only carry out manual adjustment when the device is switched off.

6 different sample trays are available for the autosampler. A matching canula holder is available for each sample tray. The canula(s) can be flushed from the inside by drawing in sample or ultrapure water before sampling.

4 sample trays are available for the AS vario ER model.

Sample tray

Max. number of samples	Sample vessel	AS vario	AS vario ER
20	100 ml	Yes	No
47	12 ml + 50 ml	Yes	Yes
52	100 ml	Yes	No
72	40 ml + 50 ml	Yes	Yes
100	20 ml	Yes	Yes
146	12 ml	Yes	Yes

Technical data

Operating voltage	24 V DC via external power supply
Power consumption	50 VA
Grid voltage of external power supply	100 to 240 V, 50/60 Hz (autosensing)
Dimensions (W x H x D)	350 x 470 x 400 mm

The autosampler is placed next to the analyzer. It can be equipped with 2 canulas.

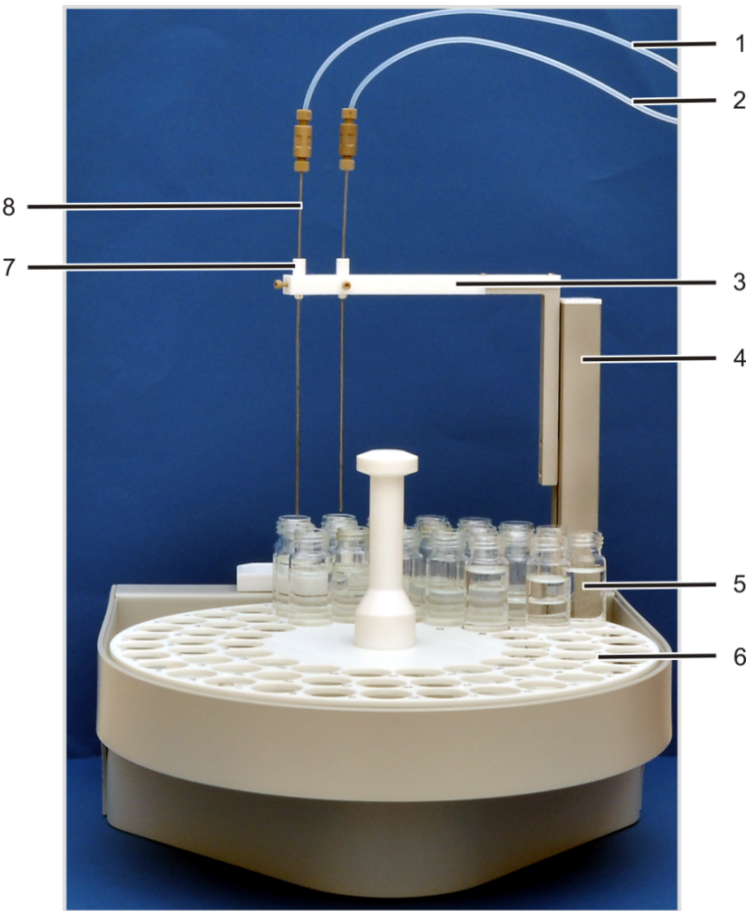


Fig. 32 Layout of the AS vario autosampler

- | | |
|---|--|
| 1 Connection hose to the analyzer (purging hose for NPOC measurement) | 2 Connection hose to the analyzer (sample intake hose) |
| 3 Canula holder | 4 Autosampler arm |
| 5 Sample vessel | 6 Sample tray |
| 7 Sleeve | 8 Canula |

The AS vario ER model is particularly suited for analyzing liquid samples with high solid particle content. The model is equipped with an additional canula flush that flushes the canula(s) with ultrapure water from the outside. When commissioning the autosampler, the ultrapure water supply for the canula flush must be installed additionally. It can be

used for all measurement methods, and in particular for NPOC analysis with parallel purging. When using different sample trays, the block with the wash cups is simply removed from the autosampler and exchanged.



Fig. 33 Layout of the AS vario ER autosampler

- | | |
|---|--|
| 1 Canula for connection to the sample intake hose | 2 Canula holder (here with no. 72) |
| 3 Sample tray for 72 | 4 Ultrapure water bottle |
| 5 Canula flush | 6 Canula for connection with the purge hose for NPOC measurement |

Removing the transport locks

The autosampler is secured for transport with a retaining screw on the bottom of the autosampler. Retain the transport lock for later transport.

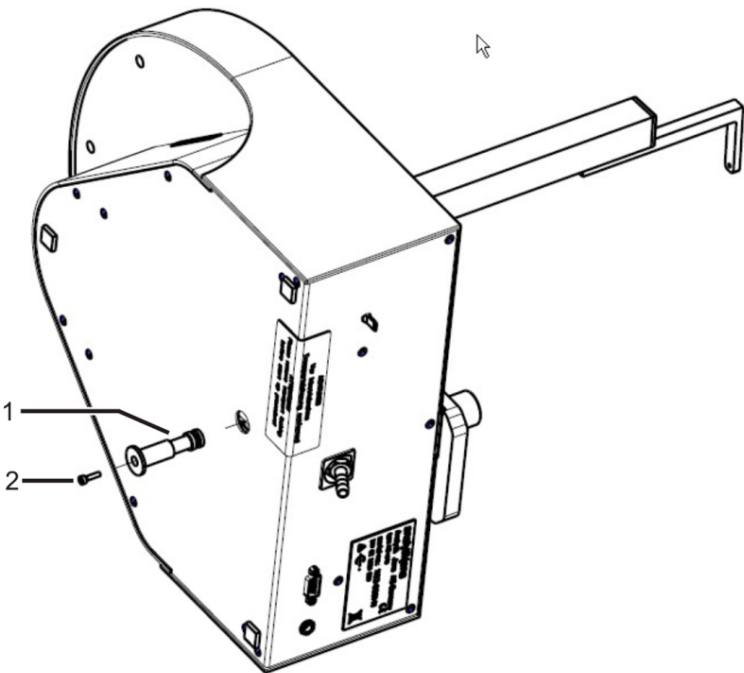


Fig. 34 Transport lock

- | | |
|------------------|---------------|
| 1 Transport lock | 2 M3x12 screw |
|------------------|---------------|

Commissioning the autosampler

- ▶ Turn the autosampler on its side and put it down safely.
- ▶ Remove the screw with the supplied hexagon socket screwdriver. Remove the transport lock (red plastic part).
- ▶ Place the autosampler on the bottom plate again.
- ▶ Switch off the analyzer before installing the autosampler.
- ▶ Plug the grounding conductor into the connection on the rear of the analyzer. Connect the grounding conductor to the connection on the rear of the autosampler.
- ▶ Plug the cable on the low voltage side of the external power supply unit into the connection on the rear of the autosampler. Do not connect the power supply unit to the mains power supply yet.
- ▶ Connect the autosampler to the analyzer with the interface cable (interface on the bottom of the sampler and "sampler" interface on the rear of the analyzer).
- ▶ Attach the outlet hose to the outlet connection on the rear of the autosampler. Insert the other end of the hose into the opening in the cover of the waste bottle.
 - i** NOTICE! Route the outlet tube at a constant downward incline. If necessary, shorten the hose. The hose must not dip into the liquid.
- ▶ Place the sample tray on the autosampler. Ensure that it clicks into place.
- ▶ Check that the correct canula holder is installed on the autosampler arm. The number engraved on the bottom must match the maximum number of sample vessels on the sample tray for this.
- ▶ Insert the canulas with matching sleeves into the canula holders.
- ▶ For NPOC measurement with parallel purging: Insert one canula each with sleeve in both positions of the canula holder (Fig. 32  52).
- ▶ For NPOC measurement with non-parallel purging: Insert both canulas in one sleeve with two holes in the position on the right (see below, not suitable for AS vario ER).



Fig. 35 Sleeve with two canulas for non-parallel purging

- ▶ Manually adjust the height of the canulas so that the canula tips protrude 1 to 2 cm over the edge of the vessel at the highest position of the autosampler arm and do not touch the vessels when the sample tray rotates.
- ▶ Secure the canulas by slightly tightening the knurled head screws.
- ▶ Connect the hoses from the analyzer to the canulas using Fingertight connections:
 - Hose AA – sample intake hose
 - Hose 7 – purging hose for NPOC measurement
 - To do this, guide the hose through the banjo bolt (see image).
 - Slide the conical nipple onto the hose with the conical side towards the banjo bolt. The conical nipple and hose must be flush.
 - Retighten the Fingertight connections.

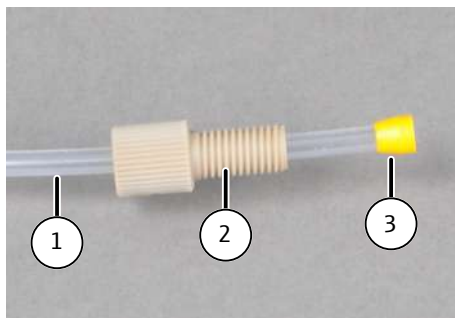


Fig. 36 Fingertight connection

- | | |
|------------------|--------------|
| 1 Hose | 2 Banjo bolt |
| 3 Conical nipple | |

Checking and changing the configuration

- ▶ Connect the power supply unit to the mains network.
- ▶ Switch on the analyzer. Start the multiWin program and initialize the analysis system.
- ▶ Check the device configuration with the **Instrument | System-Information** menu option in the **multiWin set-up info** window.
- ▶ If necessary, change the configuration:
 - Exit the multiWin program.
 - Start the setup tool in the Windows interface with **Start | Program files | multiWin | multiWin setup tool**.
 - Select the autosampler type in the **Sampler** list.
 - Exit the **multiWin set-up tool** window with **[Create]**.
 - Start the multiWin program and initialize the analysis system.
 - Open the **Options** window with menu option **Configuration | Edit options**. Go to the **Analyzer components** tab.
 - In the **Sampler** group, select the appropriate tablet and vessel size.
- ▶ Exit the window with **[OK]**.

Installing the canula flush

There is a suitable canula holder and a block with wash cups for each sample tray. The tray, the canula holder and the block are marked with the maximum sample number, e.g., 72.



Fig. 37 Canula flush on the AS vario ER model

- | | |
|----------------------------------|--------------------|
| 1 Ultrapure water connection | 2 Waste connection |
| 3 Removable block with wash cups | |

- ▶ Place the suitable block with wash cups on the autosampler.
 - For simpler installation, wet the o-ring on the bottom of the block with water.
 - Fasten the block to the autosampler with the two hexagon socket screws.
- ▶ Screw the ultrapure water connection into connection (1) and place the hose end in the ultrapure water bottle.
- ▶ Insert the waste hose in connection (2). Place the hose end in the waste container.

i NOTICE! Route the outlet tube at a constant downward incline. If necessary, shorten the hose. The hose must not dip into the liquid.
- ▶ Adjust the autosampler before the first start.

Activating the canula flush for measurements

multiWin® Demo-Version - Create method

Method | Process parameters

Name: NPOC [Edit]

Fixed method criteria

Furnace: ☒ Vertical furnace ☐ Horizontal furnace

State: liquid

Parameter: ☐ TC ☐ IC ☒ NPOC ☐ NPOC(+) ☐ TOC ☐ POC ☐ TN

☐ Consideration eluate ☐ Addition of reagent

Variable method criteria

Dimension: Concentration: mg/l [Edit]

Repetitions: min. 3 max. 3

Determination	1	2	3	4	5	6	7	8	9	10
Rinse cycles	3	1	1							
Reverse rinse	1									
2. Purging										
TIC Control										

[Comment] [Cancel] [Help] [Save]

Fig. 38 Activating the canula flush in the method

- ▶ Create a new method.
- ▶ Activate the **Reverse rinse** option under **Variable method criteria**.
- ▶ Define the number of flushing cycles by entering a number ≥ 1 in the table. One flushing process is usually sufficient.

See also

- ▢ Adjusting the AS vario autosampler [▶ 87]

4.3.3 EPA Sampler



CAUTION

Risk of injury from moving parts

There is a risk of injury in the movement range of the sampler arm. For example, hands or fingers might be crushed.

- Maintain a safety distance from the sampler during operation.



NOTICE

Risk of device damage

If the sampler arm is obstructed during operation, the drives can be destroyed.

- Do not touch the sampler arm during operation.
- Only carry out manual adjustment when the device is switched off.

The autosampler has a piercing function for sample vessels with septum caps. The sampler can be equipped with 1 to 2 canulas.

The special POC Sampler model is used for POC measurement. This model is used in connection with the POC module for automatic operation (POC-Modul "automatic"). The autosampler is equipped with 1 special canula for this.

Technical data

Maximal sample number	64
Maximum sample number (POC Sampler)	61
Sample vessels	40 ml
Operating voltage	24 V DC via external power supply
Power consumption	30 VA
Grid voltage of external power supply	100 to 240 V, 50/60 Hz (autosensing)
Dimensions (W x H x D)	500 x 560 x 400 mm

Layout



Fig. 39 EPA Sampler autosampler

- | | |
|------------------------------------|--------------------------------------|
| 1 Connection hoses to the analyzer | 2 Sample tray |
| 3 Wash cup | 4 Holding-down clamp |
| 5 Special canula | 6 Autosampler arm with canula holder |

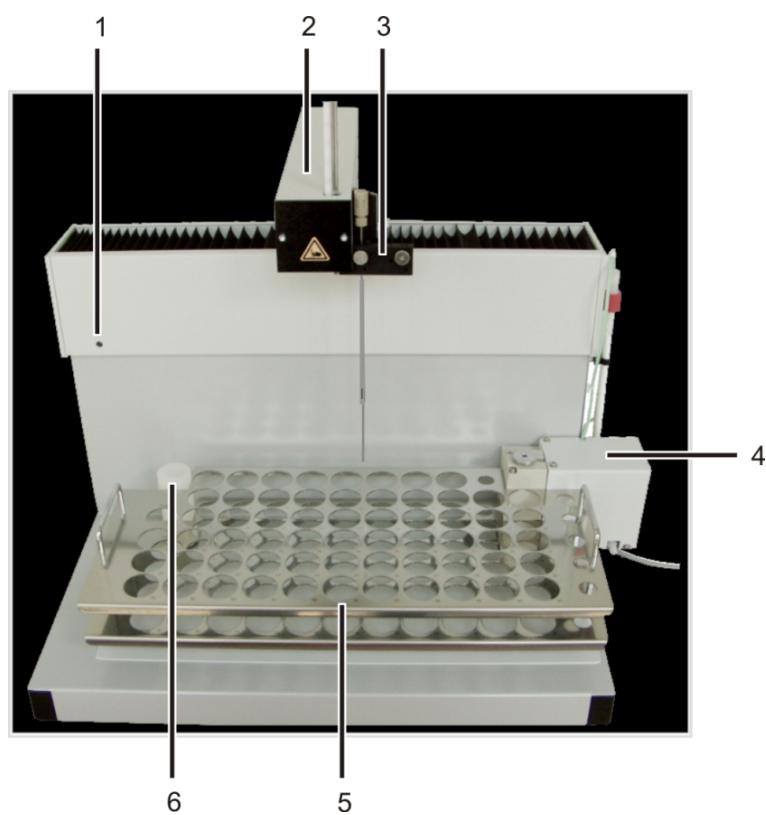


Fig. 40 POC Sampler autosampler

- | | |
|-----------------------------|-------------------|
| 1 Status LED | 2 Autosampler arm |
| 3 Canula holder with canula | 4 POC module |
| 5 Sample tray | 6 Wash cup |

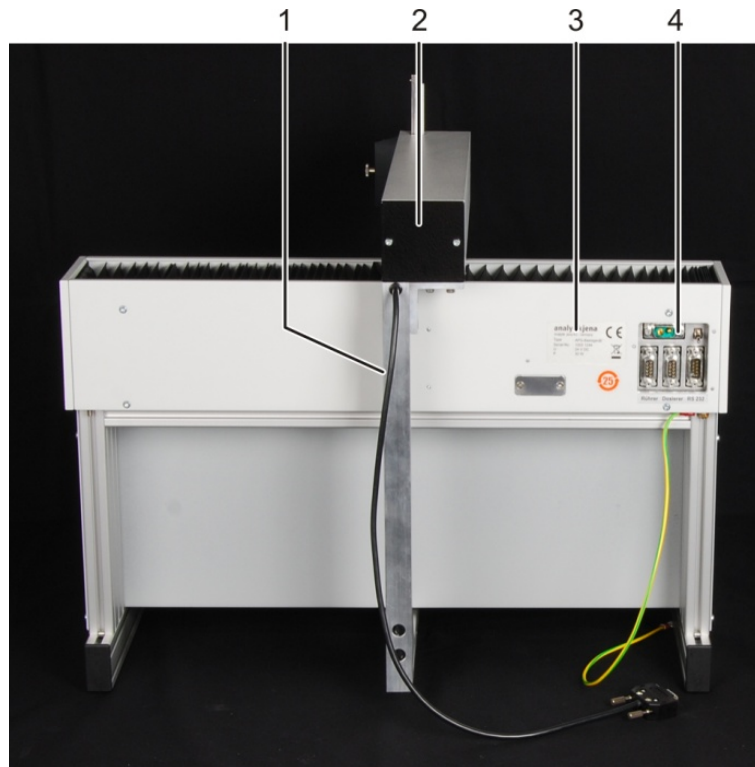


Fig. 41 Rear of the autosampler

- | | |
|----------------|--------------------------|
| 1 Stirring arm | 2 Autosampler arm |
| 3 Type plate | 4 Electrical connections |

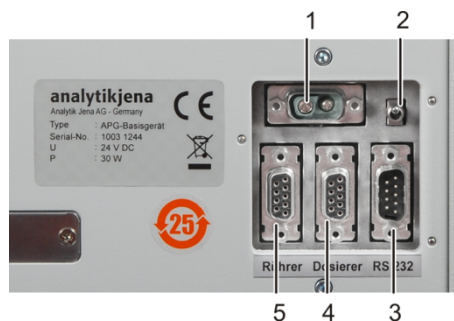


Fig. 42 Electrical connections

- | | |
|--------------------------------|-----------------|
| 1 Power supply unit connection | 2 Device switch |
| 3 Connection to the analyzer | 4 Not used |
| 5 Stirrer connection | |

Commissioning the autosampler

- Remove the transport lock:
 - Remove the two countersunk screws with the A/F3 hexagon head wrench supplied.
 - Remove the complete transport retaining clip and retain it for later transport.



Fig. 43 Transport lock

- | | |
|-------------------|----------------------------|
| 1 Autosampler arm | 2 Transport retaining clip |
| 3 Screws | |

- ▶ Install the stirring arm:
 - Install the stirring arm on the bracket on the rear of the autosampler arm.
- ▶ Screw on the arm with the supplied countersunk screws (M4x10) using the hexagon head wrench (A/F2.5).
 - Tighten the screws evenly to align the arm.
 - Connect the stirrer cable to the "Stirrer" connection on the rear of the autosampler.



Fig. 44 Installing the stirring arm

- | | |
|----------------------------------|----------------------|
| 1 Bracket on the autosampler arm | 2 Countersunk screws |
| 3 Stirring arm | |

- ▶ Place the autosampler next to the analyzer. Position the autosampler so that enough space is provided behind the device for the motion range of the autosampler arm as well.
- ▶ Connect the low voltage side cable of the table power supply unit to the rear of the autosampler. Do not connect the power supply unit to the mains power supply yet.
- ▶ Connect the supplied serial data cable to the "sampler" interface on the rear of the analyzer. Connect the other end of the data cable to the interface on the autosampler.
- ▶ Plug the grounding conductor into the connection on the rear of the analyzer.
- ▶ Connect the waste hose to the wash cup of the autosampler and to a suitable waste container or drain.
 - i** NOTICE! Route the outlet hose at a constant downward incline. If necessary, shorten the hose. The hose must not dip into the liquid.
- ▶ Install the wash cup on the autosampler.
- ▶ Place the sample tray onto the space provided.

- ▶ Note the positioning of the tray. The label must be legible when facing the front of the device. The two black centering pins on the contact surface of the autosampler protrude into the drill holes on the bottom of the tray.
- ▶ Insert the piercing canulas and holding-down clamps into the autosampler arm.
- ▶ Clamp the two canulas high enough in the holder to prevent them dipping into the vessels (basic position).

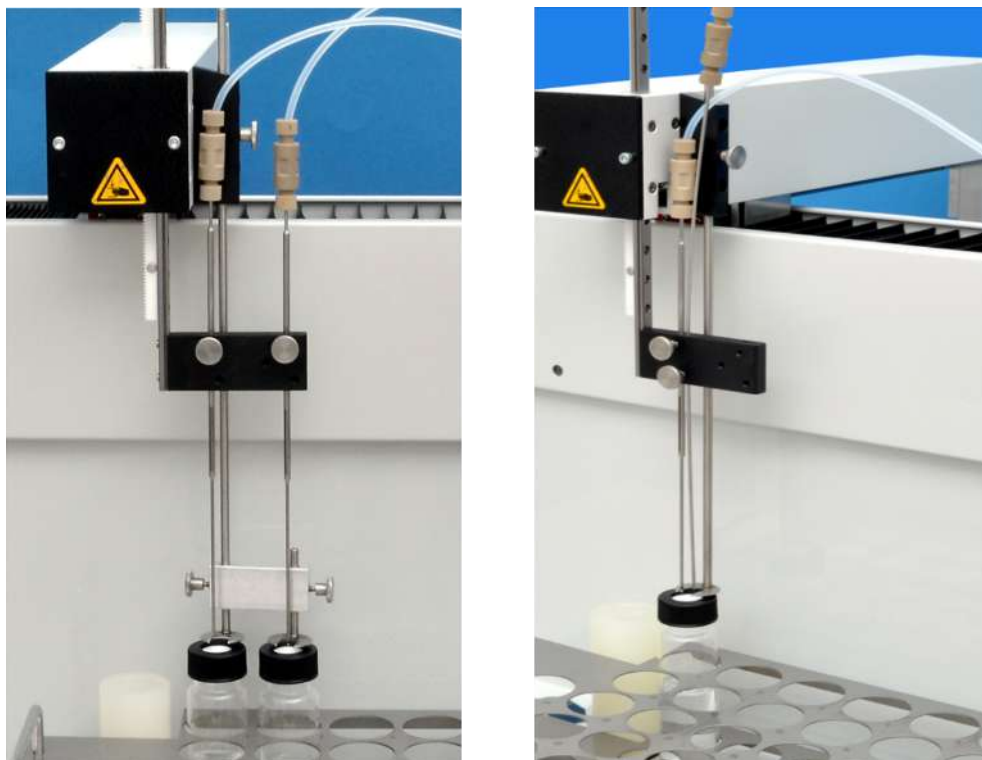


Fig. 45 Canula position for NPOC measurement with parallel (left) and non-parallel (right) purging.

- ▶ Secure the canulas by slightly tightening the knurled head screws.
- ▶ Connect the hoses from the analyzer to the canulas using Fingertight connections:
 - Hose AA – sample intake hose
 - Hose 7 – purging hose for NPOC measurement
 - To do this, guide the hose through the banjo bolt (see image).
 - Slide the conical nipple onto the hose with the conical side towards the banjo bolt. The conical nipple and hose must be flush.
 - Retighten the Fingertight connections.

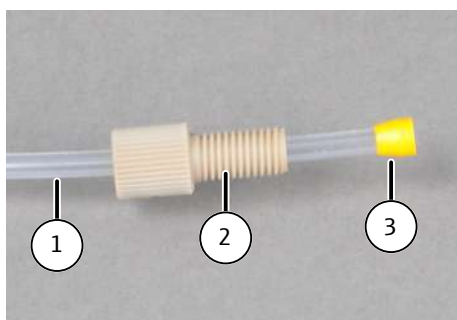


Fig. 46 Fingertight connection

- 1 Hose
- 3 Conical nipple

2 Banjo bolt

Checking and changing the configuration

- ▶ Alternatively for the POC Sampler: Install the POC module with the special canula.
- ▶ Connect the power supply unit to the mains network. Switch on the autosampler.
- ▶ Switch on the analyzer. Start the multiWin program and initialize the analysis system.
- ▶ Check the device configuration with the **Instrument | System-Information** menu option in the **multiWin set-up info** window.
- ▶ If necessary, change the configuration:
 - Exit the multiWin program.
 - Start the setup tool in the Windows interface with **Start | Program files | multiWin | multiWin setup tool**.
 - Select the autosampler type in the **Sampler** list.
 - Exit the **multiWin set-up tool** window with **[Create]**.
 - Start the multiWin program and initialize the analysis system.
 - Open the **Options** window with menu option **Configuration | Edit options**. Go to the **Analyzer components** tab.
 - In the **Sampler** group, select the appropriate tablet and vessel size.
- ▶ Exit the window with **[OK]**.
- ▶ Adjust the autosampler before the first start.

See also

- 📖 Adjusting the EPA Sampler [▶ 89]
- 📖 Installing the POC module [▶ 64]

4.3.4 POC module

The POC module is supplied in two versions:

- POC-Modul "basic" for manual operation
- POC-Modul "automatic" for automatic operation with the POC Sampler

Technical data

Parameter	POC
Digestion principle	Purging followed by thermal catalytic oxidation
Sample volume	1 ml
Sample supply	Flow injection, manual or automatic

Layout

The POC modules consist of the following components:

- POC reactor with septum
- Valve unit for automatic operation with autosampler
- Special sample intake canula for septum vials
- CO₂ adsorber with LiOH
- Sample tray with 61 positions (supplied with autosampler)

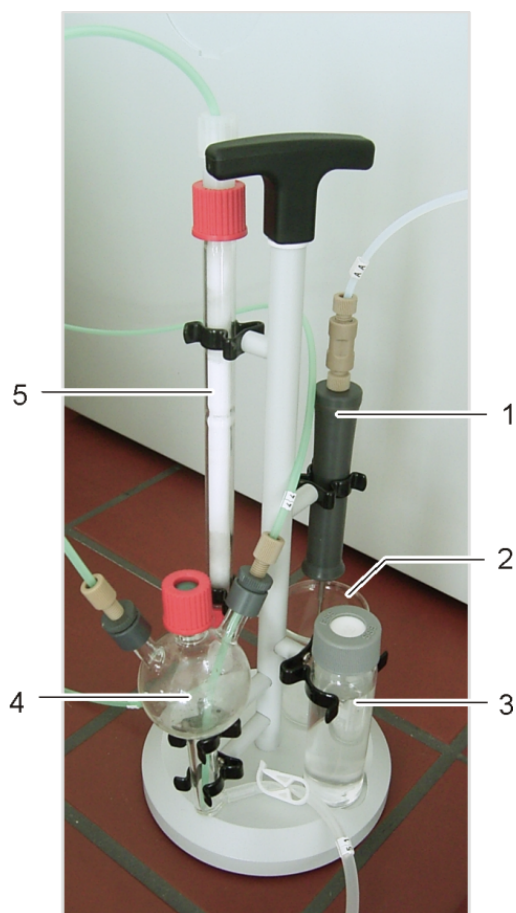


Fig. 47 POC module for manual operation

- | | |
|-----------------------------|-------------------|
| 1 Canula with handle | 2 Waste container |
| 3 Sample vessel with septum | 4 POC reactor |
| 5 Adsorption tube with LiOH | |

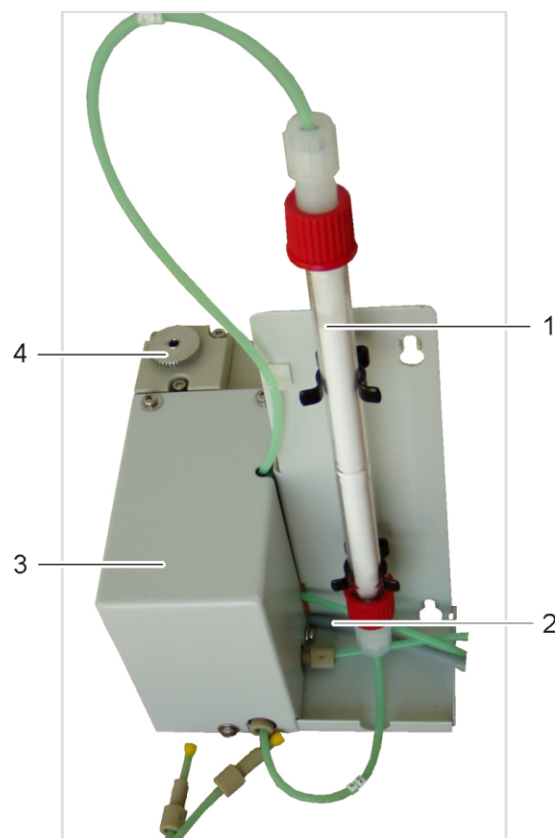


Fig. 48 POC module for automatic operation

- | | |
|-----------------------------|---|
| 1 Adsorption tube with LiOH | 2 Electrical connection to the analyzer |
| 3 Valve unit | 4 POC reactor |

4.3.4.1 Installing the POC module

Incorrect installation can create serious hazards. This may result in electric shock and explosion if the gases are not connected correctly.

- Only the Analytik Jena customer service or specialist personnel trained and authorized by them is allowed to install and commission the device and its system components.
- Unauthorized assembly and installation is not permitted.

Insufficiently secured components pose a risk of injury.

- During transport, secure the device components as specified in these operating instructions.
- Loose parts must be removed from the system components and packed separately.

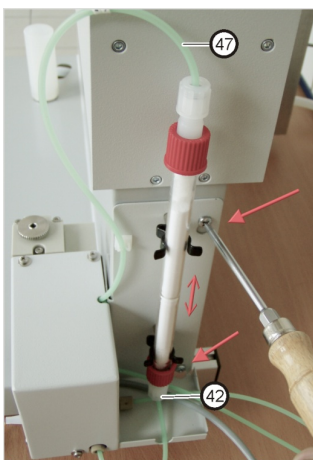
POC-Modul "automatic" installation

After connecting the POC module for automatic operation, the carrier gas will only flow through the POC reactor when measurement is carried out in POC mode.

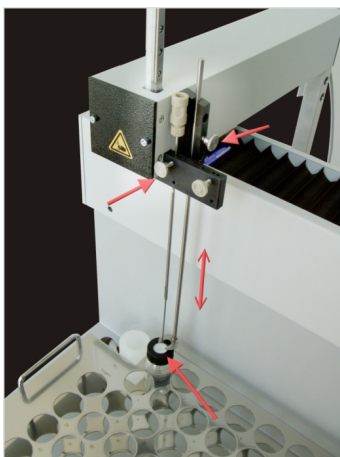
In all other operating modes (TC, NPOC, etc), the carrier gas flow is switched directly to the combustion tube in the analyzer.



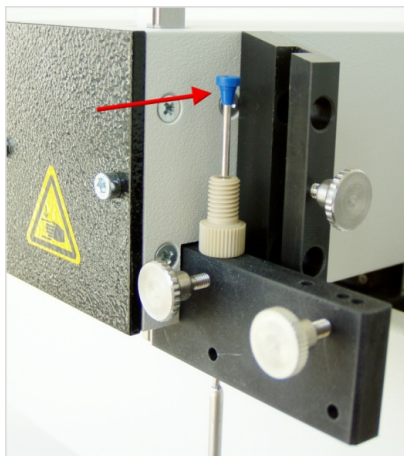
- ▶ Switch off the analyzer using the main switch on the rear of the device. Disconnect the power plug from the power supply. Switch off the autosampler.
- ▶ Loosen the housing screws on the right side of the autosampler.



- ▶ Install the filled CO₂ adsorber tube between hose 47 and 42.
- ▶ Press the adsorber tube into the clamps of the POC module.
- ▶ Attach the POC module by the housing screws. Retighten the screws.



- ▶ Slide the canula holder of the autosampler up.
- ▶ Clamp the special canula with ventilation slot into the canula holder of the autosampler.
- ▶ Install the hold-down clamp. With the canula holder moved up, the hold-ing-down clamp and the canulas must be approx. 3 mm above the sample vessels.



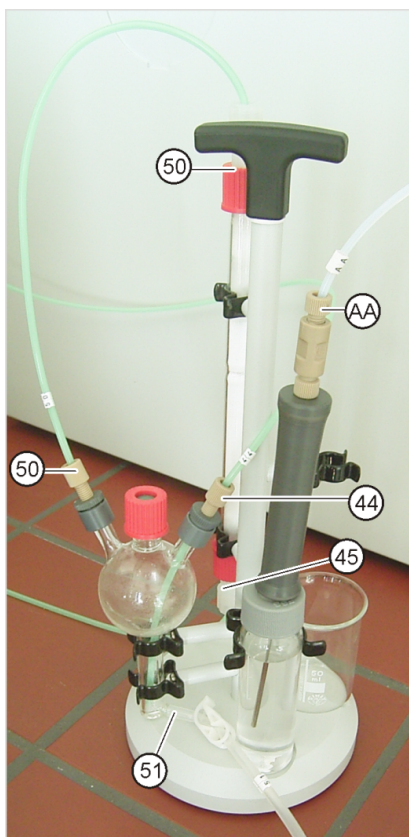
- ▶ Replace the standard intake canula with the special canula on hose AA. Position the Fingertight connection and the ferrules as shown in the adjacent image when doing this.
- ▶ Connect the POC module to the analyzer. Remove the hose protection bridge on the gas connections for the POC module on the rear of the analyzer.
- ▶ Connect hose 44 of the POC module with the 44 connection of the analyzer.
- ▶ Connect hose 45 of the POC module with the 45 connection of the analyzer.
⚠ WARNING! Do not confuse the gas connections!
- ▶ Connect the electrical connection of the POC module with the "POC" connection on the rear of the analyzer.



- ▶ Connect the waste hose to the waste connection of the POC module. Connect the hose to a suitable waste container or drain.
i NOTICE! Route the waste hose at a constant downward incline! i
NOTICE! If necessary, shorten the hose. The hose must not dip into the liquid.
- ▶ Adjust the autosampler.
- ▶ Check the system for leaks.

POC-Modul "basic" installation

After connecting the POC module for manual operation, the carrier gas flows constantly through the POC reactor into the combustion tube in the analyzer. It is best to remove the reactor when measuring in other operating modes (TC, NPOC, etc.).



- ▶ Place the module to the left of the analyzer.
- ▶ Install the CO₂ adsorber tube between hose 50 (top end of the adsorber tube) and hose 45 (bottom end).
- ▶ Connect hose 50 to the POC reactor.
- ▶ Connect the POC module to the analyzer. Remove the hose protection bridge on the gas connections for the POC module on the rear of the analyzer.
- ▶ Connect hose 44 with the POC reactor and the 44 connection of the analyzer. The hose will almost reach the bottom of the POC reactor when doing this.
- ▶ Connect hose 45 of the POC module with the 45 connection of the analyzer.
⚠ WARNING! Do not confuse the gas connections.
- ▶ Connect waste hose 51 to the bottom end of the TIC reactor. The waste hose is sealed with a hose clamp.
- ▶ Replace the standard intake canula on hose AA with the special canula with ventilation slot.

See also

- 📖 Checking the system for leaks [▶ 100]
- 📖 Adjusting the autosampler [▶ 92]

4.3.4.2 POC calibration

Recommendation: Perform calibration with sucrose.

- Procedure
- ▶ Create a POC method.
 - ▶ Open an NPOC method. Calibrate the NPOC method with sucrose standard solutions.
 - ▶ Link the measured calibration with the POC method:
 - ▶ After calibration, answer the following query with "no": "Link to the calibrated method?"
 - ▶ The methods list opens. Select the POC method in the methods list.
 - ▶ Accept the current calibration data for the POC method via the **[Accept values]** button.

Calibration can also be carried out with a dichloromethane standard solution. Dichloromethane's high volatility can lead to unreliable results, however. Recommendation: Calibrate the POC method directly by pipetting dichloromethane standard solution into the POC module.

4.3.5 Chemiluminescence detector (CLD)

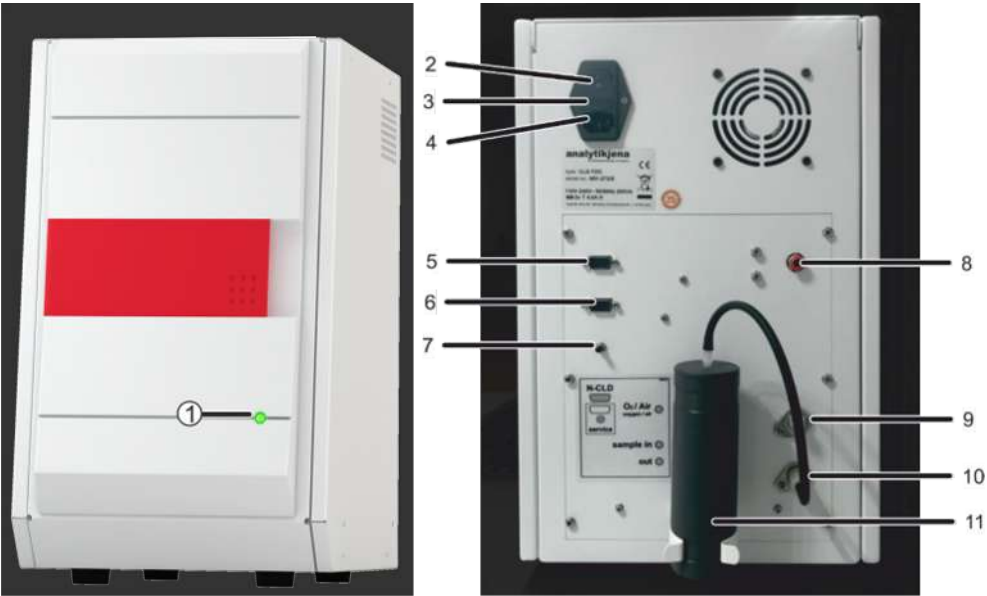


Fig. 49 Chemiluminescence detector (CLD)

- 1 Status LED

3 Fuse holder

5 RS 232 connection to the analyzer

7 Programming switch (service only)

9 "sample in" analyzer gas connection

11 Adsorber cartridge (removes NO_x from the waste air)
- 2 Mains switch

4 Power connection

6 Service connection

8 Carrier gas connection (O₂, synthetic/ purified air)

10 "out" sample outlet (gas)

Technical data	Detection principle	Chemiluminescence detector
	Parameter	TN _b (total bound nitrogen)
	Measurement range	0 to 20000 mg/l TN _b
	Detection limit	0,05 mg/l TN _b

Analysis time	3 to 5 min
Gas for ozone generation	Gas supply as for basic device 60 ml/min, 400 to 600 kPa
Dimensions (W x H x D), weight	300 x 470 x 550 mm , 12.5 kg
Electrical connection	110 to 240 V, 50/60 Hz
Fuses	2 T4.0 A H
Typical average power consumption	200 VA
Analyzer interface	RS 232



CAUTION

Risk of poisoning due to ozone

The ozone generator contained in the device produces ozone (O₃). When used in accordance with the intended use, the downstream ozone decomposer decomposes the poisonous gas. Various safety measures result in the automatic shut-down of the ozone generator. The following nevertheless applies:

- If there is a sharp smell of ozone, switch the device off immediately and inform customer service.
- To guarantee perfect and safe operation, Analytik Jena recommends annual inspection and maintenance by customer service.

Installation on the analyzer

- ▶ Set up the detector next to the analyzer.
- ▶ Connect the carrier gas to the gas connection with quick-release coupling.
- ▶ Set up the gas connection between the detector and the analyzer:
 - "sample in" connection on the detector
 - "CLD" connection on the analyzer
- ▶ Connect the "CLD/HT" interface on the rear of the analyzer with the RS 232 interface on the detector via the supplied serial data cable.
- ▶ Switch on the detector. The status LED indicates operational readiness.

Checking and changing the configuration

- ▶ Switch on the analyzer and the detector. Start the multiWin program and initialize the analysis system.
- ▶ Check the device configuration with the **Instrument | System-Information** menu option in the **multiWin set-up info** window.
- ▶ If necessary, change the configuration:
 - Exit the multiWin program.
 - Start the setup tool in the Windows interface with **Start | Program files | multiWin | multiWin setup tool**.
 - Select the detector (**CLD-TOC**) in the **N-measurement** list.
 - Exit the **multiWin set-up tool** window with **[Create]**.
 - Start the multiWin program and initialize the analysis system.
 - Open the **Options** window with menu option **Configuration | Edit options**. Go to the **Analyzer components** tab.
- ▶ In the **Sensors** group, activate nitrogen measurement with the **N-measurement active** option.
- ▶ Exit the window with **[OK]**.

4.3.6 External solids module



NOTICE

Observe accessory instructions

This accessory has separate instructions containing important information and measures for hazard prevention.

- Observe the separate instructions for the accessory during installation.

Installation of the modular multi N/C 3100 duo measurement system for automated solids analysis is described in the separate operating manual for the HT 1300 solids module.

Connection to the analyzer

- ▶ Set up the solids module next to the analyzer.
- ▶ Connect the "analyte" connection on the solids module to the "analyte" connection on the rear of the analyzer.
- ▶ Connect the "pump" connection on the solids module to the "pump" connection on the rear of the analyzer.
- ▶ Connect the connection hose for oxygen to the gas supply pressure reducer and to the "oxygen" gas connection on the rear of the solids module. Set an inlet pressure of 200 to 400 kPa on the pressure reducer.
- ▶ Connect the supplied serial data cable to the "CLD/HT" connection on the rear of the analyzer. Connect the other end of the data cable to the solids module.
- ▶ Start the multiWin program. Switch on the components of the analysis system. Initialize the analysis system.
- ▶ Open the **Options** window with menu option **Configuration | Edit options**. Go to the **Analyzer components** tab.
- ▶ Activate the **External solids module** option.
- ▶ Exit the window with **[OK]**.



Fig. 50 Connections on the backplate of the solids module

- | | |
|------------------------------|--------------------------------------|
| 1 Analyzer interface | 2 Mains connection |
| 3 Measuring gas outlet "OUT" | 4 Oxygen inlet "O ₂ " |
| 5 Pump connection "pump" | 6 Measuring gas connection "analyte" |

5 Operation

5.1 General notes



WARNING

Risk of chemical burns from concentrated acids

Concentrated acids are highly corrosive and sometimes have an oxidizing effect.

- Wear safety goggles and protective clothing when handling concentrated acids. Work under an extractor.
- Observe all instructions and specifications in the safety data sheets.

-
- When analyzing samples with high acidic or saline content, aerosols can form in the TIC condensation vessel. The capacity of the halogen trap is then depleted relatively quickly. The water trap also clogs up quickly. Both components have to be replaced frequently if this is the case. If possible, dilute such samples before measurement, for example 1:10.
 - When significant aerosol formation occurs, the analyzer is immediately protected by the integrated aerosol trap (water trap) and the carrier gas supply is automatically interrupted. Additionally, to protect the analyzer, remove the hose of the water trap on the front side.
 - To acidify samples, use analytically pure acid (HCl (2 mol/l)) and make it out of concentrated acid and TOC water.
 - For TIC detection, only use orthophosphoric acid (H₃PO₄, 10 %) made from concentrated acid (p.a.) and TOC water.
 - Solutions made from the following are suitable as standard solutions: Potassium hydrogen phthalate, sodium carbonate/sodium hydrogen carbonate, sucrose.
 - Only clean, particle-free glass containers (volumetric flasks, sample vessels) may be used for the preparation and storage of the solutions.
 - When preparing and storing solutions with very low concentrations (<1 mg/l), observe that the laboratory air components (CO₂, organic vapors) can change the solution concentrations. The following measures can remedy this:
 - Keep the free space above liquids, the so-called headspace, as small as possible.
 - During autosampler operation, cover the vessels on the sample tray with foil. This is important in particular with differential mode, as the samples remain on the sample tray for a longer time.
 - Eliminate the source of organic vapors.
 - Optionally: Fill the headspace above the samples with inert gas.

5.2 Switching on the analyzer



NOTICE

Risk of device damage due to depleted copper wool

Damage to optical and electronic components of the analyzer due to aggressive combustion products when the copper wool in the halogen trap is depleted!

- Only use the device with an operational halogen trap!
- Replace the complete filling of the halogen trap when half of the copper wool or brass wool is discolored!

Before switching on the analyzer, check the following:

- The waste hose is connected to a suitable waste container or drain. Free flow is ensured. The capacity of the waste container is sufficient.
- The gas supply is connected in accordance with regulations and the inlet pressure is 400 to 600 kPa.
- There is enough phosphoric acid in the reagent bottle. A volume of 0.5 ml acid is required per TIC determination.
- The halogen trap is connected, filled with copper and brass wool. The copper and brass wool not used up.
- All hoses are properly connected and in good working order.
- All optional accessories (autosamplers, solids modules, etc.) are connected.

Prepare the sample and switch on the analyzer as follows:

- ▶ Open the valve on the pressure reducer of the gas supply.
- ▶ Switch on the PC.
- ▶ Switch on the other optional components.
- ▶ Switch on the analyzer via the main switch. The status LED on the left front door lights up in green.
- ▶ Start the control and analysis software on the PC. Log in with username and password.
- ▶ Answer the query **Initialize analyzer?** with **[Yes]**.
 - ✓ After successful login, the software initializes the analysis system and queries the connected components. The measuring gas flow reaches its target value (160 ml/min) after approximately 1 to 2 min.
- ▶ If the analyzer is not ready for measurement after 30 min and one or more components are still displayed in red in the **System state** window:
 - Check the hose connections for proper fit and check for errors.
- ▶ For NPOC measurements: Set the NPOC purge flow.

The NPOC purge flow has been preset to 50 to 160 ml/min. The purge flow can be increased or decreased dependent on the measuring task.

 - ✓ The analyzer is ready to measure.

See also

- 📖 Troubleshooting [▶ 117]
- 📖 Setting the NPOC purge flow [▶ 94]

5.3 Switching off the analyzer

Switching to standby mode

Recommendation: Switch the analyzer to standby mode during measurement pauses of more than 30 min.

- ▶ Click on **[Exit]** in the software interface.
 - ✓ The **Program End** window opens.
- ▶ Flush the analysis device, for measurements without autosampler:
 - Activate the **Reverse Rinse Analyzer** checkbox. Place the sample intake canula into the waste container before starting the backflush.
- ▶ Flush the analysis device, for measurements with AS vario, EPA Sampler autosampler:
 - Activate the **Reverse Rinse Analyzer** checkbox. The content of the sample intake hose is automatically washed back into the wash cup.
- ▶ Flush the analysis device, for measurements with AS 10e, AS 21hp autosampler:
 - At the end of the sequence, measure one sample of ultrapure water.
(The autosamplers do not have the wash cup required for the backflush process.)
 - Activate the **Reverse Rinse Analyzer** checkbox.
- ▶ Activate the **Stand-by Analyzer** option and confirm with **[OK]**.
 - ✓ The analyzer remains in standby mode.

If backflushing is enabled, the sample intake hose is flushed with ultrapure water. The gas flow is switched off and the standby temperature is set.

Switching off the device before extended standstill periods

Recommendation: Switch off the analyzer before extended standstill periods (weekends, holidays).

- ▶ Click on **[Exit]** in the software interface.
 - ✓ The **Program End** window opens.
- ▶ Flush the analysis device, for measurements without autosampler:
 - Activate the **Reverse Rinse Analyzer** checkbox. Place the sample intake canula into the waste container before starting the backflush.
- ▶ Flush the analysis device, for measurements with AS vario, EPA Sampler autosampler:
 - Activate the **Reverse Rinse Analyzer** checkbox. The content of the sample intake hose is automatically washed back into the wash cup.
- ▶ Flush the analysis device, for measurements with AS 10e, AS 21hp autosampler:
 - At the end of the sequence, measure one sample of ultrapure water.
(The autosamplers do not have the wash cup required for the backflush process.)
 - Activate the **Reverse Rinse Analyzer** checkbox.
- ▶ Activate the **Switch off Analyzer** option and confirm with **[OK]**.
 - ✓ The analyzer is switched off.

If backflushing is activated, the sample intake hose is backflushed with ultrapure water. The TIC condensate container is pumped out. The analyzer shuts down. The gas flow is switched off and the furnace cools down.

5.4 Carrying out calibration

5.4.1 Preparing and starting calibration

The control and analysis software provides the option to adjust the analysis to the individual measuring task by selecting the method. An ideal measurement with its corresponding method requires its own calibration for each analysis parameter and each measuring channel. Not all parameters in a method need necessarily be calibrated.

Three calibration functions can be stored for each parameter in a method.

Carry out the calibration as follows:

- ▶ Select the sample feed type in the **System state** window:
[manual] or [Sampler].
 - ✓ The analyzer is initialized.
- ▶ Open the **Measurement | Calibration** menu option.
- ▶ In the query that follows, select either the method to calibrate or a preexisting calibration table.
- ▶ Carry out the software instructions that follow.
 - ✓ The **Calibration - Data of new calibration** window opens.

No.	Rep.	c (NPOC) [mg/l]
2	1	0,000
3	1	0,000
4	1	0,000
5	1	10,000
6	1	20,000
7	1	30,000
8	1	40,000

Fig. 51 Data for new calibration

- ▶ Select the calibration type in the **Calibration Parameters** group:
 - Preferably, perform Multiple point calibrations with a **constant sample volume** and variable concentrations. The volume set in the method is entered automatically in the **constant sample volume** input field. Only change the presets if the injection volume of the standard solutions differs from the volume set in the method.
- ▶ For calibrations with a **constant concentration**, enter the concentration of the standard solution in the input field.
- ▶ Enter the number of calibration points in the **Number of standards** input field.
- ▶ Select the analysis parameters of the loaded method to calibrate in the **Analysis parameters** field.
 - Activate the IC and TC parameters individually for calibration of the NPOC plus parameter and higher concentrations of $c > 0.5 \text{ mg/l}$.

- ▶ Use the calibration for the TOC/NPOC parameter for the lower concentration range. A single point calibration is sufficient in the lower concentration range.
- ▶ The software displays the sample feed type under **Sample introduction**. The indication is for information only and cannot be modified.
- ▶ Select how to take the blank value of the prepared water of the standard solutions into account in the **Preparation blank** group
 - **Measure** selection field:
The content of the preparation water is measured separately immediately before calibration. To do this, place a vessel with preparation water at the first position on the autosampler.
With manual sample supply, the software sends a prompt to provide the preparation water.
 - **Enter** selection field:
Enter the content of the preparation water as a value.
Specify the normalized preparation water blank value **1 ml**. If the preparation water blank value is not taken into account, a **"0"** is entered in the input field.
- ▶ Complete the calibration table for each parameter in accordance with the standard solutions provided.
- ▶ In the **Rep.** column, the software automatically accepts the number of repeat measurements set in the method. If outlier selection is enabled in the method, the maximum number of repeat measurements is entered.
If necessary, change the number of repeat measurements for each standard solution manually.
- ▶ If the calibration table is to be used again later: Save the calibration table with the **CalibrationTable | Save CalibrationTable** or **Save CalibrationTable as** menu option. Calibration tables are automatically assigned the *.kaltab file extension and are stored under ...\\Calibration\\Tables.
- ▶ Click the **Start measurement** button. Carry out the software instructions that follow.
 - ✓ Depending on the selected method and sample supply type, further queries appear. With sample feed via autosampler, the **Current sample data** window opens.

Pos.	Activation	State sample	Sample ID	Method	Dimension	Sample type	Sal
1	▶		Cal_NPOC_reag_Ka_210705_1127_Kona00	NPOC_reag_Ka	c1 mg/l	Preparation blank	150
2	▶		Cal_NPOC_reag_Ka_210705_1127_Kona01	NPOC_reag_Ka	c1 mg/l	Calibration	150
3	▶		Cal_NPOC_reag_Ka_210705_1127_Kona02	NPOC_reag_Ka	c1 mg/l	Calibration	150
4	▶		Cal_NPOC_reag_Ka_210705_1127_Kona03	NPOC_reag_Ka	c1 mg/l	Calibration	150
5	▶		Cal_NPOC_reag_Ka_210705_1127_Kona04	NPOC_reag_Ka	c1 mg/l	Calibration	150
6	▶		Cal_NPOC_reag_Ka_210705_1127_Kona05	NPOC_reag_Ka	c1 mg/l	Calibration	150
7	▶		Cal_NPOC_reag_Ka_210705_1127_Kona06	NPOC_reag_Ka	c1 mg/l	Calibration	150
8	▶		Cal_NPOC_reag_Ka_210705_1127_Kona07	NPOC_reag_Ka	c1 mg/l	Calibration	150
9	▶		Cal_NPOC_reag_Ka_210705_1127_Kona08	NPOC_reag_Ka	c1 mg/l	Calibration	150
10	▶		Cal_NPOC_reag_Ka_210705_1127_Kona09	NPOC_reag_Ka	c1 mg/l	Calibration	150
11	▶		Cal_NPOC_reag_Ka_210705_1127_Kona10	NPOC_reag_Ka	c1 mg/l	Calibration	150
12	▶		Cal_NPOC_reag_Ka_210705_1127_Kona11	NPOC_reag_Ka	c1 mg/l	Calibration	150
13							
14							
15							
16							
17							

Fig. 52 Current sample data

- ▶ Activate the calibration standards in the **Current sample data** window. After this, exit the window with [✓].
- ▶ The **Measurement** window opens. Click the **[Start F2]** button.
 - ✓ The calibration process starts.

5.4.2 Displaying the calibration results

After all calibration measurements have been carried out, the software automatically opens the calibration report in the **Calibration - Calibration Settings** window. The calibration report can be edited. The calibration report can also be opened later with the **Data Evaluation | CalibrationReport | Selection CalibrationReport** menu option.

The **Calibration - Calibration Settings** window has the **Calibration data** tab and the **Calibration results** tab.

The **Calibration data** tab displays the respective calibration settings. You can enter a note via the **[Comment]** button. You can sign the calibration with **[Signature]**. In the multiWin pharma software, only calibrations with signing status "authorized" can be linked to a method.

The **Calibration results** tab compiles the results for each calibrated parameter.

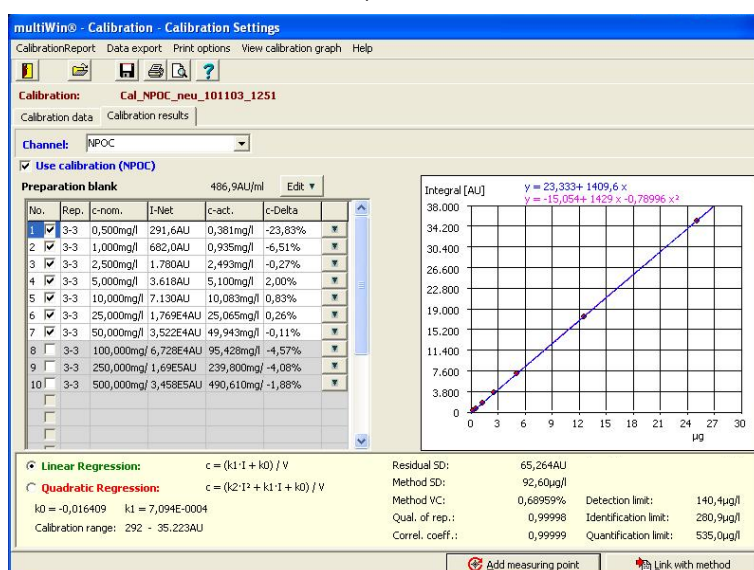


Fig. 53 Performed calibration data

Calibration results tab:

Results table	Display of: - Number of detections - Target concentration used (at constant sample volume), or - Sample volume used (at constant concentration) - Average value of the area integers - Average values of the calculated concentrations - Percentage deviation of the calculated concentration and the target concentration
Linear Regression / Quadratic Regression	Depending on the selected method, the software calculates the regression formula and the method characteristics for individual values or from the averaged values of the net integrals. The software displays the calibration coefficients for the selected regression type.
Calibration graph	In the View calibration graph menu, the display of the calibration graph can be switched: - x-axis – mass, y-axis – integral (calibration coefficient determination) - x-axis – integral, y-axis – mass (method characteristic determination)

Method characteristics	Verification/detection and determination limit: The software calculates the method characteristic data based on DIN 32645 (calibration function) with a significance level of $P = 95\%$. The software defines the determination limit for a relative results uncertainty of 33.3% (with $k = 3$ factor).
------------------------	--

5.4.3 Editing calibration

You can edit a calibration by:

- Selecting a regression type

You can select either linear or quadratic regression. The software displays the calibration coefficients and method characteristics for the selected regression type.

- Deactivating individual measuring points

All measuring points activated via check mark in the results table in the **No.** column are included in the regression calculation. You can deactivate a measuring point by removing the check mark. To do this, click in the **No.** column.

If you deactivate all measuring points, the software removes the check mark for the entire calibration channel.

- Deactivating individual measured values

You can view individual measured values by clicking on the button at the end of each line of the table. You can then deactivate individual measured values by removing the check mark in the **use** column.

- Enabling/disabling measured values for preparation water

You can view the individual measured value determined for the preparation water by clicking on the **[Edit]** button. You can then activate or deactivate individual measured values.

- Adding measuring points

You can add additional measuring points to an existing calibration. Perform a calibration measurement with the same method. When the measurement is complete, select the calibration report with the **[Add measuring point]**. Add the new measuring points individually to the calibration.

The software recalculates the calibration coefficients, method characteristics and the regression diagram after each change.

5.4.4 Linking a calibration to a method

Link the calibration parameters to a method as follows:

- ▶ Select the calibration range for the respective parameters (e.g., NPOC).
Up to three **linear calibration ranges** per parameter can be linked to a method. These ranges must overlap and be without gaps.
With **quadratic calibration functions**, only one calibration range can be linked to the method.
- ▶ Activate the **Use calibration** field with a check mark for each calibration range and analysis parameter to link with a method.
Not all calibrated parameters need to be transferred to the method.
- ▶ Click the **[Link with method]** button.
- ▶ Answer the following query: **Link with the calibration method?**
 - **[Yes]** (normal). The software links the calibration to the calibrated method.

- **[No]**. The software links the calibration to a method of your choice. The software does not check whether the method parameters of the calibration match those of the selected method here.
- ✓ The **Link with method: (...)** window opens.

The window displays the previous current calibration coefficient (right column) and the newly determined calibration coefficient (left column). The parameter display (e.g., IC/ NPOC) can be changed.

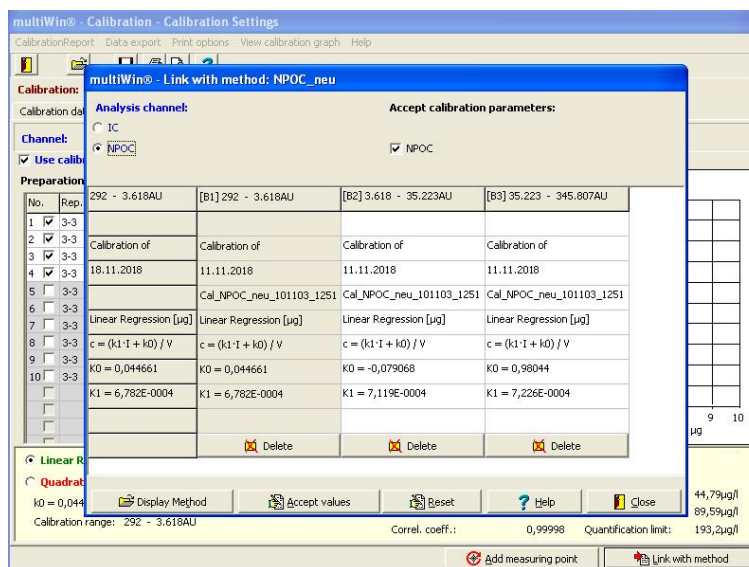


Fig. 54 Linking a calibration to a method (3 calibration ranges)

Whether the calibration ranges are accepted depends on the calibration ranges already stored in the method and the new calibration range:

No calibration range exists	The current calibration data is accepted with the [Accept values] button. The same calibration coefficients are displayed in the left and right columns.
One or two calibration ranges exist	Expanding existing calibration ranges: - Accept the new calibration data with the [Accept values] button. The software assigns the new range within the existing ranges. - Check that the ranges are linked without gaps. Replacing existing calibration ranges: - Delete the calibration range. - Accept the current calibration data with [Accept values].
Three calibration ranges exist	A maximum of three calibration ranges can be stored for each parameter in a method. If three calibration ranges already exist, the ranges can only be replaced: - Delete the desired range from the right column with the [Delete] button. - Accept the current calibration data with [Accept values]. - Check that the ranges are linked without gaps.

The following generally applies:

- After clicking **[Accept values]**, the software automatically assigns the calibration ranges.
- Pressing **[Delete]** makes an initial selection for the range to replace.

- Gapless linkage means: The largest area of the one calibration range matches the smallest area of the next calibration range.
- The software accepts the calibration parameters for all subsequent analyses carried out with the method.

5.4.5 Managing calibration data

Printing calibration data	▶ In the Calibration - Calibration Settings window, activate the Use calibration option. ▶ Define what to print in the Print options menu: – Print the calibration graph and/or – Print individual integrals for each calibrated channel ▶ Start printout with the CalibrationReport Print menu option.
Exporting calibration files	Calibration data is exported via the Data export menu in the Calibration - Calibration Settings window. You have the following options for exporting calibration data: ■ Calibration report to an export file The calibration report (with the *.ajc file extension) is saved to the ...\\Calibration export directory. ■ Export to a CSV file (with the *.csv file extension) The CSV file is stored in the preconfigured directory (default ...\\multiWin\\CSV). The directory is selected in the Options window in the Files and Directories tab. Open the window with the Configuration Edit options menu option. ■ Export to clipboard
Reopening a calibration report	▶ Click on the Data Evaluation CalibrationReport menu option in the main screen. ▶ Select the calibration report in the Selection CalibrationReport window. If necessary: Set filters in the Selection CalibrationReport window. Or sort the data records by clicking on a header. ▶ Mark the calibration report and click on the [OK] button. ✓ The calibration report is displayed.

5.5 Performing measurements

Note: In the multiWin pharma software, only methods with signing status **authorized** can be used for measurement.

5.5.1 Measurement with manual sample feed

- ▶ Insert the sample intake canula and the purging canula for NPOC measurements into the sample.
- ▶ Create a new method with the **Method | New** menu option.
- ▶ Alternatively: Load an existing method. Open the **Method selection** window with the **Method | Load** menu option. Mark the desired method. Confirm selection by clicking **[OK]**.
- ▶ Select manual sample supply by clicking **[manual]** in the **System state** window.
 - ✓ The software initializes the analyzer.

- ▶ Check the following displays in the **System state** window:
 - Optical bank
 - Nitrogen detector
 - Gas flow
 - Temperature
- ▶ If a display is red: Check for errors.
- ▶ Start the measurement: Click on **Start measurement**. Alternatively: Open the **Measurement | Measurement start** menu option.
 - ✓ The **Measurement start** window opens.
- ▶ Enter the sample ID and, if you want, a name for the analysis table. If necessary, enter the dilution, the sample type, the unit and a comment.
- ▶ Open the **Measurement** window with **[Start]**.
- ▶ Start measurement by clicking **[Start F2]**. Carry out the software instructions that follow.
- ▶ At the end of the measurement, the results appear in the analysis report or in the selected analysis table.

You can determine additional fields for entering sample information during manual measurements, such as sampling information, in the **Options** window in the **FreeStrings** tab.

The new fields are displayed in the **Measurement start** window in the **FreeStrings** tab. You can fill the fields with sample information.

See also

 Troubleshooting [▶ 117]

5.5.2 Measurement with autosampler



NOTICE

Risk of device damage

When the autosampler is maladjusted or not adjusted at all, the autosampling tool can hit a hard surface during operation. This can destroy the autosampling tool and the drive.

- Adjust the autosampler before it is used for the first time and after each modification as well as after transporting or storing it for a longer period of time.

- ▶ Create a new method with the **Method | New** menu option.
- ▶ Alternatively: Load an existing method. Open the **Method selection** window with the **Method | Load** menu option. Mark the desired method. Confirm selection by clicking **[OK]**.
- ▶ Select automatic sample feed by clicking **[Sampler]** in the **System state** window.
 - ✓ The software initializes the analyzer.
- ▶ Check the following displays in the **System state** window:
 - Optical bank
 - Nitrogen detector
 - Gas flow

- Temperature
- ▶ If a display is red: Check for errors.
- ▶ Fill the sample vessels with the sample solution. Place the sample vessels on the sample tray.
- ▶ For NPOC measurements with the AS vario: The autosampler can automatically acidify samples. To do this, fill the acid container with HCl (2 mol/l). Place the acid container at the acid position of the sample tray:
 - Position 28 on sample tray 47
 - Position 42 on sample tray 52
 - Position 55 on sample tray 72
 - Position 85 on sample tray 100
 - Position 131 on sample tray 146
 - The autosampler cannot simultaneously automatically acidify and dilute samples. For automatic acidification: Deactivate the **Use dilution algorithm** menu option under **Options | Process control**.
If the dilution algorithm is active, acidify the samples manually.
- ▶ For NPOC measurements with the EPA autosampler: Place the acid container at position 54 on the sample tray.
- ▶ For NPOC measurements with the POC autosampler: Place the acid container at position 51 on the sample tray.
- ▶ Start the measurement: Click on **Start measurement**. Alternatively: Open the **Measurement | Measurement start** menu option.
 - ✓ The **Measurement start** window opens.
- ▶ Enter a name for a new analysis table in the **Measurement start** window. Alternatively: Select an existing analysis table with **[Edit]**.
- ▶ Open the **Current sample data** window with **[Start]**.
- ▶ Open an existing rack table and add to it.
Or: Enter the sample ID in the **Sample ID** column in the new rack table. Check the sample tray assignment for this. If necessary, enter the dilution, the sample type, the unit and any comments.
- ▶ Activate all samples or activate each sample individually with **[>]**.
- ▶ Confirm the entries with **[✓]**.
 - ✓ The rack table is closed. A query follows whether the rack table should be saved.
- ▶ If the rack table is to be used again later: Answer with **[Yes]**. Save the rack table in the default window for saving files.
- ▶ Start the measurement with **[Start F2]** in the **Measurement** window. Carry out the software instructions that follow.
 - ✓ The analysis table displays the measurement results at the end of the measurement.

See also

📖 Troubleshooting [▶ 117]

5.6 Dilution

The analyzer can automatically dilute the samples on the autosampler. A special autosampler with a special sample tray is required for this: AS vario autosampler, 47 tray. If these are retrofitted, you will receive a keycode with the delivery of the dilution unit. After every change or modification to the analysis system, new activation with the keycode is required.

- ▶ Log on to the software as an administrator.
- ▶ Open the **Configuration | KeyCode** menu option.
 - ✓ The software closes automatically.
- ▶ When restarting the program: Enter the keycode in the prompt.
 - ✓ Automatic dilution is activated for the analyzer.

5.6.1 Automatic TC dilution

Use automatic dilution when measuring samples with very high TC content or with an unknown highly loaded sample matrix.

Automatic dilution provides the following advantages:

- You avoid unnecessarily loading the reactor with high inorganic salt and acid contents. This increases the service life.
- You save time due to automation.
- No extra calibration is required for the high concentration range.

Use automatic dilution as follows:

- ▶ Position the special dilution sample tray (47) and install the suitable canula holder on the autosampler.
- ▶ Open the **Edit options** window, **Analyzer components** tab with the **Configuration | Options** menu option.
- ▶ In the **Sampler** group, select tray size 47.
- ▶ Activate dilution in the **Process control** tab. To do this, activate the **Use dilution algorithm** field with a check mark.
- ▶ Activate the **Automatic dilution** option.
- ▶ Equip the sample tray with empty sample vessels (50 ml).
- ▶ Fill samples for dilution in the sample vessels (12 ml). Equip the sample tray with the samples.
- ▶ Fill samples that are not to be diluted in sample vessels (50 ml).
- ▶ Fill ultrapure water into the ultrapure water bottle.
- ▶ Open the window of the same name with the **Instrument | Sampler Alignment** menu option.
- ▶ Align the sample intake canula with the sample tray.
- ▶ Activate the **Dluent position** field. Adjust position 1 in a large sample vessel (50 ml).
- ▶ Check position 1 in a small sample vessel (12 ml).

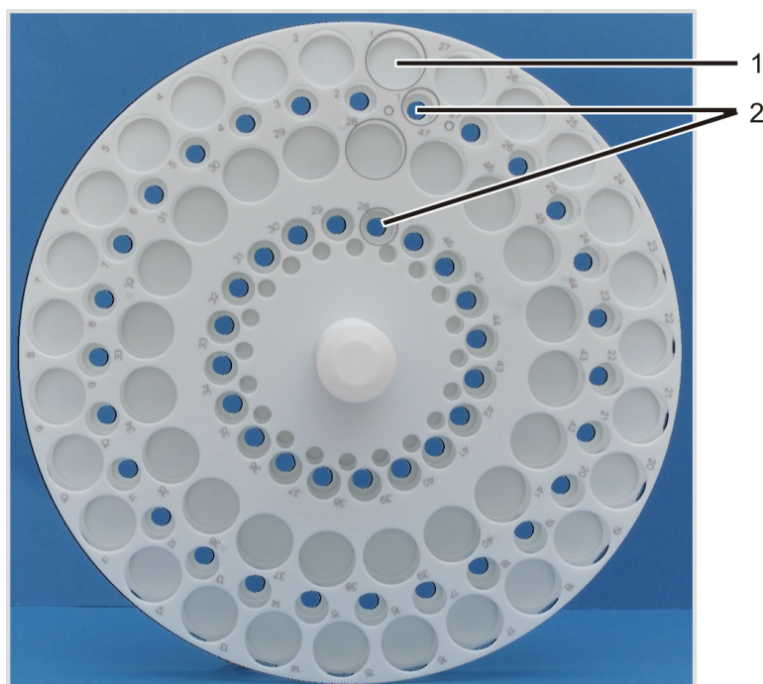


Fig. 55 Dilution tray

1 Position 1 to 47 for 50 ml vessels

2 Position 1 to 47 for 12 ml vessels

- ▶ If necessary, open the **Measurement | Preparation blank** menu option. Determine the blank value of the dilution water.
 - ✓ The process is determined internally. The water for the blank value determination is taken from the ultrapure water bottle.
- ▶ Generate the rack table again. Or: Load an existing rack table.
- ▶ Select the desired dilution rate in the **Dilution** field.
The following dilutions are possible: **1 in 5, 1 in 10, 1 in 25, 1 in 50, 1 in 100.**
- ▶ Start the measurement.

Observe the following for automatic dilution:

- The autosampler dilutes the original samples into the provided sample vessels (50 ml) at the selected dilution rate.
- When working in NPOC mode, the samples are each diluted in a complete series and then analyzed. The autosampler purges the diluted samples.
- If the dilution algorithm is active, the option for automatic acidification of a sample is deactivated in the software. For NPOC methods, you must therefore manually acidify the original samples. Alternatively, you can pipette acid into the large sample vessels the autosampler dilutes the samples into.

Check in both cases if the pH value of the sample is <2. Only then can the autosampler completely remove the inorganic carbon compounds (TIC) during purging.

- The number of possible multiple determinations is the result of the selected method, the injection volume and the number of flushing cycles. At least a threefold determination must be possible.

The software displays an error message if the sample volume of the diluted sample is insufficient. Adjust the method settings accordingly.

- The concentration of the primary sample is shown in the analysis report. The analysis report shows the surface integral measured for the diluted sample, however.

See also

- 📖 Adjusting the AS vario autosampler [▶ 87]

5.6.2 Intelligent TN dilution

Dilute highly concentrated samples for nitrogen detection to improve the recovery rate.

In dilution mode, the autosampler automatically dilutes samples with a content of $TN_b > 12$ mg/l. Intelligent dilution is particularly interesting for samples of unknown concentration or samples in an unknown sample matrix.

With intelligent dilution, the analyzer measures the original sample. After the first determination, the analyzer decides whether to automatically dilute the sample or to continue with repeat measurement based on the TN_b content.

The following threshold values apply to intelligent dilution:

- As of approx. $TN_b > 12$ mg/l: automatic 1 in 10 dilution
- As of approx. $TN_b > 120$ mg/l: automatic 1 in 20 dilution

The precise threshold value is dependent on the:

- Sample matrix
- Type of nitrogen compounds
- Sample volume
- Aging of the combustion tube

When working in dilution mode, calibrate the method up to $TN_b = 15$ mg/l. Assess the quality of the calibration function based on the regression coefficient. Also ensure that the actual concentrations deviate by a maximum of 5 % from the target concentrations across the entire calibration range. Only in this manner can precise results be achieved.

Use intelligent dilution as follows:

- ▶ Position the special dilution sample tray (47) and install the suitable canula holder on the autosampler.
- ▶ Open the **Edit options** window, **Analyzer components** tab with the **Configuration | Options** menu option.
- ▶ In the **Sampler** group, select tray size 47.
- ▶ Activate dilution in the **Process control** tab. To do this, activate the **Use dilution algorithm** field with a check mark.
- ▶ Select the **Intelligent dilution** option.
- ▶ Fill the samples into the sample vessels (50 ml). Equip the sample tray with the sample vessels.
- ▶ Place empty sample vessels (12 ml) on the sample tray.
- ▶ Fill ultrapure water into the ultrapure water bottle.
- ▶ Open the window of the same name with the **Instrument | Sampler Alignment** menu option.
- ▶ Align the sample intake canula with the sample tray.
- ▶ Activate the **Dluent position** field. Adjust position 1 in a large sample vessel (50 ml).
- ▶ Check position 1 in a small sample vessel (12 ml).

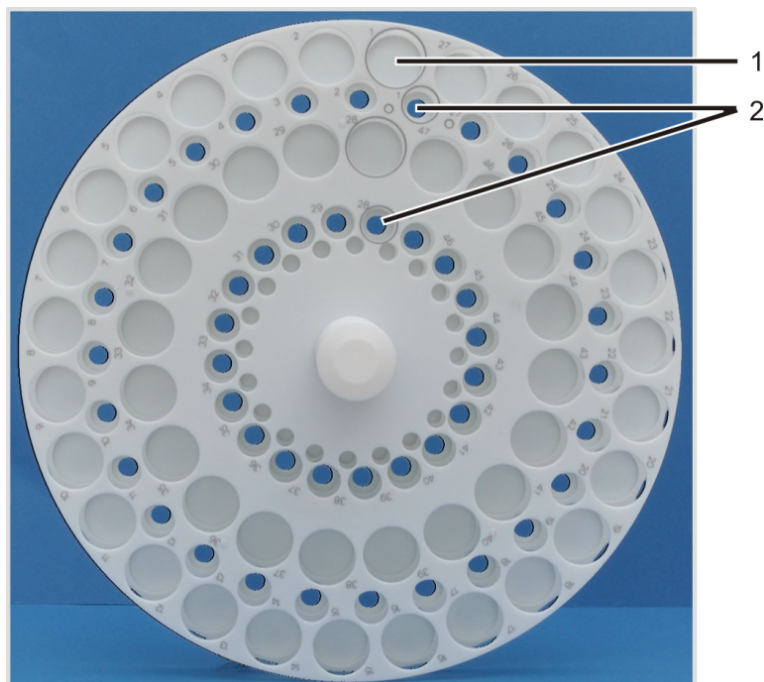


Fig. 56 Dilution tray

1 Position 1 to 47 for 50 ml vessels

2 Position 1 to 47 for 12 ml vessels

- ▶ If necessary, open the **Measurement | Preparation blank** menu option. Determine the blank value of the dilution water.
 - ✓ The process is determined internally. The water for the blank value determination is taken from the ultrapure water bottle.
- ▶ Generate the rack table again. Or: Load an existing rack table.
- ▶ The dilutions in the rack table do not refer to intelligent dilution and have no effect on this.
If you wish to dilute the samples – independent of intelligent dilution: Dilute the samples manually before measurement. Enter the dilution rate in the **Dilution** column of the rack table. The software then calculates the concentration of the undiluted samples automatically.
- ▶ Start the measurement.

Observe the following for intelligent dilution:

- The number of possible multiple determinations is the result of the selected method, the injection volume and the number of flushing cycles. At least a threefold determination must be possible.
The software displays an error message if the sample volume of the diluted sample is insufficient. Adjust the method settings accordingly.
- The concentration of the primary sample is shown in the analysis report. The analysis report shows the surface integral measured for the diluted sample, however.
- If the analyzer carries out intelligent dilution of a sample, the existing ID of the diluted sample is appended:
 - `_iV_1_10` for 1 in 10 dilutions
 - `_iV_1_20` for 1 in 20 dilutions

The diluted sample appears immediately after the original sample in the analysis table.

- Intelligent dilution is only intended for TN_b detection and not for TC detection. Intelligent dilution is only triggered by the threshold values for TN. Even if you have selected carbon determination in the method, the analyzer will only determine the carbon content (TC) when the threshold values are exceeded. The TC is then determined from the diluted samples.
- Diluted samples are not purged. For the precise determination of TC or TOC, perform a separate analysis run with no dilution or with automatic dilution (TC).
- Do not simultaneously determine TOC in differential mode with intelligent dilution. The sample volume in the small sample vessels (12 ml) is insufficient for a threefold determination in differential mode.

See also

-  Adjusting the AS vario autosampler [► 87]

6 Maintenance and care

The operator may not undertake any service or maintenance work to this device and its components other than that specified in these instructions.

Observe the information in the "Safety instructions" section for all maintenance work. Compliance with the safety instructions is a prerequisite for the error-free operation of the device. Always observe all warnings and instructions that are displayed on the device itself or indicated by the control software.

To ensure faultless and safe functioning, Analytik Jena recommends an annual inspection and servicing by its Service department.

6.1 Maintenance overview

Analyzer

Maintenance interval	Maintenance task
Weekly	▪ Clean and maintain the device. ▪ Clean the reagent bottle and the drip tray. ▪ Check the fastening screws for tight fit.
Every 12 months	▪ Replace the battery of the electrochemical NO detector (ChD, optional) in the right side part of the analyzer.

Sample supply system and autosampler

Maintenance interval	Maintenance task
Quarterly	▪ Check the syringe pump for leaks.
Every 12 months	▪ Clean the dosing syringe (earlier if required).
As required	▪ After initial start, change of the sample tray or recommissioning after transport and storage: Adjust the autosampler.

Hose system

Maintenance interval	Maintenance task
Daily	▪ Check the gas flow display in the System state window.
Weekly	▪ Check the hose connections for tight fit.
Quarterly	▪ Check the condensate and phosphoric acid pumps for leaks.
Every 12 months	▪ Replace the pump hose.

Combustion system

Maintenance interval	Maintenance task
Every 12 months	▪ Replace the combustion tube (earlier if required). ▪ When the combustion tube is replaced: Replace the catalyst.
When necessary	▪ After software notification at the latest: Check the catalyst for effectiveness and replace it. ▪ When the catalyst is replaced: Check the combustion tube for damage and clean it.

Measuring gas drying and cleaning

Maintenance interval	Maintenance task
Daily	- Check the filling of the halogen trap. - When half of the copper wool or the brass wool is discolored, replace the filling.
Quarterly	Check the TIC condensate container and the condensation coil for cracks and damage.
Every 6 months	Replace the water traps on the front and on the gas box.
Every 12 months	Clean the TIC condensate container and the condensation coil (or earlier if necessary).

POC module

Maintenance interval	Maintenance task
Monthly, earlier if required	- Check the adsorber for proper function. - Check the module for leaks. - Replace the septum on the POC port.
When necessary	If the adsorber material clumps, replace the adsorber.

Chemiluminescence detector (CLD)

Maintenance interval	Maintenance task
Every 12 months	Replace the adsorber cartridge.

6.2 Adjustment and setting

6.2.1 General notes for adjusting the autosampler

During adjustment, the canulas are adjusted to the sample tray for optimum immersion in the sample vessels and/or wash cups.

An adjustment of the autosampler is necessary:

- Before the first start
- After each change of the sample tray
- During recommissioning after transport or storage

Adjustment of the AS 10e and AS 21hp autosamplers is described under Installation and Commissioning.

See also

- Installing and commissioning the sampler [► 47]

6.2.2 Adjusting the AS vario autosampler



NOTICE

Risk of bending

The canula may bend during adjustment.

- Unscrew the screw connections on the canulas before adjustment.

- ▶ Start the software. Wait for the device to initialize.
- ▶ Open the window of the same name with the **Instrument | Sampler Alignment** menu option.
- ▶ Select **needle** in the list field in the **Please select position needing adjustment** group.
 - ✓ The autosampler arm moves over the adjustment points on the sample tray.

Fig. 57 Adjusting the autosampler

- ▶ Increase or decrease the z-axis values until the canulas are approx. 2 cm above the adjustment points. Click the **[needle adjust]** button.
- ▶ Align the canulas to the two adjustment points by carefully bending them.



Fig. 58 Adjustment points on the sample tray

- ▶ Adjust the immersion depth of the sample intake canula into the wash cup and into a sample vessel in position 1 of the sample tray.
- ▶ To do this, select the **Rinse position** or the **Position 1** entry in the **Please select position needing adjustment** group.
- ▶ To adjust position 1, place a sample vessel with a magnetic stirring rod onto the sample tray.
- ▶ To adjust the flushing position or position 1, increase or decrease the z-axis values.

- ▶ Set the canulas to the flushing position so that the canulas are immersed by at least 1 cm in the wash cup.
- ▶ For the AS vario ER autosampler: Set the maximum z-axis value of 145 mm at the flushing position. The canulas will then be sufficiently immersed in the wash cup during flushing.
- ▶ Set the canula heights at position 1 so that the stirring rod can rotate unhindered (approx. 5 mm clearance).
- ▶ After each change, click on **[Rinse position adjust]** or **[Position 1 adjust]**.
 - ✓ The autosampler will move to the new position.
- ▶ Repeat these steps until the canula position is optimized.
- ▶ Click on **[Save]** to save the adjustment values.
- ▶ Open the **Sampler Alignment** window again. Move to the flushing position and position 1 with the buttons to check.
 - ✓ The autosampler is adjusted.

Adjusting the autosampler for automatic acidification

The autosampler can automatically acidify samples for NPOC measurement. The immersion depth of the canula in the sample vessel depends on the z-axis direction adjustment for position 1.

- ▶ Adjust the canulas at position 1.
- ▶ Check the adjustment values via a test measurement.
- ▶ Ensure the canula goes through the sample lid but does not immerse in the sample liquid during acidification.

6.2.3 Adjusting the EPA Sampler



NOTICE

Risk of bending

The canula may bend during adjustment.

- Unscrew the screw connections on the canulas before adjustment.

Clamp the two canulas high enough in the holder to prevent them dipping into the vessels (basic position).

During adjustment, the sample intake canula must be adjusted to the rinse position and to sample position 1. Alignment is carried out by increasing or decreasing the x-, y- and z-axis values.

For sample vessels with septum caps, special sample intake and purging canulas with piercing function are required: Piercing needles with ventilation slot.

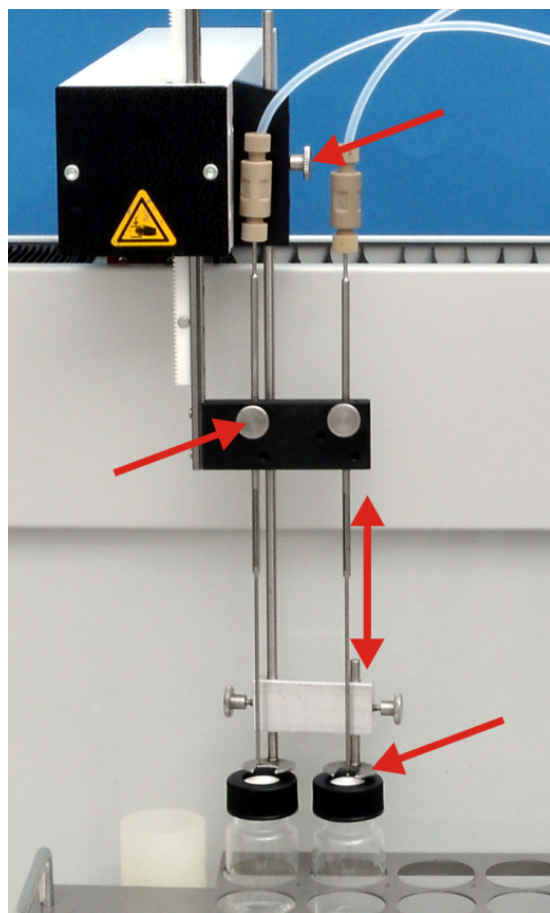


Fig. 59 Installing canulas (2 canulas for parallel purging here)

- ▶ Install the hold-down clamp and the sample intake canulas in the canula holder. Unscrew the retaining screws of the canulas before adjustment. Clamp the canulas into the holder so that the canula tip does not immerse in the sample vessel.
- ▶ Open the window of the same name with the **Instrument | Sampler Alignment** menu option.
- ▶ Adjust the immersion depth of the sample intake canula into the wash cup and into a sample vessel in position 1 of the sample tray.
- ▶ To do this, select the **Rinse position** or the **Position 1** entry in the **Please select position needing adjustment** group.

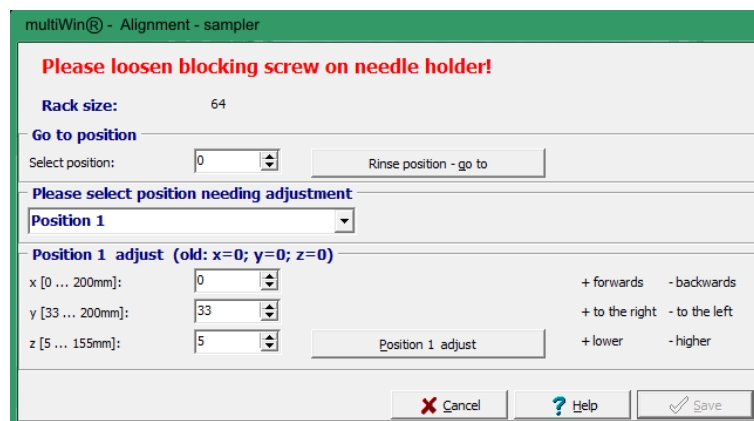


Fig. 60 Adjusting the autosampler

- ▶ Increase or decrease the x-, y- and z-axis values to align the flushing position or position 1:

- X-axis direction: Forward or backward movement
- Y-axis direction: Left or right movement
- Z-axis direction: Up or down movement



Fig. 61 Adjusting position 1

- ▶ Adjust position 1: To adjust the x-axis and y-axis positions, remove the sample vessel. Move to the position. Place the magnetic stirring rod on the sample tray at this position. If the canula is in the middle of this position, it has been adjusted correctly.
 - The y-axis value must not be smaller than 33 mm to ensure correct operation.
- ▶ To adjust the z-axis direction, place a sample vessel with a screw cap and a septum cap, e.g., an EPA sample vessel, on the sample tray.
- ▶ Adjust the special needle in the z-axis direction so that approx. 2 cm of the ventilation slot are visible above the septum. The ventilation slot must be located above and below the septum. No pressure compensation is otherwise possible in the sample vessel.
- ▶ Adjust the flushing position: Adjust the x- and y-axis positions so that the canula is located in the middle of the wash cup.
- ▶ Adjust the special canula in the z-axis direction so that the ventilation slot can be seen at the upper edge of the wash cup.
- ▶ After each change, click on **[Rinse position adjust]** or **[Position 1 adjust]**.
 - ✓ The autosampler will move to the new position.
- ▶ Repeat these steps until the canula position is optimized.
- ▶ Click on **[Save]** to save the adjustment values.
- ▶ Open the **Sampler Alignment** window again. Move to the flushing position and position 1 with the buttons to check.
 - ✓ The autosampler is adjusted.

Adjusting the autosampler for automatic acidification

The autosampler can automatically acidify samples for NPOC measurement. The immersion depth of the canula in the sample vessel depends on the z-axis direction adjustment for position 1.

- ▶ Adjust the canulas at position 1.
- ▶ Check the adjustment values via a test measurement.
- ▶ Ensure the canula goes through the sample lid but does not immerse in the sample liquid during acidification.

6.2.4 Adjusting the autosampler



NOTICE

Risk of bending

The canula may bend during adjustment.

- Unscrew the screw connections on the canulas before adjustment.

During the adjustment, the sample intake canula must be adjusted to the flushing position, to sample position 1 and to the POC reactor position on the sample tray. Alignment is carried out by increasing or decreasing the x-, y- and z-axis values.

A special sample intake canula with piercing function is required for sample vessels with septum caps: Piercing needle with ventilation slot.

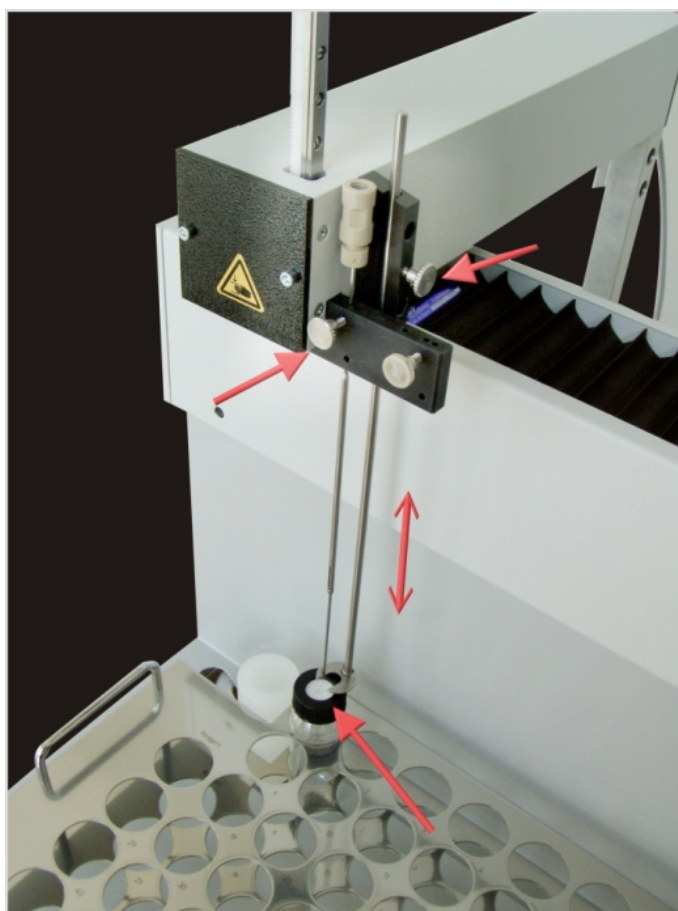


Fig. 62 Installing the canula and hold-down clamp

- ▶ Install the hold-down clamp and the sample intake canula in the canula holder. Unscrew the retaining screws of the canula before adjustment. Clamp the canula into the holder so that the canula tip does not immerse in the sample vessel.
- ▶ Open the window of the same name with the **Instrument | Sampler Alignment** menu option.
- ▶ To do this, select the **Rinse position, Position 1** or **Position POC reactor** entry in the list field in the **Please select position needing adjustment** group.

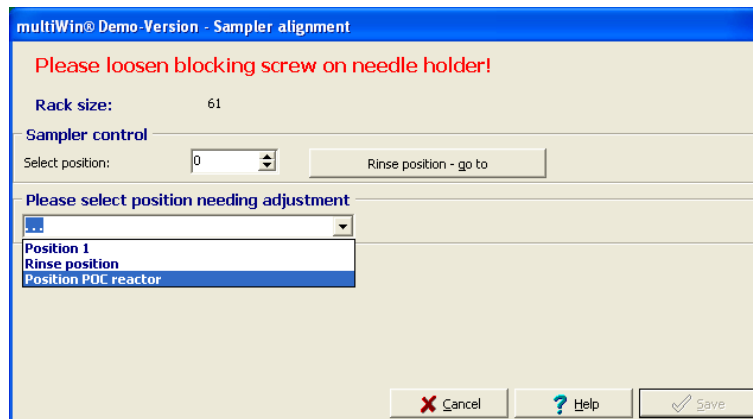


Fig. 63 Adjusting the autosampler

- ▶ Increase or decrease the x-, y- and z-axis values to align the flushing position or position 1:
 - X-axis direction: Forward or backward movement
 - Y-axis direction: Left or right movement
 - Z-axis direction: Up or down movement

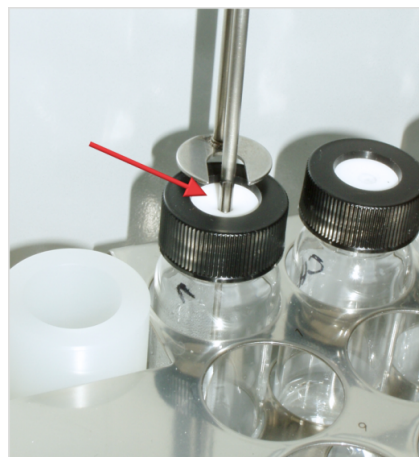


Fig. 64 Adjusting position 1

- ▶ Adjust position 1: To adjust the x-axis and y-axis positions, remove the sample vessel. Move to the position. Place the magnetic stirring rod on the sample tray at this position. If the canula is in the middle of this position, it has been adjusted correctly.
 - The y-axis value must not be smaller than 33 mm to ensure correct operation.
- ▶ To adjust the z-axis direction, place a sample vessel with a screw cap and a septum cap, e.g., an EPA sample vessel, on the sample tray.
- ▶ Adjust the special needle in the z-axis direction so that approx. 2 cm of the ventilation slot are visible above the septum.
The ventilation slot must be located above and below the septum. No pressure compensation is otherwise possible in the sample vessel.
- ▶ Adjust the flushing position: Adjust the x- and y-axis positions so that the canula is located in the middle of the wash cup.
- ▶ Adjust the special canula in the z-axis direction so that the ventilation slot can be seen at the upper edge of the wash cup.

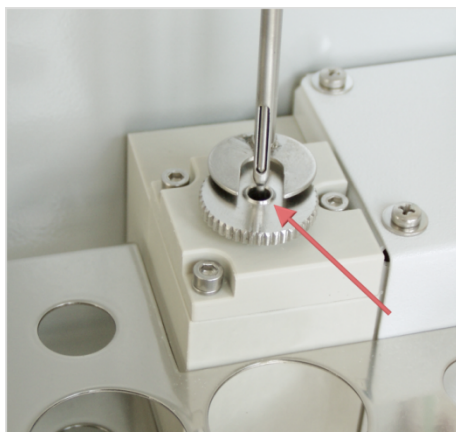


Fig. 65 Adjusting the canula on the POC reactor

- ▶ Adjust the POC reactor position: Adjust the canula as precisely as possible in the x- and y-axis directions.
- ▶ Adjust the special canula in the z-axis direction so that the entire thick needle shaft including the ventilation slot can be seen above the POC reactor port.
- ▶ After each change, click on **[Rinse position adjust]**, **[Position 1 adjust]** or **[Position POC reactor adjust]**.
 - ✓ The autosampler will move to the new position.
- ▶ Repeat these steps until the canula position is optimized.
- ▶ Click on **[Save]** to save the adjustment values.
- ▶ Open the **Sampler Alignment** window again. Move to the adjusted positions via the buttons to check.
 - ✓ The autosampler is adjusted.

6.2.5 Setting the NPOC purge flow



CAUTION

Risk of burns from the furnace

To set the NPOC purge flow, you must open the side wall of the analyzer. This presents a risk of burn injuries from the hot furnace.

- When setting the NPOC purge flow on the gas box, maintain a safe distance to the hot combustion furnace.

The NPOC purge flow is preset to approx. 100 ml/min. Depending on the measurement task, you can increase or decrease the NPOC purge flow via the NPOC needle valve. The NPOC needle valve is located behind the left side wall, to the left of the combustion furnace.

Set the NPOC purge flow as follows:

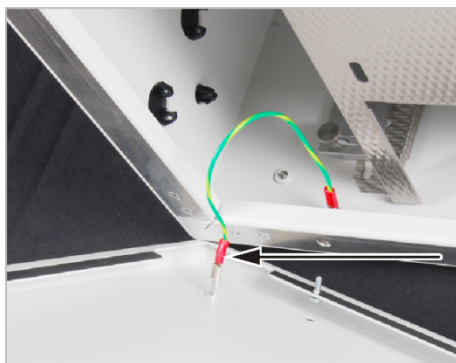


Fig. 66 Grounding conductor connection on the side wall

- ▶ Open the left side wall of the analyzer. Push the accessory modules to the side if necessary. Do not kink any connection hoses.
 - Unscrew the four attachment screws. The screws are captive and remain attached to the wall.
 - Remove the protective grounding. Set the side wall aside safely.
- ▶ Open the window of the same name with the **Instrument | Device control** menu option.

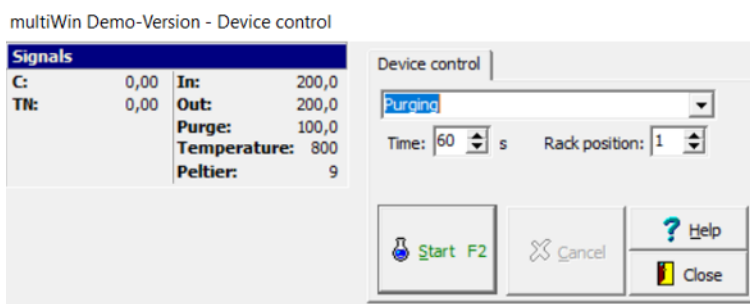


Fig. 67 Setting the purge flow

- ▶ Select the **Purgine** option in the list field.
- ▶ Select the purge time in the **Time** field in the 1 to 900 s range.
- ▶ For sample feed with autosampler: In the **Rack position** field, select any position on the sample tray to monitor the purge flow at.
- ▶ Place a sample vessel with ultrapure water at this position.
- ▶ For manual sample supply: Insert purge hose 7 in a sample vessel filled with ultrapure water.
- ▶ Click the **[Start F2]** button.
- ▶ Unscrew the adjustment screw on the NPOC needle valve.
- ▶ Set the desired NPOC purge flow:
 - Increase the NPOC purge flow: Turn the needle valve to the left.
 - Decrease the NPOC purge flow: Turn the needle valve to the right.
- ▶ Check the flow indication in the **System state** window when doing this. The current NPOC purge flow is displayed under **Purge**.
- ▶ Screw the adjustment screw on the needle valve back in.
- ▶ Close the side wall.
 - Connect the protective grounding to the left side wall.
 - Slightly tighten the screws first on the bottom side and then on the top side. Tighten the screws in turns.

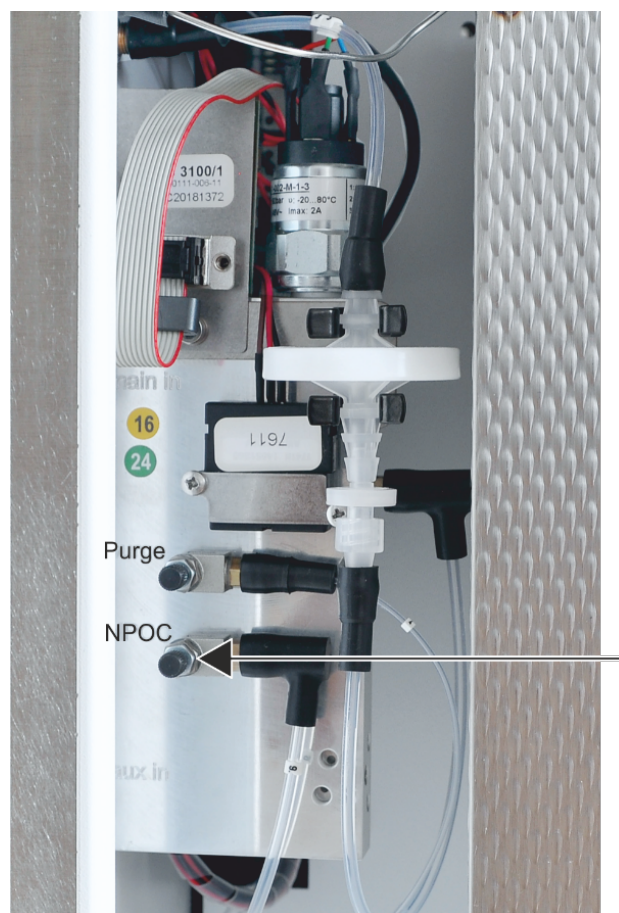
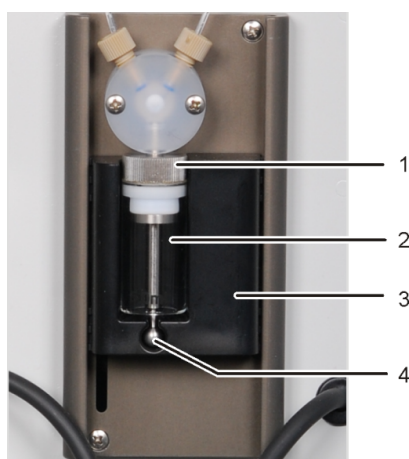


Fig. 68 Setting the NPOC purge flow

6.3 Syringe pump maintenance

Clean or replace the dosing syringe of the syringe pump as follows:



- ▶ Open the doors of the analyzer.
- ▶ Empty the syringe pump via the software:
 - Open the window of the same name with the **Instrument | Device control** menu option.
 - Select the **Change syringe** option and click **[Start F2]**.
 - ✓ The syringe is emptied and moved to the replacement position.
- ▶ Unscrew the dosing syringe on the valve (1) and remove it from the drive (3).
- ▶ Dismantle and clean the glass cylinder (2) and piston (4).
- ▶ Insert the piston rod of the new dosing syringe into the drive.
- ▶ Screw the glass cylinder onto the valve.
 - ✓ The analyzer is ready for operation again.

6.4 Replacing the pump hose



CAUTION

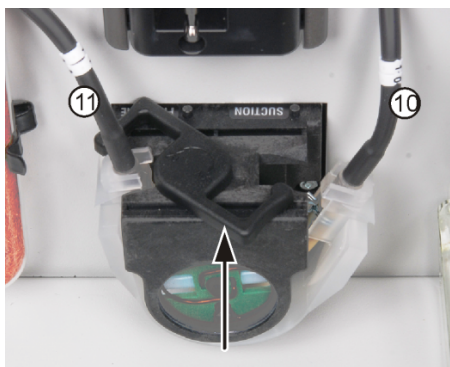
Risk of chemical burns during hose replacement

Small quantities of acidic solutions can still be in the hoses.

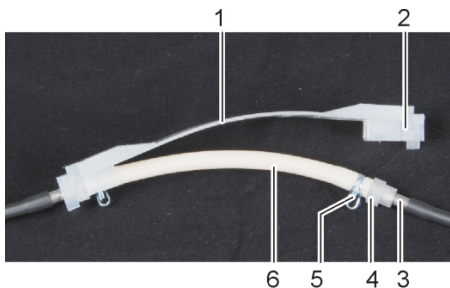
- Wear protective gloves and clothing when replacing the hoses.
- Collect any leaking liquids with an absorbent sheet.

Check the pump hoses every 3 months for leaks and replace them after 12 months at the latest.

Condensate pump



- ▶ Exit the control and evaluation software or switch off the gas flow by clicking **[OK]** next to **Gas flow** in the **System state** window.
- ▶ Open the doors of the analyzer.
- ▶ Press the bracket on the condensate pump to the left.
- ▶ Pull hoses 10 and 11 off of their connections.



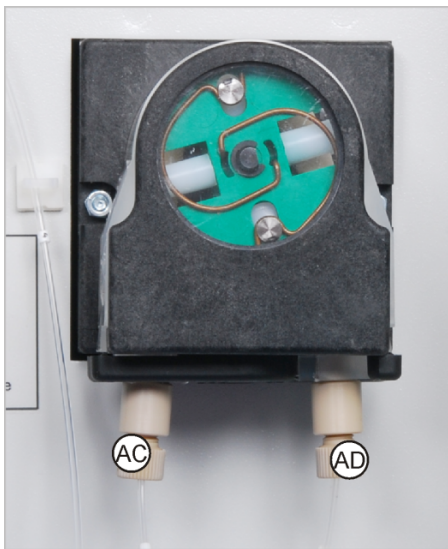
- 1 Guide piece
- 2 Groove
- 3 Metal connection
- 4 Hose guide
- 5 Hose clamp
- 6 Pump hose

- ▶ Remove the guide piece with the pump hose from the pump body.
- ▶ Check the pump hose and the connections for excessive wear and cracks. If moisture escapes the pump hose or the connections, replace the pump hose.
- ▶ Wipe the pump body and the roller carrier with ultrapure water.
- ▶ Check the pump body and roller carrier for wear.
- ▶ Press the still-intact or new pump hose back into the guide piece. Align the hose clamps downward during installation.
- ▶ Insert the hose guide in the groove of the guide piece.

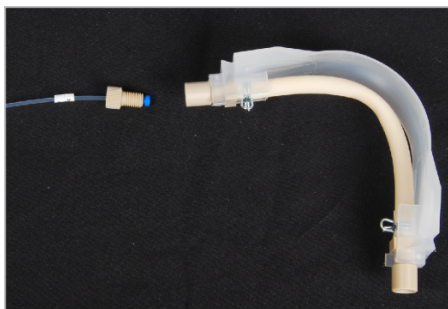


- ▶ Position the guide piece around the pump body.
- ▶ Press the guide piece upward with one hand. Turn the clip to the right until it engages with the other hand.
- ▶ Push hoses 10 and 11 back onto their adapters.
- ▶ Switch on the gas supply again and check the system for leaks.
 - ✓ The pump is once again ready for operation.

Phosphoric acid pump



- ▶ Exit the control and evaluation software or switch off the gas flow by clicking [OK] next to **Gas flow** in the **System state** window.
- ▶ Remove the pump hose as with the condensate pump.



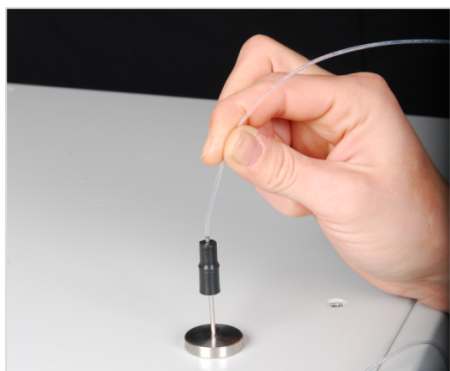
- ▶ Hoses AC and AD are connected to the pump with Fingertight connections. Unscrew the hoses with Fingertight connections from the pump.
- ▶ Check the hoses for heavy wear and cracks.
- ▶ Install the pump hoses as described above. Screw hoses AC and AD back onto the pump.
- ▶ Switch on the gas supply again and check the system for leaks.
 - ✓ The pump is once again ready for operation.

6.5 Replacing the hose connections

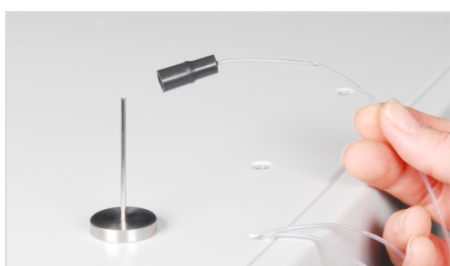
FAST connectors connect hoses with glass components. Use the threading aid to feed thin hoses into the connectors. This is included with the analyzer. Check the system for leaks after hose replacement.



- ▶ Slide the FAST connector onto the canula of the threading aid. The narrow hole of the connector points upwards.



- ▶ Thread the hose into the canula of the threading aid.



- ▶ Slide the FAST connector from the canula onto the hose.
- ▶ Pull the hose out of the canula of the threading aid. Pull the hose of the FAST connector until it no longer reaches into the wider hole.

Angled FAST connectors

With angled FAST connectors, do not slide the hose ends beyond the side length of the connector. The gas flow will otherwise be impaired.

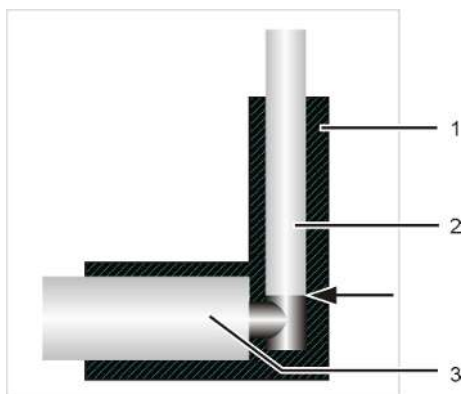


Fig. 69 FAST connector, angled

- 1 Angled FAST connector
- 3 Glass connection

2 Hose

Fingertight connections

- ▶ When replacing Fingertight connections, only use straight cut, round, uncrimped hose ends.
- ▶ Slide the conical nipple onto the hose with the conical side towards the banjo bolt. The conical nipple and hose end must be flush.
- ▶ Do not jam the banjo bolt during insertion and only tighten it hand-tight.

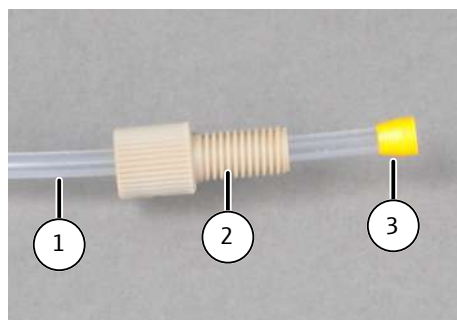


Fig. 70 Replacing the Fingertight connection

- | | |
|------------------|--------------|
| 1 Hose | 2 Banjo bolt |
| 3 Conical nipple | |

6.6 Checking the system for leaks



NOTICE

Risk of gas leakage

When the outlet flow is significantly less than the inlet flow, the device system has a gas leak.

- Check all connection pieces, for example with a foamy tenside solution.
- Only put the device into operation when the gas leak has been eliminated.

The system tightness is automatically checked at the gas outlet of the analyzer.

- ▶ Switch on the analyzer.
- ▶ Open the carrier gas supply on the pressure reducer.
- ▶ Start the control and analysis software.
- ▶ Check the flow display in the **System state** window:
 - **In** (inlet flow): 160 ml/min
 - **Out** (outlet flow): 160 ± 10 ml/min

6.7 Replacing the catalyst

If the catalyst loses effectiveness, the combustion tube must be refilled with fresh catalyst.

The software indicates when the maintenance interval of the catalyst has elapsed after a maximum of 1500 injections. You must then check if the catalyst requires replacement.

Dispose of the catalyst in accordance with disposal regulations.

See also

- 📖 Disposal [▶ 132]

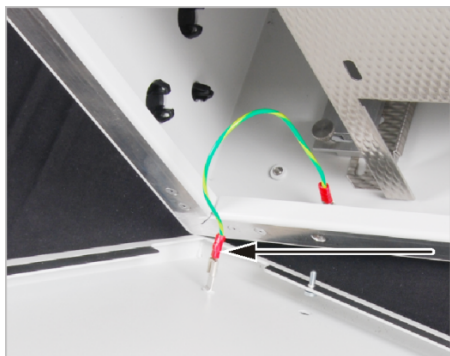
6.7.1 Removing the combustion tube



CAUTION

Risk of burns at the hot furnace

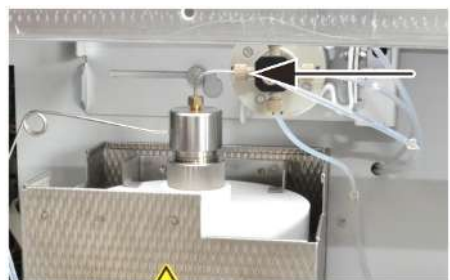
- Switch off the device and allow it to cool before performing maintenance.



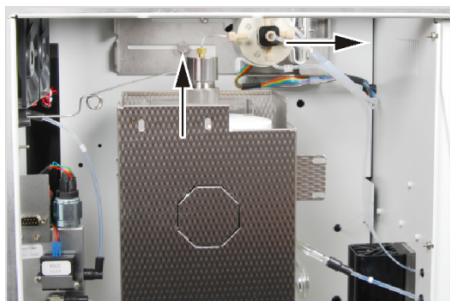
- ▶ Switch off the analyzer via the main switch. Disconnect the power plug from the socket. Shut off the gas supply on the pressure reducer in the laboratory.
- ▶ Open the left side wall of the analyzer. Push the accessory modules to the side if necessary. Do not kink any connection hoses.
 - Unscrew the four attachment screws. The screws are captive and remain attached to the wall.
 - Remove the protective grounding. Set the side wall aside safely.



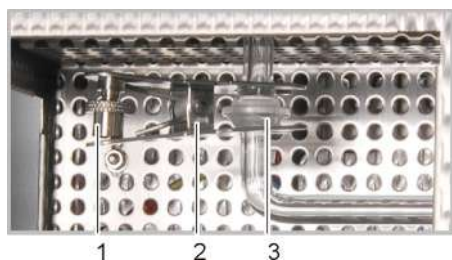
- ▶ Pull the carrier gas canula out of the FAST connector on the left side wall.



- ▶ Unscrew the Fingertight connection of the furnace canula from the change-over valve.



- ▶ Loosen the knurled head screw on the holder of the change-over valve.
- ▶ Slide the change-over valve to the right. This pulls the furnace canula out of the change-over valve.



- ▶ Release the joint connection (3) on the bottom of the combustion furnace connecting the combustion tube with the condensation coil.
- ▶ To do this, unscrew the knurled head screw (1) and remove the fork clamp (2).

- ▶ Carefully pull the combustion tube out of the combustion furnace toward the top.
- ▶ Unscrew the furnace head from the combustion tube. Remove the union nut, the pressure ring and the three sealing rings.
- ▶ Remove the used catalyst filling. Check the combustion tube for heavy crystallization, cracks and burst spots. Only re-use intact combustion tubes.
- ▶ Thoroughly rinse the empty combustion tube with ultrapure water and dry it well.

6.7.2 Filling the combustion tube



NOTICE

Sweat from your hands can reduce the service life of the combustion tube.

Alkaline salts from the sweat of your hands can cause crystallization in the quartz glass when heating the combustion furnace. This reduces the service life of the combustion tube.

- Avoid touching the cleaned combustion tube with your hands during filling. Wear protective gloves.
- Only fill completely dried combustion tubes.
- Wipe off any finger marks with a cloth wetted with pure alcohol.



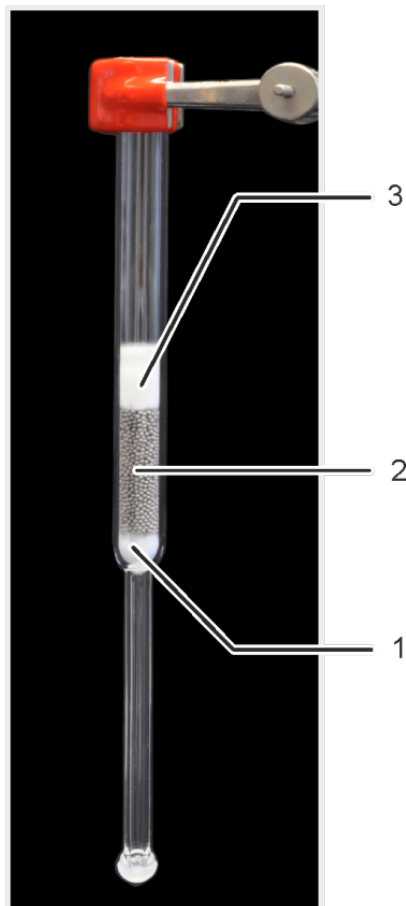
NOTICE

Risk of detector damage

The catalyst can emit gas during initial heating, this can be seen as mist formation in the TIC condensate container.

- Allow the catalyst to burn out during initial heating for approximately 30 min at operating temperature.
- During this, interrupt the gas flow at the water traps on the front side to protect the detector from the gases.

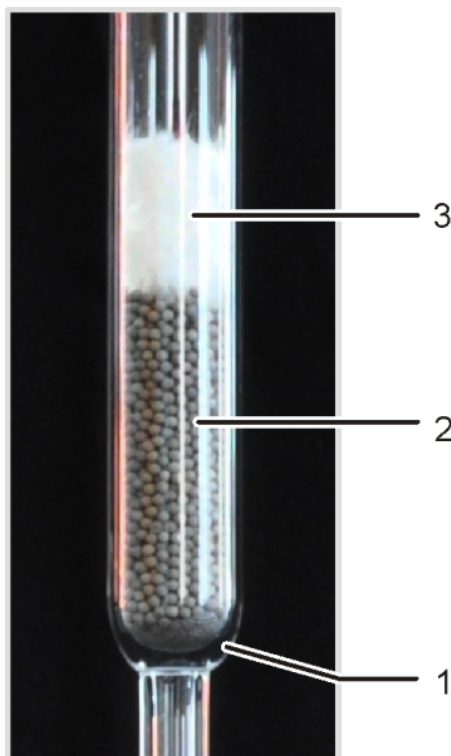
Filling the combustion tube, for normal samples



- ▶ For filling, fix the combustion tube in a stand.
- ▶ Fill quartz glass wool (1) into the combustion tube approx. 1 cm high, carefully press it down with a glass rod and press it into place. The glass wool holds back the catalyst. Ensure that no catalyst can get into the gas pathway. Also, do not pack the glass wool too tightly!
- ▶ Carefully stack platinum catalyst (2) onto the quartz glass wool approx. 4 cm high.
- ▶ Roll up the HT mat (3) from the narrow side. The roll must have a diameter of approx. 13 mm and a height of 2 cm to slide into the combustion tube easily.
- ▶ Insert the rolled-up HT mat into the combustion tube and push it down with a glass rod until the catalyst is covered.
- ▶ Only press the mat down lightly onto the catalyst.

The recommended operating temperature for this filling is 800 °C.

Filling the combustion tube, for samples with high salt loads



With samples with high salt loads, the catalyst is filled onto a platinum net.

- ▶ For filling, fix the combustion tube in a stand.
- ▶ Insert the platinum net into the combustion tube and carefully press it down with a glass rod. The platinum net holds back the catalyst. Ensure that no catalyst can get into the gas pathway.
- ▶ Carefully stack platinum catalyst (2) onto the platinum net approx. 4 cm high.
- ▶ Roll up the HT mat (3) from the narrow side. The roll must have a diameter of approx. 13 mm and a height of 2 cm to slide into the combustion tube easily.
- ▶ Insert the rolled-up HT mat into the combustion tube and push it down with a glass rod until the catalyst is covered.
- ▶ Only press the mat down lightly onto the catalyst.

The recommended operating temperature for this filling is 720 to 750 °C.

6.7.3 Installing the combustion tube



NOTICE

Sweat from your hands can reduce the service life of the combustion tube.

Alkaline salts from the sweat of your hands can cause crystallization in the quartz glass when heating the combustion furnace. This reduces the service life of the combustion tube.

- Avoid touching the cleaned combustion tube with your hands during filling. Wear protective gloves.
- Only fill completely dried combustion tubes.
- Wipe off any finger marks with a cloth wetted with pure alcohol.



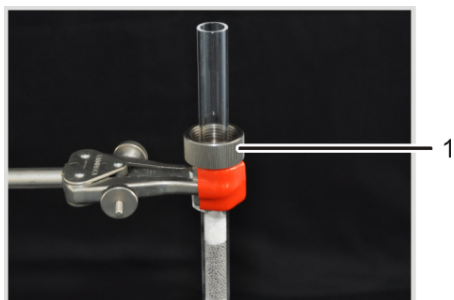
NOTICE

Preventing tightness problems

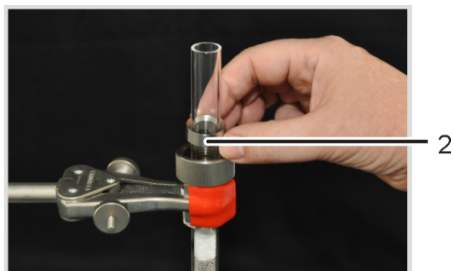
Due to slight variation in the outer diameter of the combustion tubes, a new combustion tube may not be able to be installed tightly with previously used O-rings.

- When installing a new combustion tube, always use new O-rings (402-815.102).

Install the furnace head on the combustion tube before inserting the combustion tube into the furnace.



- ▶ Slide the union nut (1) onto the combustion tube.



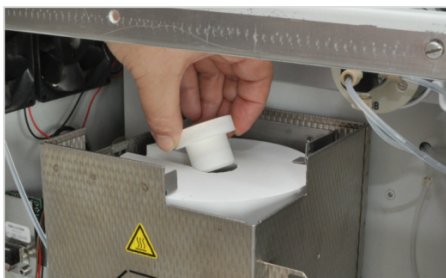
- ▶ Place the pressure ring (2) in the union nut.
The conical side of the pressure ring must point upward.



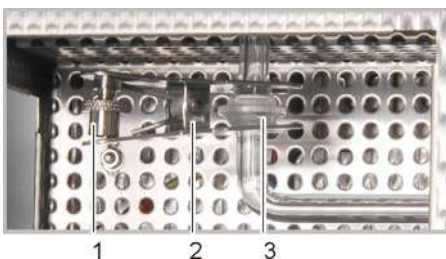
- ▶ Slide the three coated sealing rings (3) onto the combustion tube.
Ensure that the sealing rings are flush at the edge of the combustion tube.



- ▶ Carefully put the furnace head onto the combustion tube up to the stop.
- ▶ Press the furnace head lightly against the tube and tighten the union nut hand-tight. The furnace canula and the carrier gas canula have already been attached to the furnace head.



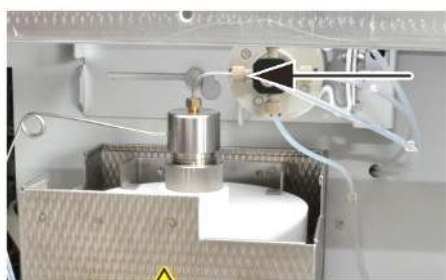
- ▶ If it is not present, insert the ceramic holder into the top opening of the combustion furnace.
- ▶ Insert the combustion tube with the furnace head into the combustion furnace.



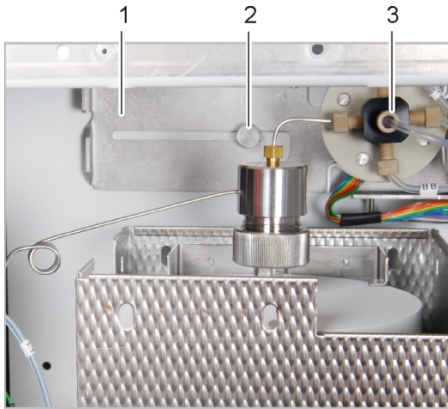
- ▶ Connect the lower end of the combustion tube and the inlet of the condensation coil via the spherical joint connection (1).
- ▶ Secure the spherical joint connection with the forked clamp (2). Tighten the knurled head screw (3) hand-tight.



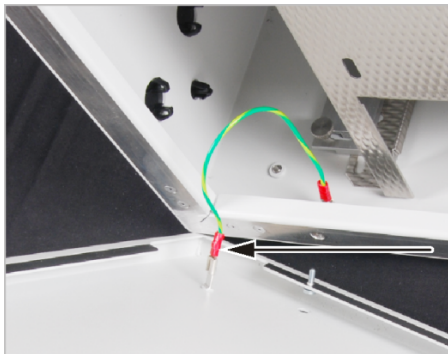
- ▶ Connect the carrier gas connection with the connection in the device wall with the FAST connector.



- ▶ Slide the change-over valve to the left until the change-over valve contacts the connection of the furnace canula.
- ▶ Screw the furnace canula with the Fingertight connection finger-tight to the change-over valve.



- ▶ Fix the change-over valve (3) in this position. Tighten the knurled head screw (2) on the holder (1) finger-tight to do so.
- ▶ Place the top cover on top of the analyzer.



- ▶ Close the side wall.
 - Connect the protective grounding to the left side wall.
 - Slightly tighten the screws first on the bottom side and then on the top side. Tighten the screws in turns.
- ▶ Open the gas supply. Connect the power plug with the socket and switch on the analyzer via the main switch.
- ▶ Check the system for leaks.
 - ✓ The analyzer is ready for operation again.

6.8 Removing and installing the combustion furnace

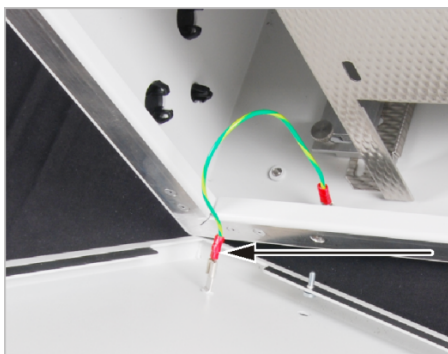
6.8.1 Removing the combustion furnace



CAUTION

Risk of burns at the hot furnace

- Switch off the device and allow it to cool before performing maintenance.



- ▶ Switch off the analyzer via the main switch. Disconnect the power plug from the socket. Shut off the gas supply on the pressure reducer in the laboratory.
- ▶ Open the left side wall of the analyzer. Push the accessory modules to the side if necessary. Do not kink any connection hoses.
 - Unscrew the four attachment screws. The screws are captive and remain attached to the wall.
 - Remove the protective grounding. Set the side wall aside safely.



- ▶ Remove the top cover.
- ▶ Remove the combustion tube. Slide the change-over valve to the right to prevent it from interfering with removal.
- ▶ Remove the condensation coil.
- ▶ Pull the plug-in connector for the combustion furnace out of its socket.



- ▶ Unscrew the knurled head screws the furnace is fastened to the device bottom with.
- ▶ Lift the furnace out of the analyzer.
- ▶ Reattach the knurled head screws to the device bottom so they are not lost.

See also

- 📄 Removing the combustion tube [▶ 101]
- 📄 Condensation coil maintenance [▶ 109]

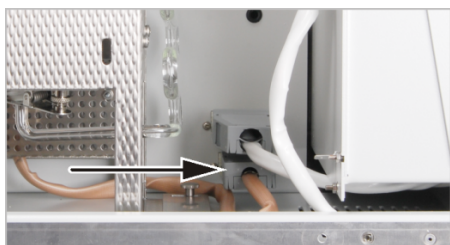
6.8.2 Installing the combustion furnace



- ▶ Open the left side wall of the analyzer. Remove the top cover.
- ▶ Loosen the knurled head screw on the holder of the change-over valve. Slide the change-over valve to the right to prevent it from interfering with installation.
- ▶ Unscrew the knurled head screws on the device bottom the furnace is fastened with.



- ▶ Insert the furnace in the middle. Align the front side of the furnace to be parallel with the device wall.
- ▶ Fasten the furnace with the knurled head screws. Tighten the knurled head screws finger-tight.



- ▶ Plug the plug-in connector for the combustion furnace into the socket at the bottom right of the rear device wall.
- ▶ Install the combustion furnace.
- ▶ Install the condensation coil.
- ▶ Push the sample intake hose and the purging hose through the top opening. Attach the top cover. Connect the hoses to the autosampler.



- ▶ Close the side wall.
 - Connect the protective grounding to the left side wall.
 - Slightly tighten the screws first on the bottom side and then on the top side. Tighten the screws in turns.
- ▶ Open the gas supply. Connect the power plug with the socket and switch on the analyzer via the main switch.
- ▶ Check the system for leaks.
 - ✓ The analyzer is ready for operation again.

6.9 Cleaning the TIC condensate vessel



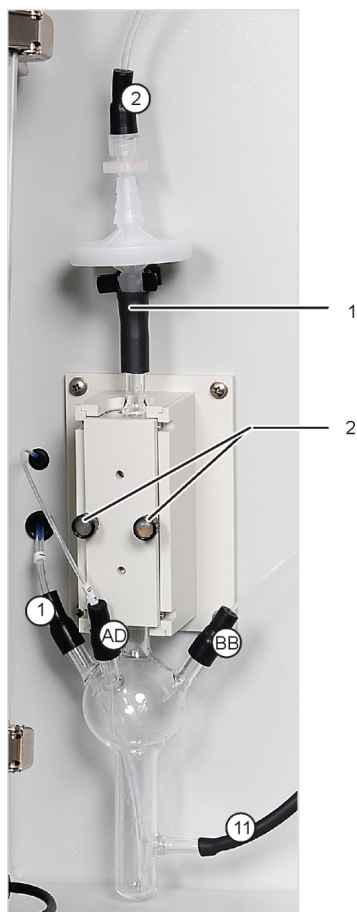
WARNING

Risk of chemical burns from phosphoric acid

The TIC condensate container contains phosphoric acid. Phosphoric acid can irritate eyes, skin and mucous membranes.

- Wear safety goggles and protective clothing when handling concentrated acids. Work under an extractor.
- Observe all instructions and specifications in the safety data sheet.

Check the TIC condensate container regularly for deposits. Only clean the TIC condensate container when the samples are no longer purged correctly.



- ▶ Exit the control and evaluation software or switch off the gas flow by clicking **[OK]** next to **Gas flow** in the **System state** window.
- ▶ Open the doors of the analyzer.
- ▶ Pull the connection hose to the water traps (1) off of the TIC condensate container.
- ▶ Pull hoses 1, AD and BB with FAST connectors off of the TIC condensate container.
- ▶ Disconnect waste hose 11 from the bottom connection on the TIC condensate container.
- ▶ Unscrew the 2 knurled head screws on the cover of the cooling block. Remove the cover and remove the TIC condensate container.
- ▶ Check the TIC condensate container for deposits and cracks and rinse it with ultrapure water.
- ▶ Fasten the hoses in accordance with the image:
 - Slide waste hose 11 at least 1 cm onto the bottom connection of the TIC condensate container.
 - Slide hoses 1, AD and BB with FAST connectors onto the connections of the TIC condensate container. Slide the FAST connector on at least 1 cm.
 - Slide hose 1 almost to the bottom of the TIC condensate container.
 - Fasten the connection hose (1) between the TIC condensate container and the water traps.
- ▶ Insert the TIC condensate container in the cooling block. Fasten the cover of the cooling block with the two knurled head screws.
- ▶ Reactivate the gas supply.
 - ✓ The TIC condensate container is again ready for operation.

6.10 Condensation coil maintenance

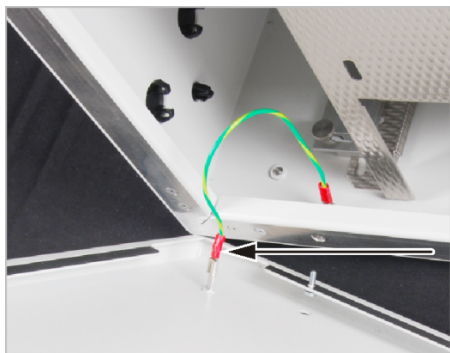
Removal and cleaning



CAUTION

Risk of burns at the hot furnace

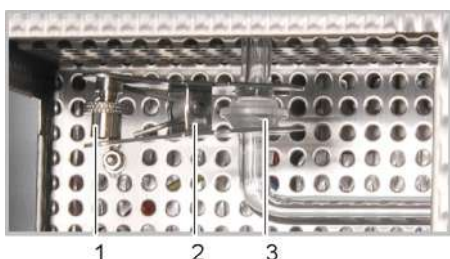
- Switch off the device and allow it to cool before performing maintenance.



- ▶ Switch off the analyzer via the main switch. Disconnect the power plug from the socket. Shut off the gas supply on the pressure reducer in the laboratory.
- ▶ Open the left side wall of the analyzer. Push the accessory modules to the side if necessary. Do not kink any connection hoses.
 - Unscrew the four attachment screws. The screws are captive and remain attached to the wall.
 - Remove the protective grounding. Set the side wall aside safely.



- ▶ Pull hose 1 out of the FAST connector of the condensation coil.



- ▶ Release the joint connection (3) on the bottom of the combustion furnace connecting the combustion tube with the condensation coil.
- ▶ To do this, unscrew the knurled head screw (1) and remove the fork clamp (2).

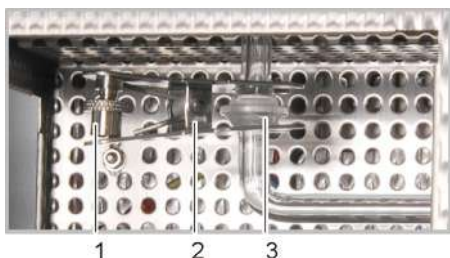
- ▶ Carefully remove the condensation coil from the analyzer. Pull the bottom part out of the opening of the combustion furnace when doing this.
- ▶ Remove the FAST connector from the glass connection of the condensation coil.
- ▶ Inspect the condensation coil for deposits and cracks.

- ▶ Rinse the condensation coil with ultrapure water and dry it well.

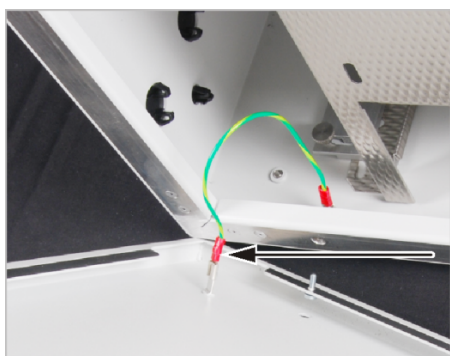
Installation



- ▶ Slide hose 1 into the FAST connector.
- ▶ Slide the FAST connector onto the glass connection of the condensation coil.
- ▶ Hold the condensation coil against the right side of the combustion furnace. The spherical joint of the coil points toward the lower opening of the furnace.



- ▶ Connect the lower end of the combustion tube and the inlet of the condensation coil via the spherical joint connection (1).
- ▶ Secure the spherical joint connection with the forked clamp (2). Tighten the knurled head screw (3) hand-tight.



- ▶ Close the side wall.
 - Connect the protective grounding to the left side wall.
 - Slightly tighten the screws first on the bottom side and then on the top side. Tighten the screws in turns.
- ▶ Open the gas supply. Connect the power plug with the socket and switch on the analyzer via the main switch.
- ▶ Check the system for leaks.
 - ✓ The analyzer is ready for operation again.

6.11 Replacing the water traps

Replace the water traps dependent on the sample matrix, but no later than after 6 months.

The water traps consist of a prefilter and a disposable retention filter. Always replace both water traps. Observe that the water traps only function properly if they are installed in the correct order and direction.

Check the system for leaks after replacing the water traps.

Water traps on the front side

You can replace the water traps on the front side while the device is switched on, but not during a measurement.

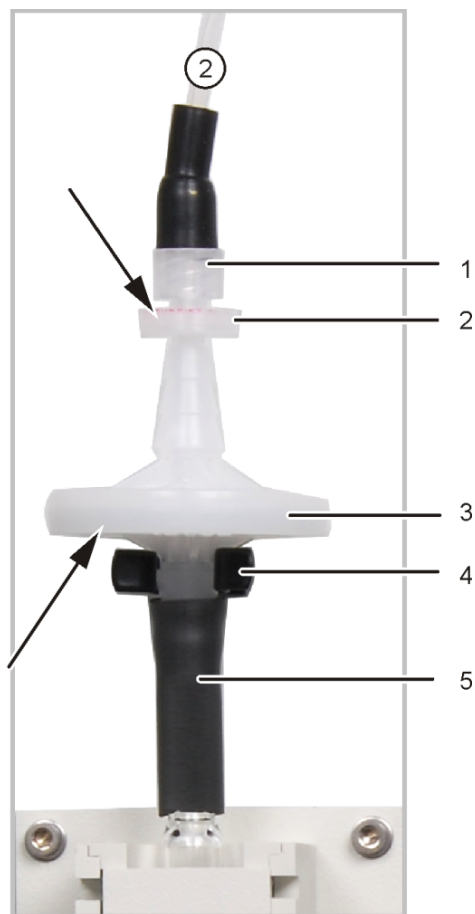


Fig. 71 Replacing the water traps on the front side

- | | |
|--|-------------------------------|
| 1 Luer adapter to hose 2 | 2 Disposable retention filter |
| 3 Clamp | 4 Aerosol trap as prefilter |
| 5 Hose connection to the TIC container | |

- ▶ Open the doors of the analyzer.
- ▶ Unscrew the upper hose connection with a rotating motion. Pull off the lower hose connection.
- ▶ Assemble the new water traps:
 - The "INLET" marking on the large water trap (aerosol trap) must face downward.
 - The labeling on the small water trap (disposable retention filter) must face upward.
- ▶ Connect the large water trap with the lower hose.
- ▶ Press the water traps into the clamp(s) on the device wall.
- ▶ Screw in the Luer connection on the top small water trap.
- ▶ Check the system for leaks.
- ▶ Close the front doors again.

Water traps on the gas box

Two water traps are installed in front of the gas box (prefilter and disposable retention filter). They protect the gas box from aerosols and rising water in case of incorrect gas pressures. The left side wall of the analyzer must be opened to replace the water traps.



CAUTION

Risk of burns at the hot furnace

- Switch off the device and allow it to cool before performing maintenance.

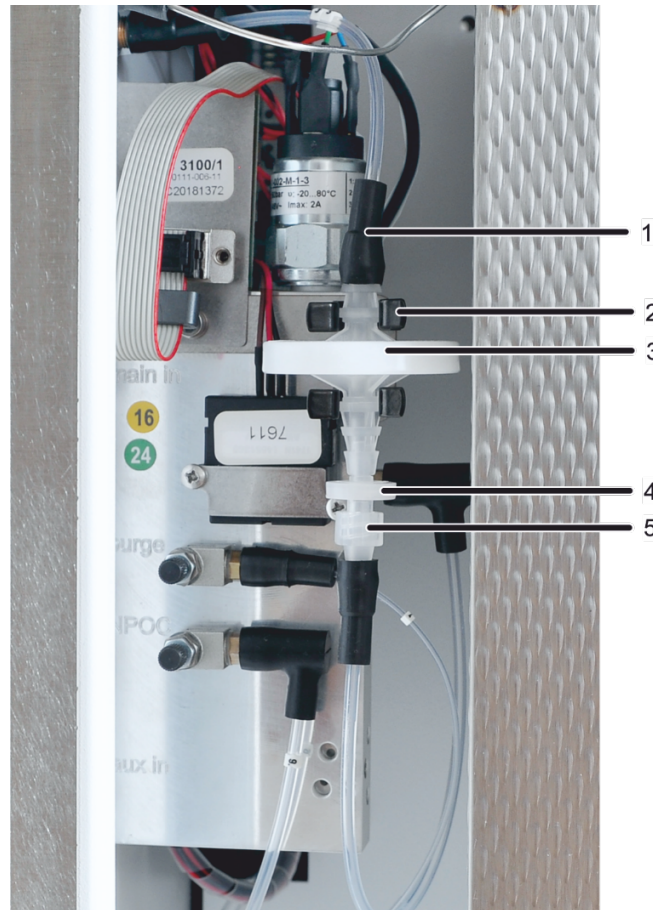


Fig. 72 Replacing the water traps on the gas box

- | | |
|----------------------------|-------------------------------|
| 1 FAST connector | 2 Clamp on the gas box |
| 3 Prefilter (aerosol trap) | 4 Disposable retention filter |
| 5 Luer connection | |
- ▶ Exit the control and analysis software.
 - ▶ Switch off the analyzer using the power switch. Disconnect the power plug from the socket. Allow the analyzer to cool down.
 - ▶ Open the left side wall of the analyzer. Push the accessory modules to the side if necessary. Do not kink any connection hoses.
 - Unscrew the four attachment screws. The screws are captive and remain attached to the wall.
 - Remove the protective grounding. Set the side wall aside safely.
 - ▶ Pull the water traps out of the two clamps on the gas box.
 - ▶ Pull the upper FAST connector off of the water traps.
 - ▶ Remove the water traps from the Luer connection.
 - ▶ Assemble the new water traps:
 - The "INLET" marking on the large water trap (aerosol trap) must face upward.

- The labeling on the small water trap (disposable retention filter) must face downward.
- ▶ Connect the large water trap with the upper FAST connector.
- ▶ Connect the small water trap to the Luer connection on the bottom.
- ▶ Press the water traps into the clamps on the gas box.
- ▶ Close the side wall.
 - Connect the protective grounding to the left side wall.
 - Slightly tighten the screws first on the bottom side and then on the top side. Tighten the screws in turns.
- ▶ Connect the power plug with the socket and switch on the analyzer again via the main switch.
- ▶ Check the system for leaks.
 - ✓ The water traps on the front side and the gas box are replaced.

See also

📖 Checking the system for leaks [▶ 100]

6.12 Replacing the halogen trap



NOTICE

Risk of device damage due to depleted copper wool

Damage to optical and electronic components of the analyzer due to aggressive combustion products when the copper wool in the halogen trap is depleted!

- Only use the device with an operational halogen trap!
- Replace the complete filling of the halogen trap when half of the copper wool or brass wool is discolored!

The analyzer can remain switched on to replace the used copper and brass wool.

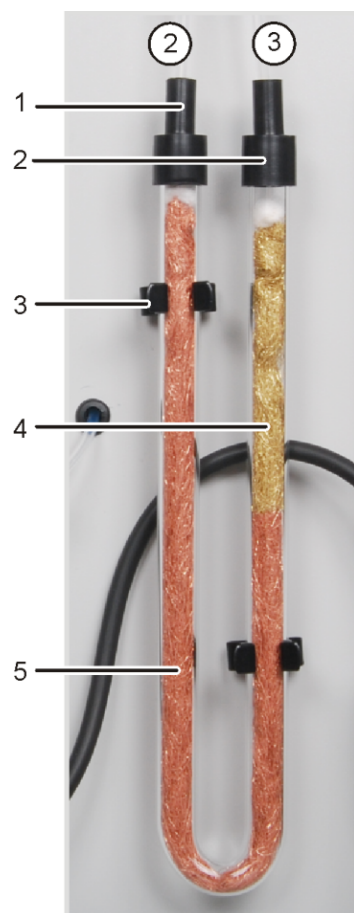


Fig. 73 Replacing the halogen trap

- | | |
|----------------------------|----------------------------|
| 1 FAST connector to hose 2 | 2 FAST connector to hose 3 |
| 3 Clamp | 4 Brass wool |
| 5 Copper wool | |

- ▶ Open the doors of the analyzer.
- ▶ Remove the FAST connectors from the halogen trap and remove the U-tube from the clamps.
- ▶ Pull out the depleted copper wool or brass wool from the U-tube with tweezers or a small hook.
- ▶ Check the U-tube for cracks. Only reuse a fully intact U-tube.
- ▶ If necessary, rinse the U-tube with ultrapure water and allow it to dry well.
- ▶ Fill the U-tube with new copper wool and brass wool using tweezers or a small hook.
 - Replace the complete contents of the U-tube. Do not pack the copper and brass wool too tightly, but do not allow any larger empty spaces.
- ▶ Cover the copper wool and the brass wool with cotton wool.
- ▶ Carefully press the filled U-tube into the clamps again.
- ▶ Reconnect the gas hoses with FAST connectors to the halogen trap:
 - Hose 2 to the branch with copper wool (connection to the water trap)
 - Hose 3 to the branch with brass wool (connection to the detector)
- ▶ Check the system for leaks.
- ▶ Close the doors of the analyzer again.

6.13 POC module maintenance

Checking the adsorber function

The LiOH adsorber can clump over time.

Check the function of the adsorber monthly. For samples with high organic carbon content, check more frequently.

- ▶ Create a TIC mix standard solution from carbonate/hydrogen carbonate (100 mg/l)
- ▶ Load a POC method in the software.
- ▶ Measure the standard solution.
- ▶ If the result is greater than 0.1 mg/l, the adsorber is exhausted. Replace the adsorber if this is the case.

Checking the module for leaks



NOTICE

Risk of gas leakage

When the outlet flow is significantly less than the inlet flow, the device system has a gas leak.

- Check all connection pieces, for example with a foamy tenside solution.
- Only put the device into operation when the gas leak has been eliminated.

The system tightness is automatically checked at the gas outlet of the analyzer.

- ▶ Switch on the analyzer.
- ▶ Open the carrier gas supply on the pressure reducer.
- ▶ Start the control and analysis software.
- ▶ Check the flow display in the **System state** window:
 - **In** (inlet flow): 160 ml/min
 - **Out** (outlet flow): 160 ± 10 ml/min
- ▶ Open the window of the same name with the **Instrument | Component Test** menu option. Switch to the **Valves** tab.
- ▶ Switch on the 3 valve.

The flow indication in the **System state** window must not decrease.
- ▶ If the flow decreases or the outlet flow is significantly below the inlet flow, check all hose connections and septums. Eliminate the leak.
- ▶ Close the **Component Test** window again.
 - ✓ The device system has no leaks.

6.14 Maintaining the chemiluminescence detector (CLD)

Replace the adsorber cartridge on the rear of the detector every 12 months. The cartridge cleans the gas which exits the detector at the "out" outlet.

The cartridge is filled with active carbon and soda lime. Do not open the cartridge. Dispose of the used cartridge as a whole in accordance with the local regulations.



Fig. 74 Replacing the adsorber cartridge

- ▶ Disconnect the hose from the cartridge.
- ▶ Pull the cartridge out of the clamp.
- ▶ Unscrew the hose connection at the top of the cartridge.
- ▶ Dispose of the used cartridge as a whole in a professional manner.
- ▶ Screw the hose connection into the top of the new cartridge.
- ▶ Push the new cartridge into the clamp. Connect the cartridge to the hose from the "out" outlet.
 - ✓ The detector can now be used for measurements again.

7 Troubleshooting



NOTICE

Risk of device damage

Contact customer service in the following cases:

- The troubleshooting measures described do not eliminate the error.
- The error occurs repeatedly.
- The error message is not featured in the following list or the list refers to customer service for troubleshooting the error.

The system is monitored as soon as the device is switched on. After starting the control software, all malfunctions of the device are reported using error messages. Error messages consist of an error code and an error message.

The following section describes a number of possible malfunctions which the operator can partly troubleshoot without the help of a customer service technician. Confirm the error message and carry out the troubleshooting measures.

Before starting a measurement, a flow control is always carried out. A flow error is detected as soon as the actual flow differs ± 10 ml/min from the target flow.

For fault analysis, log files can be recorded. After consulting customer service, activate log file recording. The software stores the log file in the ...\\multiWin\\LOG directory.

The following files can be generated and saved:

- multiWin_LOG.*
Log file for error messages, this is always created automatically.
- multiWin_ADU.*
Log file for monitoring the NDIR detector, this is created automatically after activation.

Activating the NDIR detector monitoring:

- ▶ Open the **Component Test** window, **Optical bench** tab with the **Instrument | Component Test** menu option.
- ▶ Activate the **Optical bench** with a check mark.

Notifying customer service of the error:

- ▶ Copy the ...\\multiWin\\LOG directory. To do this, use the **Copy ..\\multiWin\\LOG*.*** menu option in the **Instrument | System parameters** menu, **Error analysis** tab.
- ▶ Send the directory to customer service via email. For the customer service address, see the inside front cover.

7.1 Software error messages

Error code: Error message	VERS: Communication error – incorrect command set between PC and device!
Cause	Remedy
■ The internal and external program versions do not match.	■ Update the internal and external programs.

Error code: Error message	VERS1: Communication error – analyzer
Cause	Remedy
■ The analyzer is not switched on.	■ Switch on the analyzer.
■ The multiWin software was started too early.	■ Only start the software after 30 s.
■ The analyzer is not connected to the PC.	■ Check the connection of the analyzer to the PC.
■ The incorrect COM port is being used on the external computer.	■ Check the port on the external computer. ■ If necessary, select another interface in the software (with the Configuration Interface menu option).
Error code: Error message	-6: Analyzer is busy
Cause	Remedy
■ The analyzer is in busy state for >10 min.	■ Initialize the analyzer.
Error code: Error message	-5: Communication error – analyzer STAT, MESS, STEP or INIT
Cause	Remedy
■ Communication error.	■ Initialize the analyzer.
Error code: Error message	-4: Communication error – analyzer
Cause	Remedy
■ Communication error.	■ Check the interface cable. ■ Initialize the analyzer.
Error code: Error message	-3: Command from the analyzer CRC error
	-2: CRC error
	-1: Invalid command from the analyzer
Cause	Remedy
■ Communication error.	■ Initialize the analyzer.
Error code: Error message	1: Incomplete command from the PC
	2: PC command without STX
	3: PC command without *
	4: PC command CRC error
	5: PC command invalid command
	6: PC command invalid MESS command
Cause	Remedy
■ Faulty connection between the internal and external program.	■ Initialize the analyzer.
Error code: Error message	7: COM 2 not found
	8: COM 3 not found
	9: COM 4 not found
Cause	Remedy
■ Internal hardware problems.	■ Switch the analyzer off/on.

Error code: Error message	10: Gas pressure error
Cause	Remedy
Counterpressure in the analysis system too high: The carrier gas supply is automatically interrupted to protect the analyzer. In flow indication is approx. 0 ml/min. The condensate pump is running to reduce the overpressure in the system.	- Proceed in the specified order to execute the following steps for troubleshooting. - Detach the lower outlet of the water traps.  CAUTION! Risk of chemical burns! Acidic solution may escape. Wear protective equipment. - Check the filling level of the TIC condensate container. If the system is filled with liquid up to above the port on the side of the condensate pump, detach the hose 11 at the bottom of the TIC condensate container. Drain any acidic solution.  CAUTION! Risk of chemical burns! Wear protective equipment. Then, open the left side wall. If required, also drain the acidic solution from inside the condensation coil. To do this, disconnect the ground joint between the combustion tube and the condensation coil. - Find the component that is causing the gas pressure fault, see below.
The water trap is clogged.	- Reinitialize the analyzer. - Check if the gas pressure error occurs again. If not, replace the water traps.
No gas flow at the measuring outlet due to kinked hose for sample gas supply.	Check the hose. If necessary, eliminate any kinks.
The condensation coil is clogged with catalyst balls.	- Interrupt the measuring gas flow between the combustion tube and condensation coil. Check if the gas pressure error occurs again. If not, rinse the condensation coil with ultrapure water. - When replacing the catalyst, always fill in enough quartz glass wool as the first layer.
- Heavy salt deposits in the combustion tube. (During analysis of highly saline samples, salt deposits can form in the combustion tube.) - HT mat used up by analysis of highly saline samples.	Replace the HT mat in the combustion tube, or replace the catalyst. Select the measure depending on the number of measurements with the current catalyst filling and the activity of the catalyst.
The gas supply to the furnace head is clogged.	Clean the gas supply to the furnace head.
Error code: Error message	11: Change-over valve timing error
Cause	Remedy
- The change-over valve does not rotate. - The change-over valve does not stop rotating.	- Initialize the analyzer. - Check the valve positions (with the Instrument Component Test menu option, Valves tab).
Error code: Error message	12: Incorrect version number
Cause	Remedy
The version of the control software and the software of the internal computer do not match.	Perform a software update.

Error code: Error message	13: No connection to sampler
Cause	Remedy
■ The autosampler is not switched on. ■ The connection cable is not connected or is faulty.	■ Switch on the autosampler and initialize the analyzer. ■ Check the connection cables.
Error code: Error message	15: No gas pressure
Cause	Remedy
■ Gas connection not present or faulty.	■ Connect the carrier gas. Check the inlet pressure.
Error code: Error message	20: No connection to optics (NDIR)
	21: CRC error optics
	22: Status error optics
	26: Optics error; incorrect command return
Cause	Remedy
■ Communication error. ■ NDIR detector faulty.	■ Initialize the analyzer. ■ Inform the service.
Error code: Error message	24: Optics error, analog values out of range
Cause	Remedy
■ The analog values of the detector are outside of the working range.	■ Check the quality of the carrier gas. ■ Initialize the analyzer and check the analog values via the component test.
Error code: Error message	27: Optics error, analog values out of range
Cause	Remedy
■ The analog values of the detector are outside of the working range.	■ Check the quality of the carrier gas. ■ For solids methods and connection of the HT 1300 module: Set the carrier gas flow higher than the intake flow. ■ Initialize the analyzer and check the analog values via the component test.
Error code: Error message	30: No connection to N sensor
Cause	Remedy
■ The CLD detector is not switched on. ■ The connection cable is not connected or is faulty. ■ Incorrect connection.	■ Switch on the detector. ■ Check the connection cables. ■ Check the connection.
Error code: Error message	40: No connection to the syringe pump
Cause	Remedy
■ No communication between the analyzer and the syringe pump.	■ Initialize the analyzer. ■ Switch the PC off and back on again and initialize the analyzer.
Error code: Error message	80: No connection to temperature controller
Cause	Remedy
■ No connection to the solids module. ■ The solids module is not switched on. ■ Incorrect connection.	■ Check the connection cables. ■ Switch on the optional solids module. ■ Check the connection.
Error code: Error message	81: Thermocouple HT furnace interruption (HT) / UV cover open (UV)
Cause	Remedy
■ Faulty thermocouple. ■ Furnace not connected.	■ Inform the service. ■ Connect the furnace.

■ The furnace temperature is too high	■ Inform the service.
Error code: Error message	84: Communication error HT furnace temperature controller
Cause	Remedy
■ Communication error.	■ Inform the service.
Error code: Error message	86: No external furnace found
Cause	Remedy
■ No connection to the solids module.	■ Check the connection cables.
Error code: Error message	111: Rotator error
Cause	Remedy
■ The drive is incorrectly positioned, e.g. jammed. ■ The drive is faulty.	■ Initialize the analyzer. ■ If the error cannot be corrected, contact the service.
Error code: Error message	112: Swivel drive error
Cause	Remedy
■ The drive is incorrectly positioned, e.g. jammed. ■ The drive is faulty.	■ Initialize the analyzer. ■ If the error cannot be corrected, contact the service.
Error code: Error message	113: Lifting drive error / Sampler: z drive error (steps lost)
Cause	Remedy
■ The drive is incorrectly positioned, e.g. jammed. ■ The drive is faulty.	■ Initialize the analyzer. ■ If the error cannot be corrected, contact the service.
Error code: Error message	114: Rack detection error
Cause	Remedy
■ The sample tray is not positioned correctly.	■ Position the sample tray again and make sure it clicks into place. ■ Initialize the analyzer.
Error code: Error message	115: Wrong rack
Cause	Remedy
■ The wrong sample tray is set in the software.	■ Check the software settings. ■ If necessary, set a different sample tray.
Error code: Error message	116: Unknown sampler command
Cause	Remedy
■ Communication error.	■ Inform the service.
Error code: Error message	200: Restart computer in the analyzer
Cause	Remedy
■ Internal computer reset. ■ Overvoltage. ■ Short-term power failure.	■ If the LED status display is lit, initialize the analyzer. ■ For repeated occurrences, monitor precisely at which time the error occurs. Observe the status bar.
Error code: Error message	201: Restart the internal program
Cause	Remedy
■ Internal program error.	■ Initialize the analyzer. ■ For repeated occurrences, monitor precisely at which time the error occurs. Observe the status bar!

Error code: Error message	202: File method.txt not found 203: File init.cnf not found
Cause	Remedy
Program error.	Inform the service.
Error code: Error message	401: Syringe pump: Initialization 402: Syringe pump: invalid command 403: Syringe pump: invalid operand 404: Syringe pump: faulty command sequence
Cause	Remedy
- Communication error. - Syringe pump faulty.	- Initialize the analyzer. - Inform the service.
Error code: Error message	407: Syringe pump: syringe pump not initialized
Cause	Remedy
- The analyzer is not yet initialized after switching on. - Reset of the syringe pump.	Initialize the analyzer.
Error code: Error message	409: Syringe pump: pump sluggish
Cause	Remedy
A hose line is clogged.	- Search for the cause and remedy the fault. - Clean or replace the hose line. - Initialize the analyzer.
Syringe pump faulty.	Inform the service.
Error code: Error message	410: Syringe pump: valve sluggish
Cause	Remedy
- Syringe pump faulty. - The valve is broken.	Inform the service.
Error code: Error message	411: Syringe pump: pump step not permitted 415: Syringe pump: invalid command
Cause	Remedy
- Communication error. - Syringe pump faulty.	- Initialize the analyzer. - Inform the service.
Error code: Error message	MESSx: Analyzer error: MESSx measurement is cancelled
Cause	Remedy
Device error.	- Initialize the analyzer. - Acknowledge the error. Observe the display in the System state window. - Search for the cause and remedy the fault.
Error code: Error message	Peltier temperature outside range
Cause	Remedy
Peltier cooling insufficient.	- Inform the service. - Replace the water traps after repair.

Error code: Error message	Minimum sample volume > cup volume
Cause	Remedy
For sample supply via autosampler: Selected sample volume too large. Number of determinations too high.	- Check the following settings in the method: Sample volume/purge volume. - Adjust the number of determinations (repeat measurements) to the vessel volume.

7.2 Status errors

Status errors are displayed in red or yellow in the **System state** window.

Error indication	In 160 ml/min; Out < 150 ml/min
Cause	Remedy
- The union nut on the combustion tube is not tightened correctly (after catalyst replacement). - The carrier gas supply to the furnace head or lock is not connected properly (after catalyst replacement). - The sealing rings on the combustion tube are defective (heavily deformed) or not attached (after catalyst replacement). - The FAST connector on the TIC condensate container is leaky. - The connections on the water trap systems are loose (after water trap replacement or halogen trap maintenance).	- Check the screw connections for completeness and deformations. Tighten as necessary. - Check the carrier gas supply, in particular the FAST connector on the analyzer wall and the screw connection on the furnace head. - Check all connection points on the water traps. Replace the FAST connector as necessary.
The connection between the combustion tube and the condensation coil or the screw connections are leaky.	Check the connection of the combustion tube to the condensation coil, in particular the fork clamp position.
- The combustion tube is faulty (cracks, fractures at the edge). - TIC condensate container faulty (fractures on the connections).	Check the glass components. Replace as necessary.
Water traps clogged.	Replace the water traps.
The condensate pump hose is leaky.	Check the condensate pump. Replace the hose as necessary.
Error indication	In 160 ml/min; Out < 150 ml/min; Out > 170 ml/min
Cause	Remedy
The MFM (mass flow sensor) is faulty.	- Check the flow with an external mass flow sensor if possible to confirm the error. - Inform the service.
The filling of the halogen trap is used up.	Check the halogen trap.
Error indication	In < 160 ml/min; Out < 150 ml/min
Cause	Remedy
- No carrier gas. - The hose line is leaking.	- Turn on the carrier gas on the pressure reducer. - Search for and remedy the leak.

■ The inlet pressure of the carrier gas supply is too low.	■ Set the carrier gas inlet pressure correctly.
■ The pressure switch in the analyzer was triggered simultaneously with error 10: Gas pressure error.	■ See the remedy for 10: Gas pressure error.
■ The MFC is faulty.	■ Inform the service.
Error indication	In < 160 ml/min; Out = 160 ± 5 ml/min
■ No carrier gas.	■ Turn on the carrier gas on the pressure reducer.
■ The inlet pressure of the carrier gas supply is too low.	■ Set the carrier gas inlet pressure correctly.
■ The MFM is faulty.	■ Inform the service.
Error indication	In 160 ml/min; Out > 170 ml/min
Cause	Remedy
■ The Peltier cooling is insufficient, simultaneous with error message Peltier temperature outside range.	■ Check the cooling from above on the TIC condensate container. The formation of condensation water on the cooling block indicates that the cooling is working.
■ The MFC is faulty.	■ Inform the service.
Error indication	In; Out = 0 ml/min
Cause	Remedy
■ A hose line is clogged.	■ Remove and rinse the clogged hose line. Reinstall it again afterward. ■ Replace the clogged hose line.
■ No method loaded.	■ Load a method.
Error indication	Values of the NDIR detector displayed in yellow under Optical bench .
■ The analog values of the detector are at the edge of the working range.	■ Check the halogen trap. Replace the filling if necessary. ■ Contact the application team and get tips on application regulations for difficult sample matrices.

The analog values of the NDIR detector, the ADU values, can be checked in the software with the **Instrument | Component Test** menu option in the **Optical bench** tab.

You can continue measurement even if the ADU values are displayed in yellow. The display notifies you that the detector is leaving the optimum working range.

The ADU values slowly shrink due to aging. If the values drop after a few analyses, the analysis gas is probably causing damage to analyzer components.

7.3 Device errors

This section describes a number of device errors and analytic problems, some of which the user can rectify himself. Most of the device errors described are easy to identify. Most of the analytic problems lead to implausible measurement results. If the suggested solutions do not eliminate the errors/problems, and if such problems occur frequently, contact the customer service department of Analytik Jena.

Error	Water traps clogged
Cause	Remedy
■ The service life of the water traps has elapsed. ■ Measuring of samples with strong aerosol generation.	■ Replace the water trap.
Error	Scattering measurements
Cause	Remedy
■ The combustion tube filling is used up. ■ The dosing is faulty. ■ The canula is damaged. ■ Inhomogeneous samples.	■ Replace the catalyst. ■ Check the dosing. ■ Replace the canula. ■ Warm up cold samples before analysis. ■ Filter samples prior to analysis.
■ Stirring insufficient.	■ Stir particulate samples. When measuring with an autosampler, adjust the stirring speed in the method.
■ Sensitive samples can be affected by ambient air.	■ Prevent the addition of CO₂ or organic vapors from the ambient air. ■ Check the ambient conditions and remedy the source of the fault. ■ Cover the sample vessels on the autosampler with aluminum foil. ■ Treat the headspace of the sample with gas.
■ NDIR-based drift: Unsuitable integration criteria. The software ends measurement too early.	■ Check the method settings. ■ If necessary, increase the maximum integration time.
Error	Canula faulty
Cause	Remedy
■ The injection canula is corroded due to the sample matrix and the temperature. ■ The canula is clogged.	■ Misting up of the canula is normal. ■ Replace the canula if the sample is no longer dosed as a cohesive jet but is sprayed.
Error	Autosampler does not draw in sample without air bubbles
Cause	Remedy
■ The sample intake path is leaky.	■ Check the hose connections. ■ If necessary, tighten loose hose connections to the canula or to the valve of the syringe pump.
■ The sample intake canula is clogged.	■ Remove the canula and clean it in an ultrasonic bath. ■ Replace the canula.

■ The dosing syringe is leaky. ■ The PTFE sealing lips of the plunger are damaged.	■ Remove and check the dosing syringe. ■ Replace the dosing syringe.
Error	Incomplete dosing in the reactors
Cause	Remedy
■ The dosing path is leaky.	■ Check the hose connections. If necessary, tighten the following connections: Syringe pump to change-over valve, change-over valve to injection canula, change-over valve to TIC condensate container.
Error	Carry-over
Cause	Remedy
■ Insufficient syringe flushing.	■ Flush the dosing syringe with sample before the next injection. To do this, edit the method and enter a "3" in column 1 under Rinse cycles, no flush is usually required for all further measurements. Enter "0" here in the table columns.
Error	Low results (general)
Cause	Remedy
■ The catalyst is used up. ■ The system is leaking. ■ Faulty dosing. ■ Particulate samples are not or are insufficiently stirred.	■ Replace the catalyst. ■ Check the system for leaks. ■ Check the dosing. ■ Stir particulate samples.
Error	Low results for TC, TOC, NPOC and TNb analyses (TIC analyses OK)
Cause	Remedy
■ The catalyst is used up.	■ When using the platinum catalyst and measuring in differential mode (neutral to slightly alkaline samples): The catalyst can be regenerated. Inject ultrapure water that has been acidified six times (pH <2). Recommendation: Measure one or two sample vessels with acidified ultrapure water per analysis series. ■ Replace the catalyst. ■ After replacing the catalyst, perform calibration.
Error	Low results for TIC analyses (TC, TOC, NPOC analyses OK)
Cause	Remedy
■ No phosphoric acid in the reagent bottle. ■ Incorrect sample dosing.	■ Refill the bottle. ■ Check the dosing.
Error	Low results for TNb analyses
Cause	Remedy
■ The catalyst is used up.	■ Replace the catalyst.

▪ The sample concentration is above the calibrated range.	▪ Observe the calibrated range. ▪ Use quadratic regression. ▪ If possible, calibrate dependent on the matrix. ▪ For analysis of unknown substances, use low concentrations if possible. If possible, dilute the sample. ▪ Use synthetic air as a carrier gas.
Error	Unusual peak shape during TC and TNb analyses
Cause	Remedy
▪ The catalyst is used up.	▪ Note: Low results also occur at the same time. Regenerate or replace the catalyst.
▪ Unsuitable integration criteria.	▪ Check the integration criteria in the method.
▪ Measuring range for CLD exceeded.	▪ Dilute the sample.
▪ Faulty dosing.	▪ For manual sample supply: Ensure even injection.
Error	Incorrect TNb analyses with CLD (TC analyses OK)
Cause	Remedy
▪ Faulty hose connection between the analyzer and the detector. ▪ Ozone generator faulty.	▪ Check the hose connections. ▪ Inform the service.
Error	Condensate pump or phosphoric acid pump leaky
Cause	Remedy
▪ Leaking hose connections. ▪ Defective pump hose.	▪ Check the connections. ▪ Replace the hose.
Error	5 V, 24 V indicator lamps on LED strip not lighting
Cause	Remedy
▪ Power supply or electronics fault.	▪ Check the electrical connections. ▪ Check the laboratory power supply.
▪ Device fuse faulty.	▪ Inform the service.
Error	Status LED on the analyzer not lighting (Lockin)
Cause	Remedy
▪ The internal program has not been started.	▪ Switch the analyzer off and on again.
Error	Heating monitoring lamp on LED strip not lighting
Cause	Remedy
▪ Incorrect temperature settings in the software.	▪ Check the temperature settings under Configuration Options in the Analyzer components tab.
▪ Faulty thermocouple (furnace). The "Broken Thermocouple" lamp is lit on the LED strip.	▪ Inform the service.
▪ Faulty electronics component.	▪ Inform the service.
▪ The combustion furnace is not connected correctly.	▪ Check the connection of the combustion furnace.

8 Transport and storage

8.1 Transport

When transporting the device, observe the safety instructions in the "Safety instructions" section.

Avoid the following during transport:

- Impact and vibration
Risk of damage due to shock, impact or vibration!
- Large temperature fluctuations
Risk of condensation!

8.1.1 Preparing the analyzer for transport



CAUTION

Risk of burns from the furnace

The combustion furnace is still hot after the device has been switched off. There is a risk of burns.

- Allow the device to cool before removing the combustion furnace.
-



CAUTION

Risk of injury

A risk of injury due to broken glass is present when handling glass parts.

- Handle glass parts with extreme caution.
-



NOTICE

Risk of device damage due to unsuitable packaging material

- Only transport the device and its components in the original packaging.
 - Empty the device completely and attach all transport locks before transporting the device.
 - Add a suitable desiccant to the packaging to prevent damage from moisture.
-

Prepare the analyzer for transport as follows:

- ▶ Rinse the phosphoric acid pump and the corresponding hoses with ultrapure water. Empty the components.
- ▶ Switch off the analyzer via the main switch. Allow the device to cool down.
- ▶ Cut the gas supply. Disconnect the power plug from the power socket.
- ▶ Disconnect all cables and gas hoses on the rear of the analyzer.
- ▶ Open the doors of the analyzer.

- ▶ Remove the reagent bottle, the drip tray and other loose accessories. Wipe off the hose(s) with a clean paper towel.
⚠ CAUTION! The hoses contain acid residue.
- ▶ Detach the canulas from the hoses. Put the canulas in the canula packaging.
i NOTICE! Package the canulas with care. The canulas may bend.
- ▶ Remove the hoses from the connections on the halogen trap. Remove the halogen trap from the clamps.
- ▶ Remove and empty the TIC condensate container.
- ▶ Pack open hose ends in protective bags and secure them in the analyzer, for example with adhesive tape.

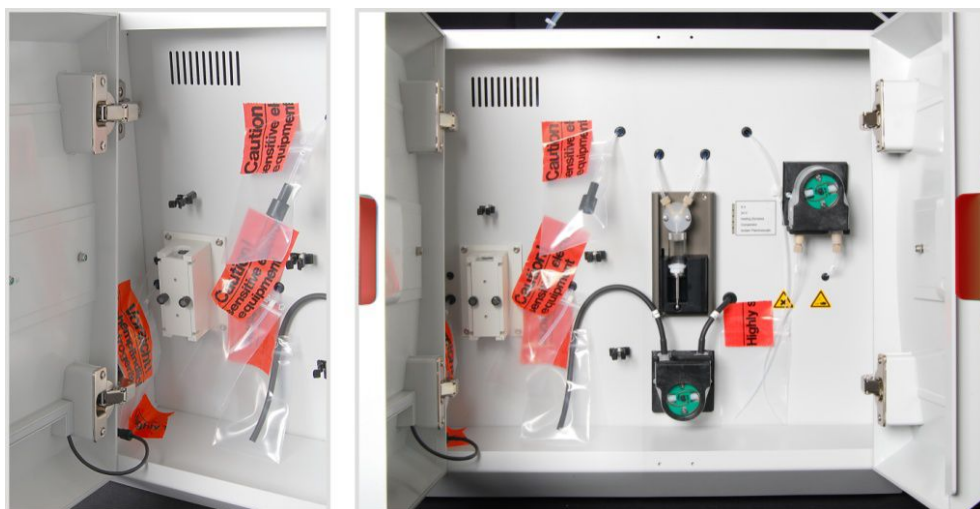


Fig. 75 Components secured behind the front doors for transport

- ▶ Open the left side wall:
 - Unscrew the four attachment screws. The screws are captive and remain attached to the wall.
 - Remove the protective grounding. Set the side wall aside safely.
- ▶ Carefully remove the condensation coil from the holder, empty it and set it aside safely.
- ▶ Remove the combustion tube.
- ▶ Remove the combustion furnace.
- ▶ Pack open hose ends inside the device in protective bags and secure them on the analyzer with adhesive tape.
- ▶ Close the left side wall of the analyzer:
 - Attach the protective grounding to the side wall.
 - First screw in the screws on the bottom side and then on the top side. Tighten the screws in turns.
- ▶ Position the top furnace cover and secure it with adhesive tape.
- ▶ Close the front doors of the analyzer.
- ▶ Carefully package the accessories. Ensure that the glass components are packed to prevent breakage.
- ▶ Package the analyzer and the accessories in the original packaging.
 - ✓ The analyzer is securely packed for transport.

See also

📄 Maintenance and care [► 86]

8.1.2 Preparing the AS vario autosampler for transport



NOTICE

Risk of device damage when transporting without transport locks

During transport without transport locks, the device can become damaged.

- Always apply a transport lock before transport.

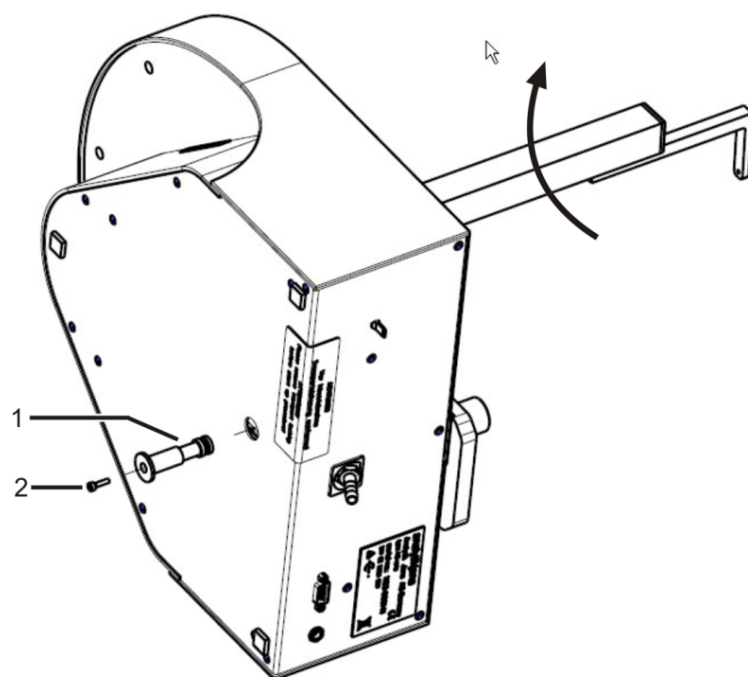


Fig. 76 Securing the autosampler for transport

1 Transport lock

2 M3x12 screw

- ▶ Turn the autosampler on its side and put it down safely.
- ▶ Turn the autosampler arm clockwise up to the stop.
 - ✓ The drives are in the correct position.
- ▶ Slide the transport lock into the opening of the bottom plate up to the stop.
- ▶ Fasten the transport lock with the screw and the supplied hexagon socket key.
- ▶ Put the autosampler into its original packaging.
 - ✓ The autosampler can now be safely transported.

8.1.3 Moving the device in the laboratory



CAUTION

Risk of injury during transport

Dropping the device poses a risk of injury and damage to the device.

- Proceed carefully when moving and transporting the device. Two persons are required to lift and carry the device.
- Grip the device firmly at the bottom with both hands and lift it simultaneously.

Observe the following when moving the device within the laboratory:

- Insufficiently secured components pose a risk of injury!
Before moving the device, remove all loose parts and disconnect all connections from the device.
- For safety reasons, two persons are required to transport the device, one person on each side of the device.
- As the device does not have carrying handles, grip the device firmly with both hands at the lower end. Lift the device simultaneously.
- Observe the guide values and adhere to the legally mandated limits for lifting and carrying loads without auxiliary means.
- Observe the installation conditions at the new location.

8.2 Storage



NOTICE

Risk of device damage due to environmental conditions

Environmental influences and condensation can destroy individual components of the device.

- Only store the device in air-conditioned rooms.
- Ensure that the atmosphere is free of dust and corrosive vapors.

If the device is not installed immediately after delivery or not required for longer periods, it should be stored in its original packaging. A suitable desiccant should be added to the equipment to prevent damage from moisture.

The requirements for the climatic conditions of the storage location can be found in the specifications.

9 Disposal

Waste water	Waste water containing acids and samples occurs during device operation. Dispose of the neutralized waste in accordance with the legal requirements.
Halogen trap	The halogen trap contains copper and brass. Contact the responsible institution (authority or waste disposal company). There you will receive the information regarding recycling or disposal.
Catalyst	The special catalysts contain $\text{Pt}(\text{Al}_2\text{O}_3)$ or CeO_2. Dispose of the used catalyst properly in accordance with the legal disposal requirements. Analytik Jena will accept the special catalyst back for disposal. Please contact the customer service department. For the customer service address, see the inside front cover.
Analyzer	At the end of its service life, the device and its electronic components must be disposed of as electronic waste in accordance with the applicable regulations.

10 Specifications

10.1 Technical data

General characteristics	Designation/type		multi N/C 3100 multi N/C 3100 pharma multi N/C 3100 duo
	Basic device dimensions (W x H x D)		513 x 464 x 550 mm
	Basic device mass		30 kg
Methods data	Digestion principle		Thermocatalytic oxidation
	Digestion temperature		Up to 950 °C, depending on catalyst
	Measuring methods		TC, TIC, TOC (differential method), NPOC, DOC, TN _b , POC
	Sample feed		Flow injection
	Sample volume		100 to 1000 µl
	Particle handling capacity		In accordance with DIN EN 1484
	Carbon detection principle		NDIR (coupled with the VITA method)
	TC, TOC, NPOC, TIC measurement range		0 to 30000 mg/l
	TC, TOC in solid measurement range (with the HT 1300 solids module)		0 to 500 mg
Nitrogen detection	Nitrogen detection principle (optional)		CLD ChD
	TN _b measurement range (CLD)		0 to 20000 mg/l
	TN _b measurement range (ChD)		0 to 10000 mg/l
Process control	Control and analysis software		multiWin
	Software function scope		Real-time graphics, status indication during analysis, graphical display of the measured results, result print-out Data integrity and conformity with pharmaceutical guidelines 21 CFR Part 11 and Eu-draLex Volume 4 Annex 11 in the pharmaceutical software.
Gas supply	Option 1	Oxygen	≥4.5
	Option 2	Synthetic air (from a compressed gas cylinder)	Hydrocarbon and CO ₂ -free
	Option 3	Purified compressed air (provided by a TOC gas generator)	CO ₂ <1 ppm Hydrocarbons (as CH ₄) <0.5 ppm
	Inlet pressure	400 to 600 kPa	
	Flow rate	15 l/h, depending on measuring mode	

	Analyte gas flow	160 ml/min
	NPOC purge flow	50 to 160 ml/min
Electrical variables	Voltage	115/230 V
	Frequency	50/60 Hz
	Fuses	2 T6.3 A H
	Typical average power consumption	400 VA
	Maximum power consumption	500 VA
	PC interface	USB 2.0
	Module/accessory interface	RS 232
	Only use original fuses from Analytik Jena!	
Ambient conditions	Operating temperature	+10 to 35 °C (air-conditioning recommended)
	Maximum humidity	90 % at 30 °C
	Air pressure	0.7 to 1.06 bar
	Storage temperature	5 to 55 °C
	Humidity during storage	10 to 30 % (use desiccant)
	Operating altitude (max.)	2000 m
Control computer minimum requirements	Processor	Min. 3.2 GHz
	Disk drive	Min. 40 GB
	RAM	Min. 4 GB
	Screen resolution	Min. 1280 x 780
	USB port	Min. 1 USB 2.0 interface, for connecting the basic device
	CD/DVD drive	For software installation
	Operating system	Windows 10 or higher, Windows 7/8.1 is supported, 32 or 64 bit

10.2 Standards and directives

Protection class and protection type	The device is protection class I.
Device safety	The device complies with the following safety standards: ■ EN 61010-1 ■ EN 61010-2-081 ■ EN 61010-2-010 ■ EN 61010-2-051 (for operation with autosampler)
EMC compatibility	The device has been checked for transient emissions and noise immunity. It meets the requirements for transient emissions according to ■ EN 61326-1 (EN 55011 group 1, class B)

	The device meets the requirements for noise immunity according to ■ EN 61326-1 (requirements for use in a basic environment)
Environmental and ambient influences	This device has been tested in environmental simulations under operation and transport conditions and is in accordance with the requirements in: ■ ISO 9022-2 ■ ISO 9022-3
EU directives	The device meets the requirements of the directive 2011/65/EU. The device is designed and tested in accordance with standards meeting the requirements of EU directives 2014/35/EU and 2014/30/EU. The device leaves the factory in a sound condition with regard to technical safety. To maintain this condition and to ensure safe operation, the user must strictly observe the safety and operating instructions contained in this operating manual. For accessories delivered with the device and system components from other manufacturers, the information provided in their respective operating manuals has priority.
Guidelines for China	The device contains substances subject to regulation (according to the directive GB/T 26572-2011). Analytik Jena guarantees that, if the device is used as intended, these substances will not leak within the next 25 years and therefore will not pose a threat to the environment or health within this time period.

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