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# Operating Manual

multi N/C 3100

multi N/C 3100 pharma

TOC/$TN_b$ Analyzers

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# Content

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# 1 Basic Information

## 1.1 User manual notes

The user manual describes the following two models of the multi N/C series:

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

In the text below these devices are collectively called multi N/C 3100. Differences are explained in the corresponding section.

The analyzer multi N/C 3100 is intended for operation by qualified specialist personnel observing this user manual.

The user manual informs about the design and operation of the analyzer and provides personnel familiar with TC/TN analysis the necessary know-how for the safe handling of the equipment and its components. The user manual further includes notes on the maintenance and service of the equipment and potential causes and remedies of any faults.

Conventions

**Instructions for actions** which occur in chronological order are numbered and combined in action units.

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

The elements of the control and analysis program are indicated as follows:

- Program terms are indicated by small caps (e.g., SYSTEM menu).
- Buttons are shown by square brackets (e.g., [OK]).
- Menu items are separated by arrows (e.g., SYSTEM ▸ DEVICE).

Symbols and signal words

The operating instructions use the following symbols and signal words to indicate hazards or instructions. The warnings are always placed before an action.

**WARNING**

Indicates a potentially hazardous situation, which might cause death or very serious injuries (deformities).

**CAUTION**

Indicates a potentially hazardous situation, which might cause light or minor injuries.

**Attention**

Provides information on potential material or environmental damage.

## 1.2 Application fields

The analyzer multi N/C 3100 is a device to determine the total carbon content and/or total nitrogen content in aqueous samples. Detection is by way of thermocatalytic digestion in the presence of a special catalyst in accordance with the national and international standards.

Complementing the analyzer with a chemiluminescence detector (CLD) or an electrochemical NO detector (ChD, not for multi N/C 3100 pharma) permits the detection of $TN_b$ (total nitrogen bound).

The analyzer multi N/C 3100 is versatile in its application due to its large measuring range, high degree of automation and expandability.

multi N/C 3100 pharma

- Pharmacy, medicine, biotechnology

The multi N/C 3100 pharma offers tailored solutions for ultrapure water control and extractables testing from packaging materials in pharmaceutical samples. The multiWin pharma software fully complies with the requirements of the FDA 21 CFR part 11 and EudraLex Vol. 4 Annex 11.

multi N/C 3100

- Water treatment

The multi N/C 3100 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.

- Environmental monitoring

The analysis of surface water (e. sea water), which often has a low TOC content and high TIC concentrations as well as high salt content, is possible using special analysis modes (see section "NPOC analysis according to the NPOC plus method" p. 32).

- Power stations and laboratory

With its dynamic measuring range the multi N/C 3100 meets the relevant conditions for TOC detection in power stations and steam generators.

- Waste and soil

The carbon detection (TC/TOC detection) in solid samples is possible with the extension of the multi N/C 3100 by the solids module HT 1300. Eluates can also be analyzed efficiently due to the possibility of the simultaneous TC/$TN_b$ detection in liquid samples.

- Research and teaching

Due to the many configuration options the multi N/C 3100 suitable for use in teaching and research. In conjunction with a solids module HT 1300 the TC in solids can be determined.

## 1.3 Intended use

The analyzer multi N/C 3100 must only be used for the methods for the detection of the total carbon and/or the total nitrogen content in aqueous samples as described in this user manual and in conjunction with an external solids module for the detection of the total carbon content of solid samples. Any other use is not as intended! Only the operator is liable for any damages that result from this.

In particular it is prohibited to use the analyzer to analyze flammable liquids or substances that could farm explosive mixtures. No concentrated acids may be analyzed with the analyzer.

The device must only be operated with synthetic air, purified air or oxygen as carrier gas (see section "Technical data" p. 136).

The operational safety of the analyzer N/C 3100 is only ensured during proper use according to the information in this user manual. The intended use also includes the adherence to the installation conditions prescribed by Analytik Jena GmbH which are available from the customer service address stated above.

## 1.4 Warranty and liability

The warranty duration and liability comply with the legal requirements and the provisions in the general terms and conditions of Analytik Jena GmbH.

Deviations from the intended use described in this user manual result in limitations of warranty and liability during a damage event. Damage to wearing parts is not included in the warranty.

Warranty and liability claims are excluded for personal injury and property damage due to one or several of the following causes:

- use of the analyzer multi N/C 3100 other than intended
- improper commissioning, operation and service of the analyzer
- modifications of the equipment without prior consultation with Analytik Jena GmbH
- unauthorized intervention in the equipment
- operation of the equipment with faulty safety equipment or improperly fitted safety and protection equipment
- inadequate monitoring of the equipment components subject to wear
- use of other than original spare parts, wearing parts or consumables
- improper repairs
- faults due to the non-observance of this user manual

# 2 Safety instructions

For your own safety and to ensure error-free and safe operation of the analyzer multi N/C 3100, please read this chapter carefully before using the appliance.

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

## 2.1 Safety markings on the analyzer

Safety symbols have been attached to the analyzer and accessories whose content must always be observed.

Damaged or missing safety symbols can cause incorrect actions leading to personal injury or material damage! The safety symbols must not be removed! Damaged safety symbols must be replaced without delay!

The following safety symbols have been attached to the analyzer and accessories:

Fire, naked flame and smoking prohibited!

Warning against dangerous electrical voltage

Hazardous substances warning

Warning against hot surface

Warning against corrosive substances

Before opening the housing switch off the device and disconnect the mains plug from the mains connection.


The device contains restricted substances. Analytik Jena GmbH guarantees, that those hazardous substances may not leak out during the next 25 years when the device is used in accordance with its intended purpose.

## 2.2 Technical condition

The analyzer corresponds in its design and construction to the current state of the art technology. Unauthorized modifications or changes, especially such that affect the safety of the staff and the environment, are generally not allowed.

The following has to be observed:

- Any manipulation of the safety equipment is prohibited! In case of an accident manipulations of the safety equipment will be interpreted as deliberate!

- The operator must only operate the analyzer in a sound and operationally safe condition. The technical condition must always comply with the legal requirements and regulations.
- Prior to every use the analyzer must be checked for damage and sound condition.
- Any changes in the analyzer affecting its safety must be reported by the operating personnel to the operator without delay.
- The equipment components must only be connected to supply cables intended and designed for this purpose.
- All safety equipment and interlocks must be well accessible and regularly checked for proper operation.

## 2.3 Requirements for the operating personnel

The analyzer multi N/C 3100 must only be operated by qualified specialist personnel instructed in the use of the analyzer. The instruction must also include conveying the content of this user manual and the user manuals of other system components or add-on equipment.

The analyzer may pose dangers if it is not used by trained personnel, improperly or other than intended.

Therefore, every person tasked with the operation of the analyzer must have read and understood this user manual and the user manuals of any additional equipment before carrying out the respective tasks. This also applies if the respective person has already worked with or been trained on this kind of analyzer.

It is recommended that the operator have the operating personnel confirm the knowledge of the content of the user manual in writing. The ultimate responsibility for the accident-free operation of the analyzer rests with the operator or the specialist personnel authorized by him.

In addition to the safety at work instructions in this user manual the generally applicable safety and accident prevention regulations of the respective country of operation must be observed and adhered to. The operator must ascertain the latest version of these regulations.

The user manual must be accessible to the operating and service personnel at any time!

The following has to be observed:

- The analyzer must only be commissioned, operated and serviced by trained personnel instructed in technical safety.
- The operation or servicing of the analyzer by minors or individuals under the influence of alcohol, drugs or medication is not permitted.
- It must be ensured that only authorized personnel works at the analyzer.
- The operating personnel must be familiar with the dangers arising from measuring liquids. The appropriate protective equipment must be used.
- Prior to pauses or at the end of the work appropriate skin cleaning and protection measures must be carried out.
- Eating, drinking, smoking or handling open flames in the operating room of the analyzer is prohibited!

## 2.4 Safety instructions, transport and installation

The analyzer is always installed by the customer service department of Analytik Jena GmbH or its authorized and trained specialist personnel. Independent assembly and installation are not permitted. Incorrect installation can create serious hazards.

The following has to be observed:

- Insufficiently secured components pose a risk of injury! During transport the components of the equipment must be secured in accordance with the instructions in the user manual.
- Only transport the analyzer in its original packaging! Ensure that the transport protections have been fitted and the analyzer is completely empty.
- To prevent health damage the following must be observed when moving the analyzer in the laboratory (lifting and carrying):

For reasons of safety 2 persons are required to transport the analyzer and must position themselves on both sides of the equipment.

Because the analyzer does not feature any handles, firmly grip the device from the bottom and make sure prior to simultaneous lifting the device that the sensitive components at the front are protected by the closed doors.

The guide values and statutory limits for lifting and carrying loads without auxiliary equipment must be observed and adhered to.

## 2.5 Safety instructions - operation

### 2.5.1 General

The operator of the analyzer must make sure before each commissioning that the condition of the analyzers including the safety equipment is sound. This applies in particular after each modification or extension of the analyzer or its repair.

The following has to be observed:

- The analyzer must only be operated if all protective equipment (e.g. covers, drip pans for chemicals and doors) are present, 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 decommissioned during operation.
- Free access to the power switch on the back of the enclosure must be ensured during operation.
- The ventilation equipment on the N/C 3100 and the extension modules must be in good working condition. Covered vents or ventilation slits etc. may cause the device to break down or may cause damage to it.
- The furnace works at temperatures of up to 950 °C. Hot components (furnace, condensation coil) must not be touched during or directly after the operation of the analyzer.
- Keep all combustible materials away from the analyzer.

### 2.5.2 Safety instructions - Protection against explosion and fire

The analyzer must not be operated in an explosive environment. Smoking or handling open flames in the operating room of the analyzer is prohibited!

The operating personnel have to be familiar with the location of the fire-fighting equipment in the operating room of the analyzer.

### 2.5.3 Safety instructions - electrical equipment

Work on electrical components of the analyzer may only be carried out by a qualified electrician in accordance with the applicable electrical engineering rules. Life-threatening electrical voltages may occur in the right-hand side component of the analyzer.

The following has to be observed:

- Extension modules or system components must always be connected to or disconnected from the analyzer in a deactivated condition.
- Before opening the analyzer it must be switched off from the equipment switch and the mains connector must be disconnected from the mains outlet!
- Any work on the right-hand side component of the analyzer may only be carried out by the customer service of Analytik Jena GmbH and specially authorized technicians.
- The electrical components must be checked regularly by a qualified electrician. Any defects, such as loose connections, faulty or damaged cables, must be repaired without delay.
- The analyzer must be switched off immediately at the power switch (on the equipment backplate) and the power supply disconnected from the mains if there is any interference with the electric components.

### 2.5.4 Safety instructions for compressed gas containers and systems

The carrier gas (synthetic air and/or oxygen) is taken from compressed gas containers or local compressed gas systems. The required purity of the carrier gas must be ensured (→ see chapter "Technical data" p. 136)!

Work on compressed gas containers and systems must only be carried out by individuals with specialist knowledge and experience in compressed gas systems.

The following has to be observed:

- For the operation of compressed gas containers or systems the safety instructions and guidelines applicable at the operating site must be adhered to in full.
- High pressure 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.
- All pipes, hoses and screw connections must be checked regularly for leaks and externally visible damage. Leaks and damaged must be repaired without delay.
- Prior to inspections, service and repairs the valves must be closed and the analyzer vented!

- After successful repair and service of the components of the compressed air containers or system the analyzer must be checked for sound operation prior to recommissioning!
- Independent assembly and installation are not permitted!

### 2.5.5 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 dangerous substances local safety codes and guidelines must be observed.

The following general notes do not replace the specific local regulations or the regulations in the EU safety data sheets of the manufacturers for the auxiliary and operating materials.

The following has to be observed:

- The relevant regulations and the notes in the EU safety data sheets of the manufacturers have to be observed and complied with regards to storage, handing, use and disposal for all auxiliary and operation materials used during operation or maintenance of the analyzer.
- Auxiliary and operation materials may never be placed in containers or vessels for food. The approved containers for the relevant substance are to be used and these have to be labeled accordingly. The notes on the labels have to be observed!
- Protective goggles and rubber gloves have to be worn when handing reagents. The notes on the labels have to be observed.
- The regulations and notes on the safety data sheets for the handling of orthophosphoric acid ($H_3PO_4$) or hydrochloric acid (HCl) have to be observed!
- The catalyst supplied by the manufacturer should be handled with the usual caution when handling chemicals.
- Biological samples have to be handled according to local guidelines regarding the handling of infectious material.
- Caution when handing quartz glass and glass parts. Risk of broken glass and therefore risk of injury!
- Avoid dust formation and inhalation of dust when handing the quartz glass wool during filling of the combustion tube.
- Auxiliary and operating materials as well as their containers may not be disposed in domestic waste or enter the sewage system or the soil. The applicable regulations for disposal of these materials must be meticulously observed.
- Ensure good room ventilation in working rooms.

### 2.5.6 Safety instructions - service and repair

The analyzer is usually serviced by the customer service department of Analytik Jena GmbH or its authorized and trained specialist personnel.

Independent servicing can misadjust or damage the analyzer. Therefore, the operator may only carry out the tasks listed in the chapter "Service and care".

The following has to be observed:

- The exterior of the analyzer may only be cleaned with a damp, not dripping, cloth after the analyzer has been switched off.
- Any service and repair work at the analyzer may usually only be carried out in the switched-off condition (unless stated otherwise).
- Service tasks and the replacement of system components (removal of the combustion tube, catalyst replacement) must only be carried out after a sufficiently long cooling down phase.
- Prior to servicing or repair, the energy and gas supplies must be disconnected and the analyzer must be v vented!
- Only use original accessories and original replacement parts from Analytik Jena GmbH. The notes in the chapter "Maintenance and care" must be observed.
- All protective equipment must be reinstalled correctly immediately after completion of the service and repair work and be checked for operation!

## 2.6 Behavior during emergencies

The analyzer must be switched off from the power switch (on the equipment backplate) and the power supply has to be disconnected from the mains in case of dangerous situations or accidents.

Because a rapid response can save lives during an emergency, the following has to be ensured:

- The operating staff must be familiar with the location of safety equipment, accident and danger alarms as well as first aid and rescue equipment as well as their handling.
- The operator is responsible for the respective training of the operating staff.
- All equipment for first aid (first-aid kit, eyewash bottles, stretcher, etc.) as well as equipment for firefighting (fire extinguishers) must be within reach and easy to access. All equipment has to be in a sound condition and should be checked regularly.

# 3 Function and layout

## 3.1 System design

The analyzer multi N/C 3100 is a compact laboratory device with permanently installed main components. The complete measuring design further includes accessory parts and reagents, which must be connected to the analyzer or made ready prior to a measurement.

The control of the analyzer and the analysis of the measurements takes place via the control and analysis software multiWin® installed on an external PC.

All components of the analyzer to be operated or serviced by the user can be reached via 2 doors at the front, the left-hand removable side wall or the top cover.

The analyzer multi N/C 3100 consists of the following main components:

- Components for sample preparation
- Hose system
- Combustion system
- Components for measuring gas drying and cleaning
- Detectors
- Display and control elements, connectors
- Electronic component
- Accessories

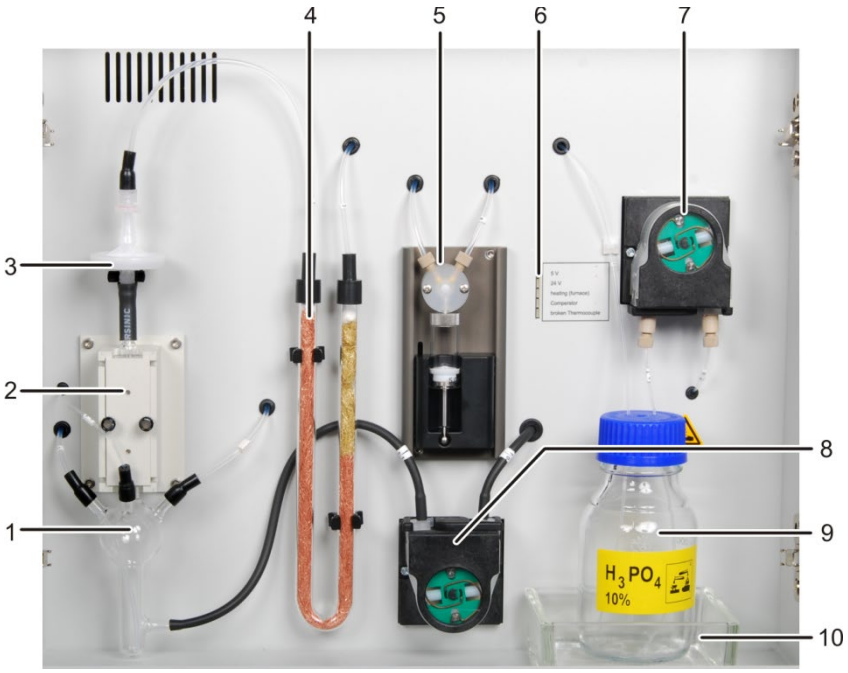



**Fig. 1 Front view (doors open)**

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 Collecting dish

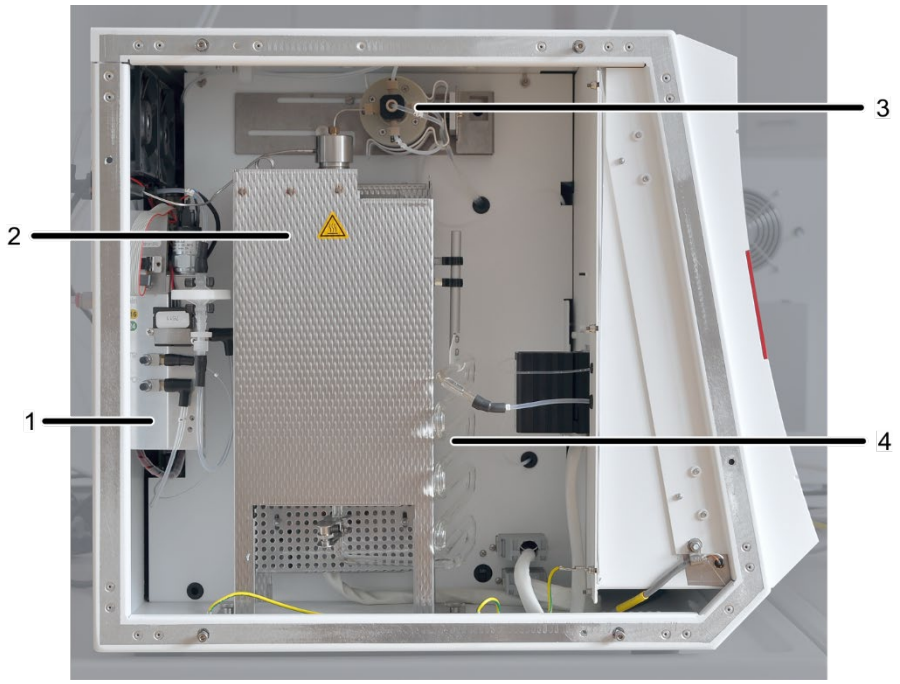


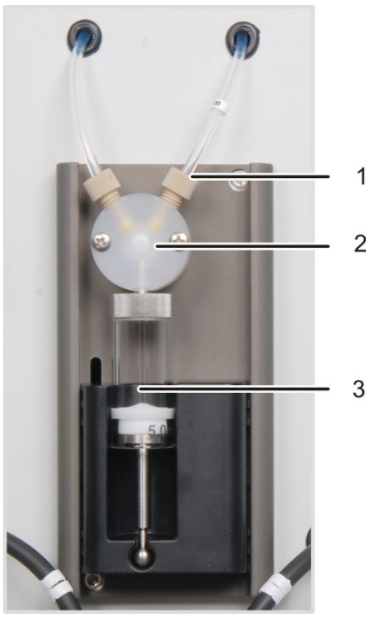

**Fig. 2 Lateral view left (side wall removed)**

1 Gas box
2 Combustion system
3 5-port directional valve
4 Condensation coil

### 3.1.1 Components for sample preparation

Sample feeding in the analyzer N/C 3100 takes place via flow injection over a syringe pump with 2-port valve (see fig. 3). The injection volume is 100 – 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.




1 Fingertight connection
2 2-port valve
3 Metering syringe

**Fig. 3 Syringe pump with 2 port valve**

The hoses at the 2-port valve are labeled and connected to the following components:

- Hose no. 8 to the ultrapure water bottle
- Hose no. 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 encircled numbers and letters in the hose diagram correspond to the labels on the hoses in the analyzer.

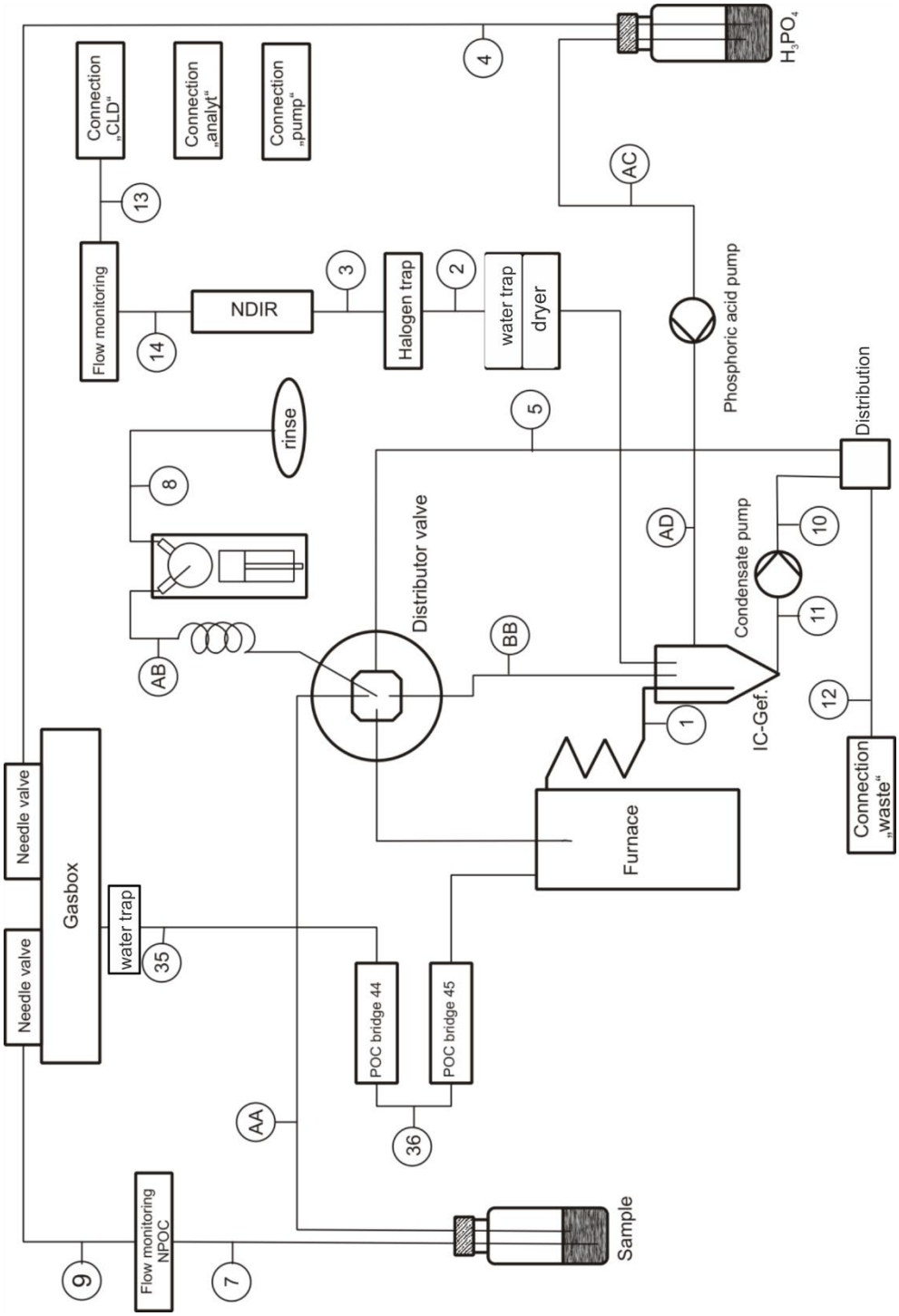


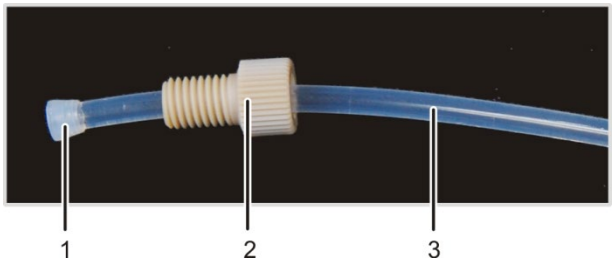

**Fig. 4 Hose diagram**

Connection method

Within the device most gas connections have been implemented via a FAST connector (FAST – **Fa**st, **S**ave, **T**ight). These connectors provide a tight transition between the hoses and connections with different diameters. The soft sleeves prevent the risk of glass fractures compared to rigid screw connections. There are different versions of the connectors.

**Fig. 5 Different FAST connector designs**

In additions, so-called Fingertight screw connections are used in the analyzer multi N/C 3100. 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.




1 conical nipple
2 banjo bolt
3 Hose type

**Fig. 6 Fingertight screw connection**

Components for flow adjustment

The flow of the carrier gas is adjusted automatically in the multi N/C 3100 and the inlet flow is controlled via an MFC (mass flow controller). At the device outlet the carrier gas flow is measured using an MFM (mass flow meter), i.e. an automatic tightness check is carried out. The result is displayed in the control and analysis software multiWin in the window SYSTEM-STATUS.

The NPOC purging flow is adjusted via a needle valve left to the combustion furnace at the gas box. It is only accessible with the left side wall removed. The NPOC purging flow is measured using an MFM and displayed in the control and analysis software in the window SYSTEM-STATUS.

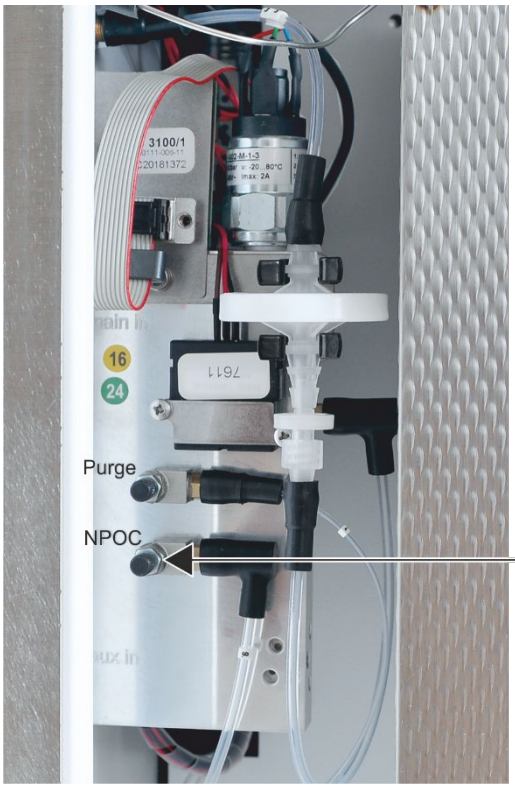



**Fig. 7 Needle valve to adjust the NPOC purging flow (see arrow)**

Hose pumps

Condensate pump

Via the condensate pump the condensate or the waste solution from TIC detection are automatically pumped off after each measurement. The condensate pump is located behind the right door next to the halogen trap.

**Fig. 8 Condensate pump**

Phosphoric acid pump

The phosphoric acid pump transports the 10% phosphoric acid to the TIC condensate container with the phosphoric acid being permanently vented.

Fig. 9 Phosphoric acid pump

### 3.1.3 Combustion system

The combustion system is on the rear left side wall of the analyzer.

The combustion furnace is a resistor-heated vertical furnace for digestion temperatures 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 has to 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.

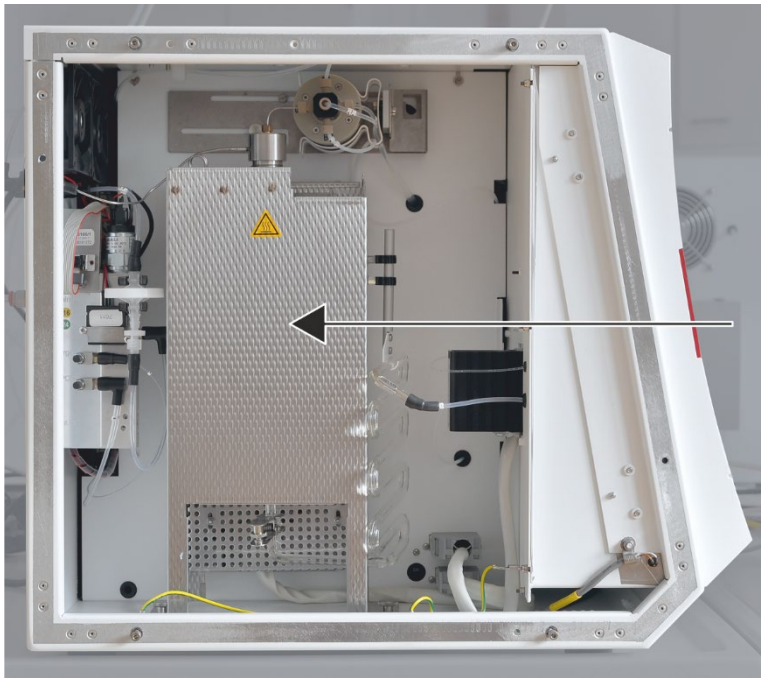


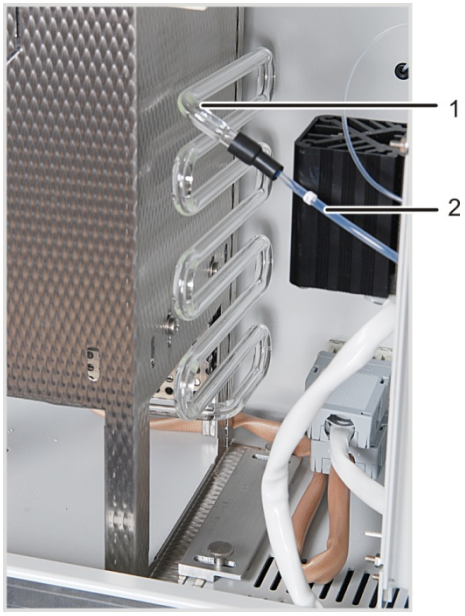
Fig. 10 Combustion furnace (see arrow)

### 3.1.4 Components for 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 measuring gas is cooled down quickly in the condensation coil and the water vapor condenses. The measuring gas water mixture is routed to the TIC condensation container via a hose.




1 Condensation coil
2 Hose no. 1 to the TIC condensation container

**Fig. 11 Clean condensation coil**

TIC condensation module

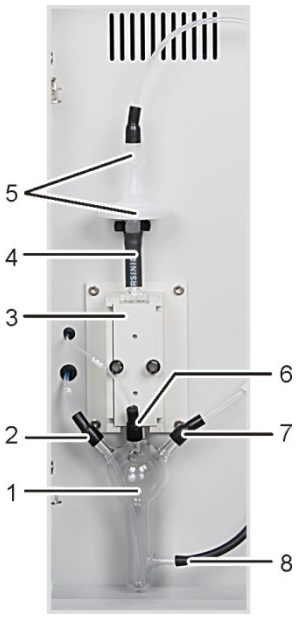
The TIC condensation module consists of a TIC condensate container and a cooling block. In the TIC condensate container the TIC reactor and the gas/liquid separator are combined. At the same time the measuring gas is dried via the cooling block.

The TIC condensation module is located on the left front side. The measuring gas/water mixture is fed via the top left connection via hose no. 1.

Via the top center connection at the glass container 10% phosphoric acid is automatically provided before each measurement.

The measuring gas is dried by freezing in the cooling block. 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 or the waste solution of the TIC detection is automatically pumped out via the lower lateral outlet at the glass container.

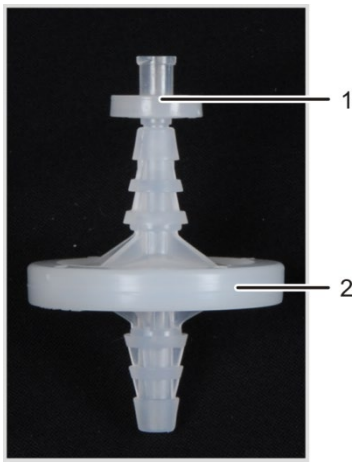



1 TIC condensate container
2 Connection hose no. 1 / measuring gas supply from the condensation coil
3 Cooling block
4 Connection at the water traps
5 Water traps
6 Connection hose no. AD / phosphoric acid supply
7 Connection hose no. BB / direct sample supply for TIC detection
8 Connection at the condensate pump (waste hose no. 11)

**Fig. 12 TIC condensation module**

Water traps

The analyzer multi N/C 3100 contains water traps for the greatest possible removal of interfering components in the measuring gas and to protect the detector and the gasbox. They are installed in the gas path after the cooling block (5 in Fig. 12) and after the gasbox. The water traps each consist of a larger water trap (TC Pre-filter), which retains aerosol during operation, and a smaller water trap (disposable retention filter), which collects rising water.




1 Disposable retention filter
2 TC Pre-filter

**Fig. 13 Water traps**

Halogen trap

The analyzer multi N/C 3100 contains a halogen trap (U tube) after the TIC condensate container and the water traps for the greatest possible removal of interfering components in the measuring gas and to protect the detector.

The U-tube is filled with special copper wool and brass wool. The filling of the halogen trap has to be renewed at the latest once half of the copper wool is discolored black or the brass wool is discolored.

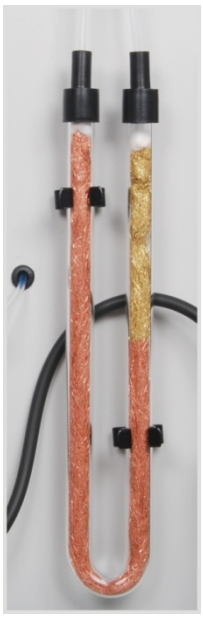


Fig. 14 Halogen trap

### 3.1.5 Detectors

NDIR detector

The NDIR detector (non-dispersive infrared absorption detector) is on the rear 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 gases in the active IR range, these gas components absorb the characteristic wave lengths with their proportional share of the total radiation according to their concentration in the gas mixture.

The radiation receiver inside the NDIR detector used is selective for the $CO_2$.

Measurements using the VITA method

The $CO_2$ molecules are detected by the measuring technology as long as they remain in the cell of the NDIR detector. Due to fluctuations of the measuring gas during the $CO_2$measurement (e.g. on account of evaporation and condensation processes when metering the liquid samples) $CO_2$ molecules are sometimes detected by spectrometry for longer (low gas flow) or shorter (high gas flow) periods of time.

Using the VITA method (dwell time coupled integration for TOC analyses) the measuring gas flow is detected parallel to the NDIR signal. Occurring flow variations are compensated to a constant gas flow by computer-controlled normalization of the signal and only integrated afterwards.

To this end, a high-accuracy, digital flow sensor is arranged very close to the NDIR detector flow.

Electrochemical NO detector (ChD, optional)

For $TN_b$ detection the electrochemical NO detector can be used with model multi N/C 3100, but not with model multi N/C 3100 pharma. The NO detector is behind the rear right side wall of the analyzer. It analyzes the nitrogen oxide (NO) content in the carrier gas.

The measuring gas formed by thermal oxidation enters the detector where nitrogen oxides contained in it are diffused via a highly selective diaphragm into the electrochemical measuring cell. The oxidation of the nitrogen oxides occurring at the anode modifies the flow between the electrodes proportionally to the nitrogen oxide concentration. The change of the flow is evaluated as a signal and enables the detection of

the nitrogen content of the analyzed sample ($TN_b$ detection). The electrolyte of 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). To maintain the internal balance of the ChD when measurements are paused (the analyzer is switched off), a support voltage is required. This is implemented via a battery (U9VL).

Chemiluminescence detector CLD (optional)

The extension of the analyzers N/C 3100 with a chemiluminescence detector enables the $TN_b$ detection. The CLD must be positioned as an external device to the right of the multi N/C 3100.

The measuring gas formed by the thermal oxidation of the sample is dried and then enters the reaction chamber of the chemiluminescence detector for the $TN_b$ detection. There the nitrogen monoxide present in the measuring gas is oxidized with ozone into activated nitrogen dioxide. By emitting light photons (luminescence) the molecules of the nitrogen dioxide return to their original state. The luminescence is detected using a photomultiplier and is proportional to the nitrogen monoxide concentration. It thus permits the detection of the total nitrogen content of the analyzed sample.

A digestion for the $TN_b$ detection cannot result in a 100% NO recovery. During the cooling and condensation process of the combustion nitrogen oxides can also occur at higher oxidation levels.

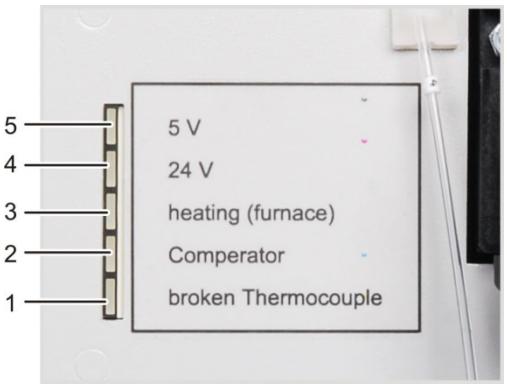
### 3.1.6 Display and control elements, connectors

LED displays

The green LED at the left door of the analyzer illuminates after the analyzer has been switched on.


**Fig. 15 Diode to indicate readiness for operation**

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




1 broken thermocouple
2 furnace comparator
3 Furnace heating
4 device voltage
5 internal firmware controller

**Fig. 16 LED strip (right door open)**

Main switch, interfaces, gas connections at the back of the equipment

Main switch, mains connection, equipment fuse, media connections (gases and waste) and the interfaces for connecting the PC and the accessories are at the back of the multi N/C 3100.

A diagram at the center of the backplate explains the different connections.

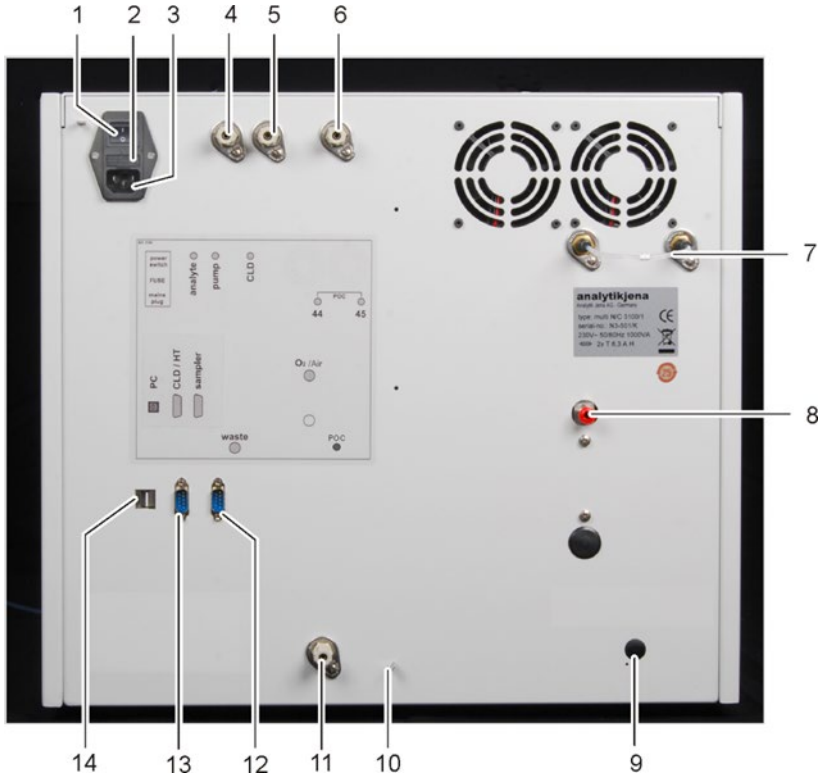



**Fig. 17 Connections on the rear of the device**

1 Main switch to switch the analyzer on and off "power switch"
2 Holder for mains fuse "FUSE"
3 Mains connection "main plug"
4 Gas connection "analyte"
5 Gas connection "pump"
6 Connection "CLD"
7 Bridge for the gas connection of the POC module
8 Carrier gas connection "$O_2$/Air"
9 Connection for POC module (optional)
10 Connection of neutral conductor at the sampler
11 Waste
12 RS 232 interface for sampler "sampler"
13 RS 232 interface for CLD and HT module "CLD/HT"
14 USB port for PC

### 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 pan for phosphoric acid $H_3PO_4$, 250 ml
- ultrapure water bottle, 2.5 l

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

### 3.1.8 Extension options for the analyzer

Sampler

Six different autosamplers are available for the multi N/C 3100 and multi N/C 3100 pharma:

- AS vario with various tray sizes
- AS vario ER with needle rinsing
- AS 21 for 21 samples
- AS 10 for 10 samples
- POC sampler for POC measurements
- EPA sampler with piercing function

The AS vario (ER), the POC sampler and the EPA sampler must be positioned on the left-hand side of the basic device. The AS 21 and AS 10 samplers must be screwed to the right-hand side of the basic device.

Chemiluminescence detector CLD (optional)

For $TN_b$ detection a chemiluminescence detector (CLD) can be connected to the analyzers multi N/C 3100 and multi N/C 3100 pharma.

POC module

The extension of the analyzer N/C 3100 with a POC module enables the direct detection of POC in aqueous samples.

External solids module HT 1300

The extension of the analyzer multi N/C 3100 with the external solids module HT 1300 enables the catalyst-free digestion of solid samples in a ceramic combustion tube. At temperatures up to 1300 °C the ceramic boats can hold big sample quantities (up to 3 g) in order to account for more inhomogeneous samples.

TIC solids module

The TIC in solid samples can be determined by extending the multi N/C 3100 with the TIC solids module. Big sample quantities can be weighed into Erlenmeyer flasks. Samples are digested by acid while stirred on a heating plate in order to release $CO_2$ from carbonates and bicarbonates.

## 3.2 Principle of operation

The analyzer multi N/C 3100 is a compact and powerful device to determine the total organic carbon content and/or total nitrogen content in aqueous samples.

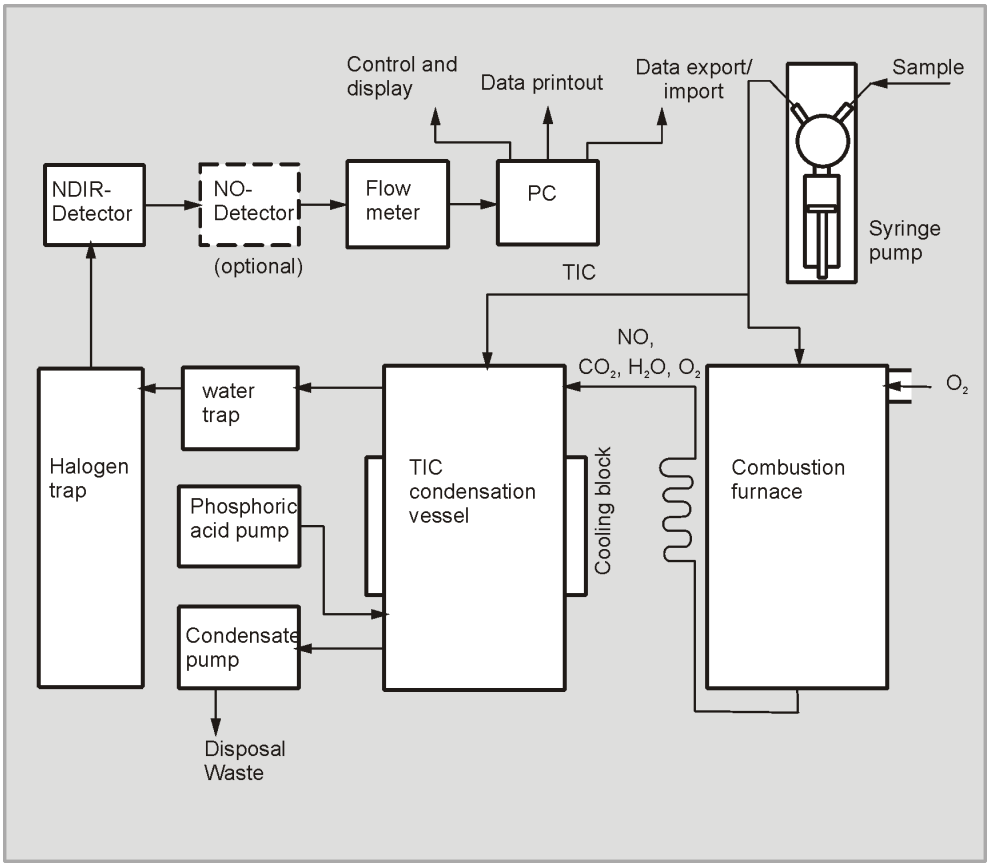



**Fig. 18 Principle of operation**

The digestion is performed in the multi N/C 3100 by thermocatalytic high-temperature oxidation in the presence of special catalysts. This enables a quantitative digestion even for very stable, complex carbon and nitrogen compounds.

The sample aliquot is directly dosed 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 (e. equations (1) - (3)). The carrier gas is also used as an oxidation agent.

$$R + O_2 \rightarrow CO_2 + H_2O \quad (1)$$

$$R - N + O_2 \rightarrow NO + CO_2 + H_2O \quad (2)$$

$$R - Cl + O_2 \rightarrow HCl + CO_2\ H_2O \quad (3)$$

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 acting gases, the measuring gas $CO_2$ is added to the NDIR detector or NO detector (CLD or ChD).

Inorganic carbon is detected by injecting a sample aliquot into the acidic TIC reactor and driving out the formed $CO_2$ via the NDIR detector.

The $CO_2$ or NO concentration is detected several times every second. An integer is calculated from this signal sequence. The integer 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.

## 3.3 Measuring method

The analyzer multi N/C 3100 is used to determine the following parameters as sum parameters.

- TC - total carbon
- TOC - total organic carbon
- TIC - total inorganic carbon
- NPOC - non-purgeable (non-volatile) organic carbon
- POC - purgeable (volatile) organic carbon
- $TN_b$ - total bound nitrogen

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

### 3.3.1 TC analysis

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

The sample is metered automatically via a syringe into the combustion tube, digested and the generated carbon dioxide is detected.

Parallel to the TC detection the $TN_b$ detection is possible.

### 3.3.2 TOC analysis

During the TOC analysis the total organic carbon content of a sample is detected.

The TOC determination in the analyzer is performed according to the differential method, which can be described with the following equation (4).

$$TOC = TC - TIC \quad (4)$$

TOC ... total organic carbon
TC ... total carbon
TIC ... total inorganic carbon

Two sequential measurements are used in the same sample consecutively 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.

The TOC analysis should be used when the sample contains easily purgeable organic substances as benzene, cyclohexane, chloroform, etc. The TOC analysis should not be used when the TIC content of the sample is significantly higher than the TOC content.

Parallel to the TOC detection the $TN_b$ detection is possible.

### 3.3.3 TIC analysis

During the TIC analysis the total inorganic carbon from carbonates and hydrocarbonates as well as dissolved $CO_2$ is detected.

Cyanides, cyanate, isocyanate and carbon particles are not detected.

An aliquot of the sample is directly dosed into the TIC reactor to determine the inorganic carbon materials (TIC). The $CO_2$ is purged and detected.

### 3.3.4 NPOC analysis

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

The sample is acidified outside of the analyzer with 2 N HCl (pH 2) and the resulting $CO_2$ is purged. Afterwards the remaining carbon from the sample prepared in this manner is determined via combustion.

Parallel to the NPOC detection the $TN_b$ detection is possible.

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

### 3.3.5 NPOC analysis according to the NPOC plus method

This method is particularly suited for the detection of low TOC contents in samples with high TIC contents or a high level of dissolved $CO_2$. Generally, the NPOC method is recommended for the analysis of such samples. With high and, in particular, unknown TIC contents very long time periods ($t > 10$ min) may, however, be required for the complete purging of the $CO_2$.

As far as the process is concerned, the NPOC plus method is a combination of the NPOC and differential method.

As with the NPOC analysis the sample is acidified with 2 N HCl (pH 2) outside 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.

Parallel to the NPOC plus detection the $TN_b$ detection is possible.

Highly volatile organic substances are purged during the first step and not detected.

### 3.3.6 POC analysis

During the NPOC analysis the total purgeable organic carbon is detected. Together with the volatile organic components parts of inorganic carbon in the form of carbonates and bicarbonates can be purged as $CO_2$ in dependence on the pH value of the sample.

To determine the purgeable inorganic carbon an aliquot is transferred into the POC reactor and the volatile components are purged using a carrier gas. The use of an additional reagent can contribute to improved purging conditions (pH value).

The purged components reach the combustion tube via an adsorber tube (which can e.g. be used for the adsorption of $CO_2$ components) where they are oxidized using a catalyst. The resulting $CO_2$ is detected.

### 3.3.7 $TN_b$-Analysis

Parallel to all analyses via high temperature incineration, the detection of the total bound nitrogen is possible in the $TN_b$ analysis. The thermocatalytic oxidation results in nitrogen oxides which can be detected alternatively using an external chemiluminescence detector (CLD) or an electrochemical detector (ChD, not for multi N/C 3100 pharma).

## 3.4 Catalysts

Various suitable solids can be used as catalysts or oxygen carriers in the multi N/C 3100 supporting the combustion of the components of the sample material in a temperature range of 700 °C to 950 °C.

For the multi N/C 3100 the use of the catalyst "platinum catalyst for multi N/C" with a reaction temperature of 800 °C is recommended. This catalyst has been specially developed and can be used universally over the whole operating range of the analyzer both for the carbon and the nitrogen detection. Because of its very low individual blank value it enables a safe and precise analysis for low carbon and nitrogen contents on the one hand On the other hand it also works stable during the analysis of highly loaded aqueous materials.

Alternatively, the catalyst "special catalyst for multi N/C" ($CeO_2$) can be used with a reaction temperature of 850 °C.

## 3.5 Calibration

### 3.5.1 Calibration strategies

Multiple point calibration with constant sample volume

For most applications a multiple point calibration with **constant sample volume** and **variable concentrations** is carried out.

The concentration series for the ranges to be calibrated are prepared and the settings measured in the selected method. The calibration range should be defined in accordance with the expected sample concentrations.

Multiple point calibration with constant concentration

A multiple point calibration with **variable metering volumes** and **constant concentration** can also be used.

A standard solution for the range to be calibrated is prepared measured for different volumes in accordance with the settings in the selected method.

The calibration should be verified using a second independently prepared standard to preclude the incorrect preparation of the calibration standard.

For measurements in the range of low concentrations (< 10 mg/l) the blank value of the preparation water must be taken into account.

Single point calibration

For low TOC concentrations a single point calibration is permitted for multi N/C 3100 – the blank value of the device is low and the NDIR detector linear.

To minimize sources of error during a single point calibration due to an incorrect standard preparation, the following procedure is recommended:

- Preparation of 3 standards with identical concentration
- Measuring these standards
- Calculation of the calibration curve from the mean of these standards

When using a single point calibration the blank value of the preparation water must be taken into account.

### 3.5.2 Day factor

Via the day factor it is possible to check and correct the calibration with a standard solution. All subsequent measurement results are multiplied by this factor.

The day factor is calculated in accordance with the equation (5).

$$F = \frac{c_{nominal}}{c_{actual}} \quad (5)$$

### 3.5.3 Calibration method in multiWin

Every parameter (procedure) of a method can be calibrated. The parameters of a method to be calibrated can be individually defined. Not all parameters need necessarily be calibrated.

For every parameter up to three calibration functions can be stored in a method. The allocation is automatic.

The calibration function is calculated in relation to the mass m per injected sample. Linear and quadratic calibration functions are calculated in accordance with the equations (6) and (7) through regression calculation.

$$c = (k_1 * I_{net} + k_0)/V \quad (6)$$

$$c = (k_2 * I_{net}^2 + k_1 * I_{net} + k_0)/V \quad (7)$$

c ... target concentration of the standards
V ... sample volumes
$I_{netto}$ ... net integer
$k_0$, $k_1$, $k_2$ ... calibration coefficients

The net integer is the raw integer corrected by the preparation water.

The regression type (linear or quadratic) can be defined by the user. It is possible to select individual measuring points or measured values for the calculation of the current calibration (manual outlier selection). Individual standards can, where required, also be redetected or additional measuring points added to the calibration.

Up to 20 calibration points can be used. For each calibration tenfold detection can be carried out. The calibration function can be calculated from the mean values of the repeated measurements or from all individual detections.

The selection of the suitable calibration method is made by the user.

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

The calibration functions are calculated in accordance with the equations (6) or (7); the following applies:

$$c_{TC} = f(I_{TC}) \quad (8)$$

The calculated parameters appear in the method in the TC analysis channel. The calculation of the analysis results is based on the calculated calibration function.

TIC

The TIC channel is calibrated. The calibration functions are calculated in accordance with the equations (6) or (7); the following applies:

$$c_{TIC} = f(I_{TIC}) \quad (9)$$

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 (Diff)

Generally separate calibration functions are calculated for the channels TC and TIC in accordance with the equations (6) or (7). The equations (8) and (9) apply.

The calculation of the analysis results is based on the calculated calibration functions for TC and TIC. The TOC content is then the result of the equation (10).

$$c_{TOC} = c_{TC} - c_{TIC} \quad (10)$$

The calculated parameters appear in the method in the TIC and TC analysis channels.

The calibration takes place parallel by default, usually with mixed standards (e.g. carbonate/hydrocarbonate and potassium hydrogen phthalate or saccharose).

The calibration of the TIC and TC channel can also be carried out consecutively with separate standards. This is often useful if different ranges are to be calibrated for TC and TIC.

NPOC plus

The method NPOC plus is calibrated the same way as the method TOC(Diff). Before the analysis the TIC must be sufficiently purged for the use of the differential method to be meaningful.

- separate calibration of TIC and TC channel
- Measurement of samples and calculation of the analysis results

  Purging the acidified sample (3 to 5 min)

  Detection of the remaining TIC content – concentration is calculated in accordance with the calibration curve

  Detection of the remaining TC content – concentration is calculated in accordance with the calibration curve

  Calculation of the TOC content in accordance with the equation (10) from the calculated concentration difference.

It is useful to carry out a matrix-dependent calibration. For this the carbonate standard is added in the range of the sample concentration to be expected. This comes closest to the NPOC plus principle.

Calibration $TN_b$

The TN channel is calibrated. The calculated calibration functions correspond to the equations (6) or (7); the following applies:

$$c_{TN} = f(I_{TN}) \quad (11)$$

The calculated parameters appear in the method in the TN analysis channel.

### 3.5.4 Method characteristics

Remaining standard deviation
The remaining standard deviation (remaining variance) expresses the dispersion of the integers around the regression function (regression precision).

Standard deviation of the method
The standard deviation of the method describes in a unique and general way the quality of the calibration. For the unique 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) should be 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, then the correlation coefficient is +1 or -1. For positive correlation coefficients the regression function is increasing, for negative ones it is decreasing.

Coefficient of determination
The square of the correlation coefficient is called the coefficient of determination.

Verification limit
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.

Detection limit
The detection limit of the calibration specifies the lowest concentration for which a verification is possible with a given probability.

Determination limit
The determination limit of the calibration specifies the lowest concentration that can be differentiated quantitatively from the zero point with a given probability.

### 3.5.5 Other calculations

For all measurements where multiple injections are carried out the average value (AV), standard deviation (SD) and 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 multiWin offers the option for an automatic outlier selection. In the method a maximum limit for a variation coefficient or also for a standard deviation can be entered.

The minimum number of measurements agreed in the method will be carried out. If the distribution of the measured values is then above the agreed 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 variation coefficient or standard deviation are calculated for all combinations of measurements. If the variation coefficient or the standard deviation of at least one combination is smaller than the specified maximum variation coefficient or standard deviation, no further measurements are carried out. The combination of measurements with the smallest variation coefficient or the smallest standard deviation is used to calculate the analysis results. The unused measurements are considered as 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.

## 3.6 Blank values

### 3.6.1 Blank water values

Blank preparation water value

Especially for measurements with low TOC concentrations (µg/l range) the TOC content of the water used for preparing the standard can often not be neglected. The weighted-in standard concentration and the TOC blank value of the preparation water are often in the same order of magnitude. This blank value can be taken into account during calibration.

The TOC content of the preparation water is measured separately before the calibration. The mean integer determined for the preparation water is then deducted at calibration from the determined integer of each measuring point (gross integer).

$$I_{net} = I_{gross} - I_{PreparationWater} \quad (12)$$

The calibration function is calculated from the net integers. Mathematically this corresponds to a parallel movement of the calibration line.

The blank value for the preparation water is also considered when determining the daily factor.

Dilution blank value

If the sample needs to be diluted the blank value of the dilution water might also be of interest. This value can be determined separately or entered manually in multiWin. The blank value of the dilution water will then be considered automatically when calculating the concentration of diluted samples.

This value can change over time and must be redetermined before the start of each measuring series. Otherwise the value last entered will be used.

The dilution blank value is always specified in multiWin standardized to 1 ml.

Use of the dilution blank value:

For every measurement the actual dilution water integer ($I_{VdBW}$) is calculated from the dilution blank value in accordance with the sample volume and the dilution ratio used (equation (13)) and deducted from the experimentally determined raw integer (equation (14)). The raw integer determined for each measurement $I_{raw}$ is corrected by the blank value of the dilution water used.

$$I_{DBV} = V_{DBV} * \left(V_{sample} - \frac{NumberUnitsPrimarySample}{NumberUnitsDilution} * V_{sample}\right) \quad (13)$$

$$I_{eff} = I_{raw} - I_{DBV} \quad (14)$$

$V_{DBV}$ ... Dilution blank value
$V_{sample}$ ... sample volume
$I_{eff}$ ... effective integer
$I_{raw}$ ... raw integer
$I_{Dbv}$ ... dilution water integer

Definition of the dilution:

Parts of the primary sample: in the total parts (e. g. 10 parts in 100 parts), i.e. e.g. 10 ml primary sample are diluted with dilution water to a total volume of 100 ml.

For a dilution ratio 1:1 the result is $I_{Dbv} = 0$.

Calculation of the sample concentration:

To calculate the sample concentration c the sample volume and the dilution ratio are used (equation (15)).

$$c = \frac{m}{V_{sample}} * \frac{NumberUnitsDilution}{NumberUnitsPrimarySample} \quad (15)$$

For the linear calibration function (equation (6)) the result is then equation (16).

$$c = \frac{k_1 * I_{eff} + k_0}{V_{sample}} * \frac{NumberUnitsDilution}{NumberUnitsPrimarySample} \quad (16)$$

The integer values determined for a sample can be easily entered. If the primary sample has been diluted and the dilution ratio entered in multiWin, the concentration of the primary sample is specified in the analysis report.

Eluate blank value

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

The eluate blank value is activated in the method and is thus a permanent method parameter. It can be determined separately and entered in the control and analysis software multiWin. This value can change over time and must be redetermined before the start of each measuring series. Otherwise the value last entered will be used.

The eluate blank value is always specified in multiWin standardized to 1ml.

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

If a sample measurement is carried out with a so-called eluate method, the integer of the blank value is deducted from the integer of the sample measurement (dependent on the injection volume) (equation (17)).

$$I_{eff} = I_{raw} - I_{EBV} \quad (17)$$

$I_{eff}$ ... effective integer
$I_{raw}$ ... raw integer
$I_{EBV}$ ... eluate blank value

## 3.6.2 Boat blank value

The boat blank value is determined by introducing an empty boat or a boat with additives for the sample into the combustion furnace and analyzing it.

The boat blank value can be determined separately and entered in the control and analysis software multiWin. This value can change over time and must be redetermined before the start of each measuring series. Otherwise the value last entered will be used.

# 4 First commissioning

## 4.1 Location requirements

### 4.1.1 Setup conditions

The following requirements are placed on the climatic conditions in the operating room of the analyzer:

- Temperature range: +10°C to +35 °C
- Max. humidity: 90 % at 30°C
- Air pressure: 0.7 bar to 1.06 bar

The laboratory atmosphere should be as low as possible in TOC, nitrogen oxide and dust and free of draught, corrosive vapors and vibration. Smoking is prohibited in the operating room of the analyzer!

The following requirements are placed on the location of the analyzer:

- Do not locate the analyzer directly near a door or window.
- Place the analyzer on a heat-resistant and acid-resistant surface.
- Do not locate the analyzer near sources of electromagnetic interference.
- Avoid direct sunlight and radiation from heaters onto the analyzer; if necessary ensure air-conditioning.
- Never obstruct the front doors, the left side wall and the ventilation slots of the analyzer with other equipment or furnishings!
- Keep a safety distance of at least 5 cm from the back and the right side of the equipment to other equipment or walls!

### 4.1.2 Required space

**Attention**

The AS vario autosampler, the POC and EPA sampler must be positioned on the left of the analyzer. The external solids module or the chemiluminescence detector (CLD) must be located to the right of the analyzer. The AS 21 and AS 10 samplers are attached to the right-hand side of the analyzer.

The layout of the other components can be adapted to the local conditions.

The space required is a function of all components needed for the measurement. Leave adequate space for the PC, monitor, printer and any add-on equipment.

### 4.1.3 Energy supply

**WARNING**

The analyzer multi N/C 3100 must only be connected to a properly grounded mains outlet in accordance with the voltage specifications on the type plate!

The multi N/C 3100 is operated from the single phase alternating current mains.

The installation of the electrical equipment of the laboratory must comply with the standard DIN VDE 0100. At the connection point an electrical current in accordance with the standard IEC 38 must be available.

### 4.1.4 Gas supply

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

The connection hose with outer diameter 6 mm and inner diameter 4 mm is included with the delivery. The length is 3 m. If other lengths are preferred, please contact the customer service department at Analytik Jena GmbH.

## 4.2 Unpacking and placing the analyzer

**Attention**

The analyzer multi N/C 3100 must only be set up, assembled and installed by the customer service department of Analytik Jena GmbH or trained specialist personnel authorized by Analytik Jena GmbH!

Any unauthorized intervention in the analyzer can endanger the user and the operational safety of the equipment and limits or completely invalidates any warranty claims.

Retain the transport packaging! Return transport for service must be in the original packaging. This alone prevents transport damage.

The analyzer multi N/C 3100 is unpacked and assembled by the customer service department of Analytik or its authorized and trained specialist personnel.

Please check when unpacking the device for completeness and soundness of the delivery in accordance with the packing list included.

After assembly, the customer service tests the analyzer and documents the test.

# 5 Connecting add-on devices

**Attention**

Before connecting add-on devices switch off the analyzer. Always connect the add-on devices to the multi N/C 3100 when it is switched off!

## 5.1 Sampler

### 5.1.1 AS vario / AS vario (ER) autosampler

**CAUTION**

Make sure that no liquid reaches the cable connections or the inside of the equipment or the power supply. There is a danger of electric shock.

Caution in the movement range of the sampler arm and the cannula during operation. There is a risk of injury (crushing etc.)!

**Attention**

Before commissioning the autosampler remove the transport lock. Do not obstruct the sampler during running operation. In both cases the drives might be damaged.

Six different sample trays are available for the AS vario autosampler – four trays for the AS vario (ER). A matching cannula holder is available for each sample tray. Before the sample supply the cannulas can be rinsed from the inside with the sample or ultra-pure water.

Sample tray

| | | | | | | |
|---|---|---|---|---|---|---|
| Max. no. of samples | 20 | 47 (ER) | 52 | 72 (ER) | 100 (ER) | 146 (ER) |
| Sample tubes | 100 ml | 12 and 50 ml | 100 ml | 40 and 50 ml | 20 ml | 12 ml |

Technical data

| | |
|---|---|
| Operating voltage | 24 V DC via external power supply |
| Power consumption | 50 W |
| Grid voltage of external power supply | 100 – 240 V, 50 – 60 Hz auto-sensing |
| Dimensions (WxDxH) | 350 mm x 400 mm x 470 mm |

The autosampler AS vario is positioned on the left of the analyzer. It is loaded with 1 to 2 cannulas.

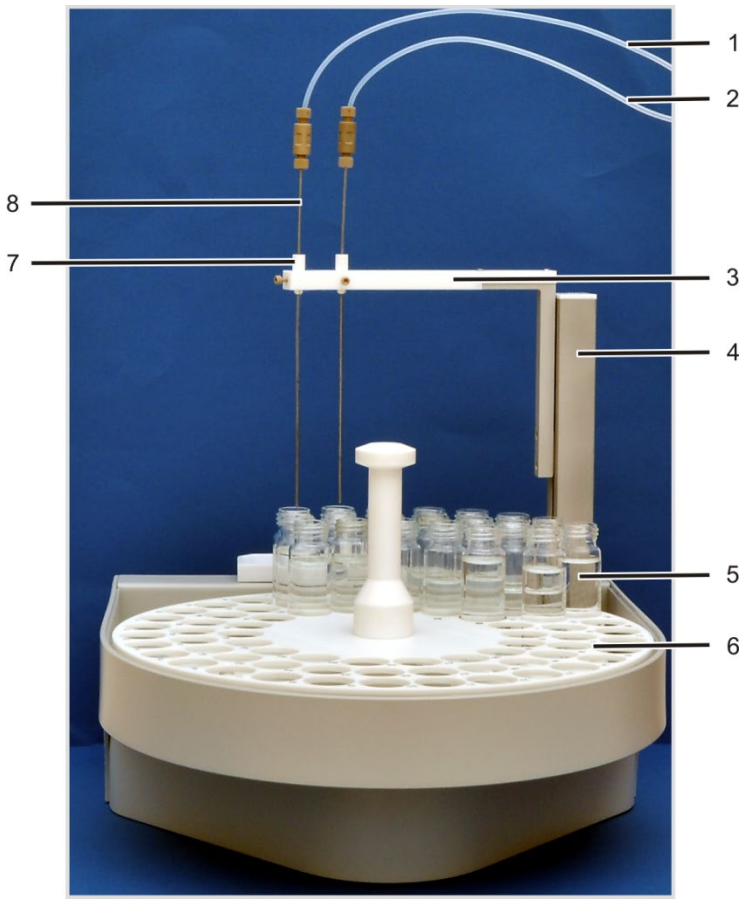



**Fig. 19 Layout of the AS vario autosampler**

1 Connection hose to the analyzer (purging hose for NPOC measurements)
2 Connection hose to the analyzer (sample aspiration hose)
3 Cannula holder
4 Autosampler arm
5 Sample tubes
6 Sample tray
7 Sleeve
8 Cannula

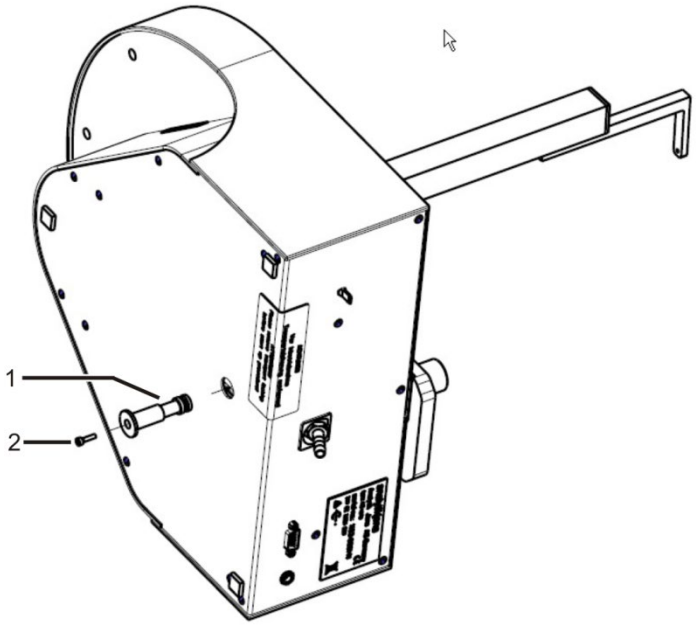
The model AS vario ER is equipped with a bloc for extra needle rinsing. The cannulas are rinsed from the outside with ultra-pure water. It is suitable for all measuring methods, in particular for NPOC analysis with parallel purging. During the installation of the autosampler the ultra-pure water supply must also be connected. If you want to use another sample tray with the AS vario ER the block with the rinsing cups has to be removed.


1 Cannula for sample aspiration
2 Cannula holder (with No. 72)
3 Sample tray for 72 samples
4 Ultra-pure water supply
5 Block with rinsing cups
6 Cannula for purging (NPOC analysis)

**Fig. 20 Layout of the AS vario ER autosampler**

Removing the transport lock

The autosampler AS vario has been fitted with a transport safety lock on the bottom of the autosampler. Keep the transport lock for a later transport.




1 Transport lock
2 Screw M3x12

**Fig. 21 Transport lock on autosampler AS vario ER**

1. Place the autosampler on the side as shown in Fig. 21.
2. Unscrew the screw (2 in Fig. 21) with the Allen wrench (included in the scope of delivery) and remove the red transport lock.
3. For commissioning replace the autosampler on the baseplate.

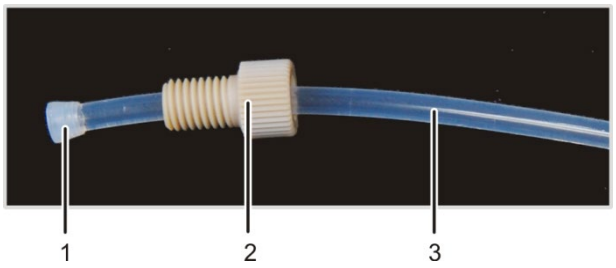
Commissioning the autosampler

4. Switch off the analyzer!
5. Plug cable on the low voltage side of the table power supply included in the delivery into the connection on the rear of the autosampler. Do not yet connect the power supply to the grid.
6. Plug the grounding conductor into the connection on the rear of the analyzer.

7. Connect the autosampler to the analyzer with the interface cable (port on the rear of the autosampler and "sampler" port on the rear of the analyzer).
8. Attach the outlet tube to the outlet connector on the rear of the autosampler. Insert the other end of the tube into the opening in the cover of the waste bottle. ATTENTION! Position the outlet tube at a constant incline. If necessary shorten the tube. The tube must not dip in the liquid.
9. Place the sample tray onto the autosampler housing. Make sure it clicks into place.
10. Check that the correct cannula holder has been installed at the autosampler arm. To do so, compare the number engraved on the bottom with the max. number of sample tubes. They have to be identical.
11. Insert the cannulas with the matching sleeve into the cannula holder.
    - For NPOC measurements with parallel purging: Insert 1 cannula with sleeve into each of the two cannula holder positions (see Fig. 19).
    - For NPOC measurements with non-parallel purging: Insert both cannulas in one sleeve with two holes in the position on the right (see Fig. 22, not suitable for the AS vario ER)

**Fig. 22 Sleeve with 2 cannulas for NPOC measurements (non-parallel purging)**

12. Adjust the cannulas so that approx. one third of the cannulas is visible above the sleeve. Secure the cannulas by slightly tightening the screw.
13. Connect the two connection hoses to the analyzer to the cannulas:
    Hose no. AA = sample aspiration hose
    Hose no. 7 = purging hose for NPOC measurements
    - Release the upper Fingertight connection of the cannula.
    - Guide the hose through the banjo bolt.
    - Slide the conical nipple with the conical side towards the banjo bolt onto the hose. The conical nipple and hose must be flush.
    - Retighten the Fingertight connection.




1 Conical nipple
2 Banjo bolt
3 Hose

**Fig. 23 Hose in Fingertight connection**

14. Attach the sample cover (if present) so that it is positioned in the guide rail.
15. Connect the power supply to the grid.

16. Check the configuration in the multiWin program via the INSTRUMENT ▸ SYSTEM INFORMATION menu command in the SET-UP INFO window. If necessary, modify the configuration:
    - Exit multiWin.
    - In the Windows user interface, start the SET-UP TOOL under START ▸ PROGRAM FILES ▸ MULTIWIN ▸ MULTIWIN SET-UP TOOL.
    - Select the sampler type in the SAMPLER list.
    - Exit the SET-UP TOOL with [CREATE].
    - Start the multiWin program and select the CONFIGURATION ▸ EDIT OPTIONS menu command to open the OPTIONS window on the ANALYZER COMPONENTS tab. In the SAMPLER group, select the correct tray and tube size. Exit the OPTIONS window with [OK].

Installing the needle rinsing at AS vario ER


1 Ultra-pure water connection
2 Waste connection
3 Removable block with rinsing cups

**Fig. 24 Needle rinsing at autosampler AS vario ER**

1. Screw the connector of the water tubing in connection (1). Immerse the other end of the tube in the ultra-pure water bottle.
2. Attach the waste tube to the waste connector (2). Insert the other end of the tube into the opening in the lid of the waste bottle.
   ATTENTION! Take care about a sufficient decline of the waste tube. If necessary shorten the tube. The tube must not dip in the liquid.

Before the first start the sampler must be adjusted (see "Adjusting the AS vario (ER) autosampler" section, p. 88).

Activating the needle rinsing in multiWin

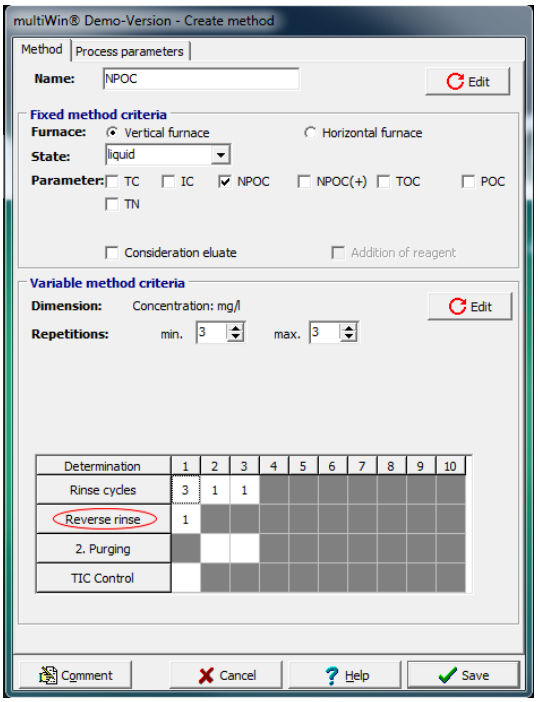



Fig. 25 Window Create method

1. Create a new method.
2. Activate the option REVERSE RINSE in the method parameters (VARIABLE METHOD CRITERIA).

   Determine the number of rinsing cycles by entering a number ≥ 1. Note that one rinsing cycle is usually sufficient.

## 5.1.2 AS 21 autosampler

### CAUTION

Make sure that no liquid reaches the cable connections or the inside of the equipment or the power supply. There is a danger of electric shock.

Caution near the movement area of the sampler arm and the cannula during operation. There is a risk of injury (crushing etc.)!

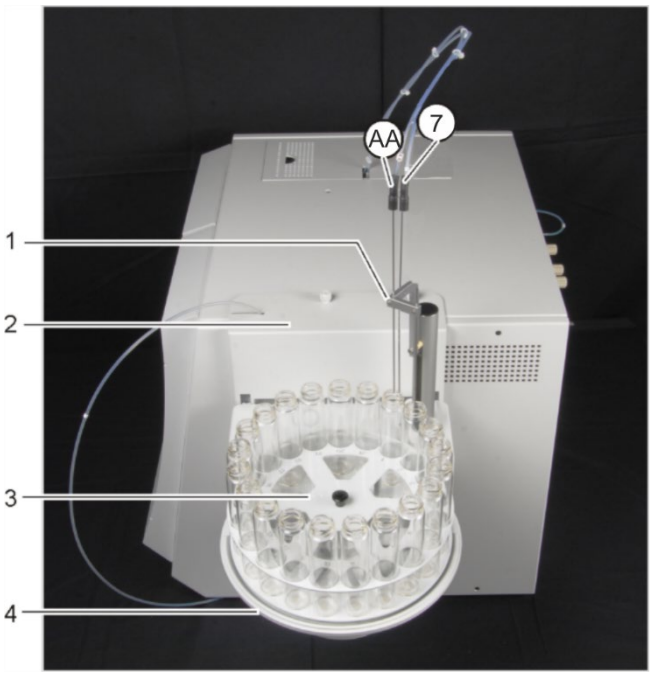
### Attention

Only adjust the sampler in the switched-off state. Do not obstruct the sampler during running operation. The drives might be damaged.

Technical data

| | |
|---|---|
| Number of samples | max. 21 |
| Sample cups | 50 ml |
| Operating voltage | 24 V DC via external power supply |
| Power consumption | 30 W |
| Dimensions (WxDxH) | 260 mm x 350 mm x 310 mm |
| Mains voltage of external power supply | 100 – 240 V, 50 – 60 Hz auto-sensing |

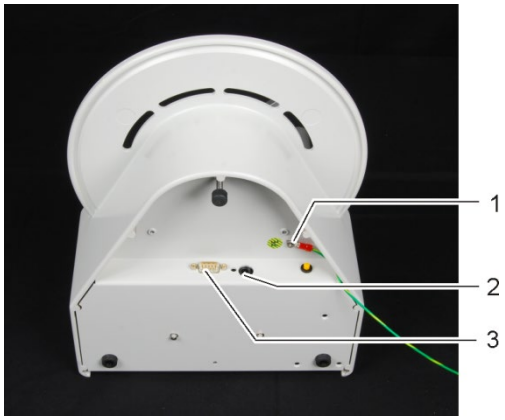
The sampler is installed on the right side of the analyzer in a holder. The sampler can be populated with 2 cannulas. The design of the cannula holder may vary.




1 sampler arm with cannula holder
2 holder at the analyzer
3 Sample tray
4 drive unit

hose no. AA
sample intake hose
hose no. 7
purging cannula

**Fig. 26 Layout of the sampler AS 21**




1 grounding conductor
2 switching power supply connection
3 RS 232 interface – analyzer connection

**Fig. 27 Connections at the bottom of the sampler**

Installation at the analyzer

1. Screw the holder to the right-hand side of the analyzer.
2. Place drive onto the holder.
3. Plug the grounding conductor into the connection on the rear of the analyzer (10 in Fig. 17 p. 28).
4. Plug cable on the low voltage side of the table power supply included in the delivery into the connection at the bottom of the sampler. Do not yet connect the power supply to the mains.
5. Plug the interface cable into the bottom of the equipment. Connect the sampler to the interface at the rear of the analyzer (12 in Fig. 17 p. 28).
6. Place a sample cup into position 1 of the sample tray under the two cannulas.
7. Insert the cannulas into the autosampler arm. Manually adjust the height of the cannulas so that the cannula tips protrude 1 – 2 cm over the tube edge and cannot touch the tubes when the autosampler arm is moving.
8. Secure the cannulas by tightening the screws lightly.

ATTENTION! The screws must not bend the cannulas under any circumstances.

9. Connect the switching power supply to the mains. Switch on the switching power supply.
10. Check the configuration via the menu command INSTRUMENT ▸ SYSTEM INFORMATION in the window SET-UP INFO. If necessary, modify the configuration:
    - Exit the program multiWin.
    - On the windows user interface start the set-up tool under START ▸ PROGRAM FILES ▸ MULTIWIN ▸ MULTIWIN SET-UP TOOL.
    - In the list SAMPLER select the sampler type.
    - Exit the SET-UP TOOL with [CREATE].
    - Start the multiWin program and select the CONFIGURATION ▸ EDIT OPTIONS menu command to open the OPTIONS window on the ANALYZER COMPONENTS tab. In the SAMPLER group, select the correct tray and tube size. Exit the OPTIONS window with [OK].
11. Adjust the immersion depth of the cannulas in the tube (z direction) in multiWin.
    - Select the INSTRUMENT ▸ SAMPLER ALIGNMENT menu command to open the window with the same name.
    - In the "Please select position needing adjustment" group, select POSITION 1.
    - Click [POSITION 1 ADJUST].
      The autosampler arm now lowers the cannulas into the tube in position 1.
    - If necessary, increase or decrease the z values. Click [POSITION 1 ADJUST] again after each change to verify the change.
    - Once adjustment is complete, click [SAVE] to close the window.
12. Position the hoses in the clip and attach the clip to the housing of the multi N/C using one of the screws of the autosampler.

    ATTENTION! The hoses may not hinder the movement of the autosampler arm.

Retrofit to the function "parallel purging"

By mounting a special cannula holder to the autosampler arm the autosampler can be retrofitted for the "parallel purging" function.


**Fig. 28 Parallel purging**

1 Aspirating cannula
2 Purging cannula
3 Fixing screw on the spacer
4 Spacer
5 Clamping of the aspirating cannula
6 Clamping of the purging cannula
7 Cannula holder for "parallel purging"
8 Screw connection of the cannula holder
9 Clip for the attachment of the hoses

1. Screw the cannula holder (7) to the autosampler arm.
2. Push the spacer (4) onto the two cannulas. Lightly secure the spacer below the hose connections with the fixing screw, so that it cannot move.
3. Place two sample cups into positions 1 and 2 of the sample tray under the two cannulas.
4. Insert the cannulas into the cannula holder according to the figure and attach them only lightly using the knurled head screws. Manually adjust the height of the cannulas so that the cannula tips protrude 1 – 2 cm over the tube edge and cannot touch the tubes when the autosampler arm is moving.
5. Secure the cannulas by tightening the screws lightly.

   ATTENTION! The screws must not bend the cannulas under any circumstances.
6. Adjust the immersion depth of the cannulas in the tube (z direction) in multiWin.
   - Select the INSTRUMENT ▸ SAMPLER ALIGNMENT menu command to open the window with the same name.
   - In the "Please select position needing adjustment" group, select POSITION 1.
   - Click [POSITION 1 ADJUST].
     The autosampler arm now lowers the cannulas into the tube in position 1.
   - If necessary, increase or decrease the z values. Click [POSITION 1 ADJUST] again after each change to verify the change.
   - Once adjustment is complete, click [SAVE] to close the window.

7. Position the hoses in the clip (9) and attach the clip to the housing of the multi N/C using one of the screws of the autosampler.

   ATTENTION! The hoses may not hinder the movement of the autosampler arm.

### 5.1.3 AS 10 autosampler

**CAUTION**

Make sure that no liquid reaches the cable connections or the inside of the equipment or the power supply. There is a danger of electric shock.

Caution in the movement range of the sampler arm and the cannula during operation. There is a risk of injury (crushing etc.)!

**Attention**

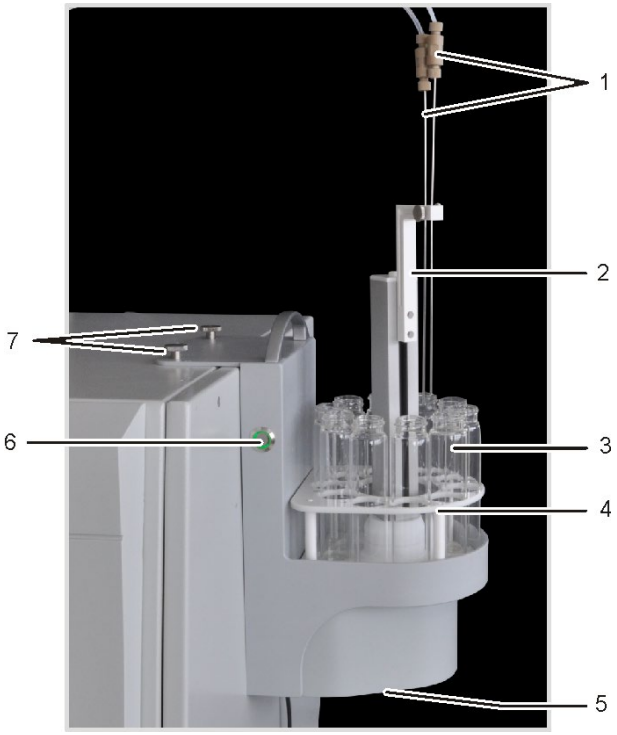
Do not obstruct the sampler during running operation. The drives might be damaged.

Technical data

| | |
|---|---|
| Number of samples | max. 10 |
| Sample tubes | 50 ml |
| Operating voltage | 24 V DC via external power supply |
| Power consumption | 30 W |
| Grid voltage of external power supply | 100 – 240 V, 50 – 60 Hz auto-sensing |
| Dimensions (WxDxH) | 160 mm x 130 mm x 300 mm |

The autosampler is installed on the right side of the analyzer in a holder. It can be loaded with 2 cannulas.

Layout




1 Cannulas
2 Autosampler arm
3 Sample tubes
4 Sample tray
5 Grid connection and connection to the analyzer (hidden)
6 On/Off switch
7 Fastening screws

**Fig. 29 Layout of the AS 10 autosampler**

Installation at the analyzer

1. Plug cable on the low voltage side of the table power supply included in the delivery into the connection at the bottom of the autosampler and connect the power supply to the grid.
   Make sure the AS 10 is switched off. (The green LED of the On/Off must be off.)
   Connect the autosampler to the analyzer with the interface cable (port on the bottom of the autosampler and "sampler" port on the rear of the analyzer).
2. Screw the sampler with the two fastening screws to the right-hand side of the equipment.
3. Attach the autosampler hoses to the top of the analyzer with the provided knurled screw and the tube holder. Ensure that are not subjected to tensile stress.

4. Place a sample tube into position 1 of the sample tray.
5. Insert the cannulas into the autosampler arm. Manually adjust the height of the cannulas so that the cannula tips protrude 1 – 2 cm over the tube edge and cannot touch the tubes when the autosampler arm is moving.
6. Secure the cannulas by slightly tightening the screw.
   ATTENTION! The screw must not bend the cannula under any circumstances.
7. Switch on the AS 10 at the On/Off switch.
8. Check the configuration in the multiWin program via the INSTRUMENT ▸ SYSTEM INFORMATION menu command in the SET-UP INFO window. If necessary, modify the configuration:
   - Exit multiWin.
   - In the Windows user interface, start the SET-UP TOOL under START ▸ PROGRAM FILES ▸ MULTIWIN ▸ MULTIWIN SET-UP TOOL.
   - Select the sampler type in the SAMPLER list.
   - Exit the SET-UP TOOL with [CREATE].
   - Start the multiWin program and select the CONFIGURATION ▸ EDIT OPTIONS menu command to open the OPTIONS window on the ANALYZER COMPONENTS tab. In the SAMPLER group, select the correct tray and tube size. Exit the OPTIONS window with [OK].
9. Adjust the immersion depth of the cannulas in the tube (z direction) in multiWin.
   - Select the INSTRUMENT ▸ SAMPLER ALIGNMENT menu command to open the window with the same name.
   - In the "Please select position needing adjustment" group, select POSITION 1.
   - Click [POSITION 1 ADJUST].
     The autosampler arm now lowers the cannulas into the tube in position 1.
   - If necessary, increase or decrease the z values. Click [POSITION 1 ADJUST] again after each change to verify the change.
   - Once adjustment is complete, click [SAVE] to close the window.

### 5.1.4 POC sampler

**CAUTION**

Always disconnect the power plug before opening the device!

Make sure that no liquid reaches the cable connections or the inside of the equipment or the power supply. There is a danger of electric shock.

Caution in the movement range of the sampler arm and the cannula during operation. There is a risk of injury (crushing etc.)!

Pay attention to the movement range of the autosampler arm when setting up the device. Make sure that there is also sufficient space behind the device.

**Attention**

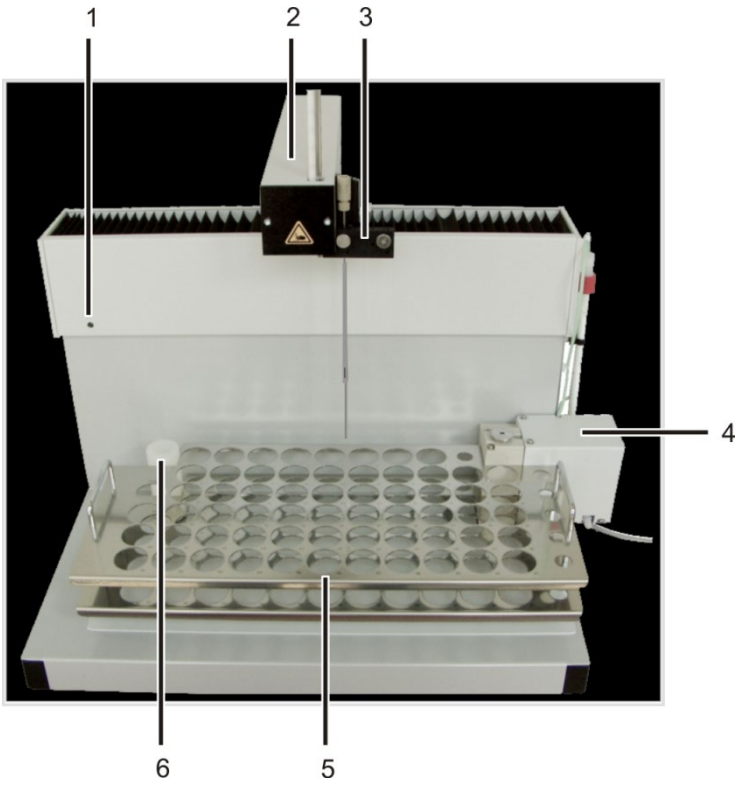
Do not obstruct the sampler during running operation. The drives might be damaged.

The POC sampler is a special autosampler for POC measurements. It is used in combination with the POC module for automatic operation (see "POC module" section, p. 62). It has a piercing function for sample tubes with septum caps.

Technical data

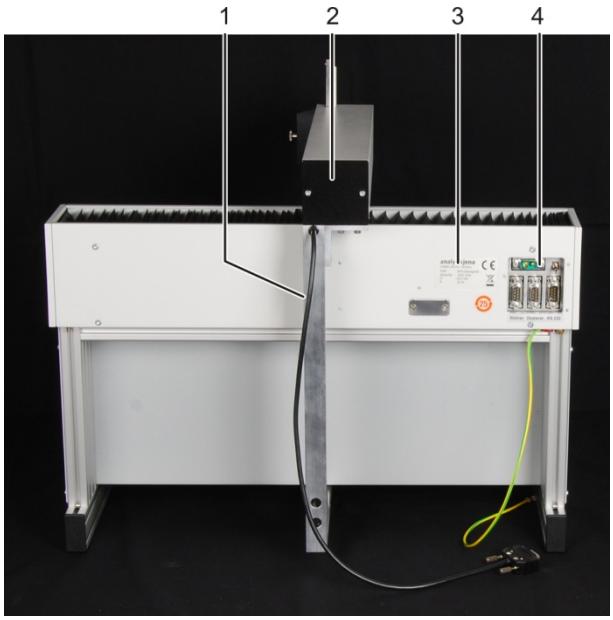
| | |
|---|---|
| Number of samples | max. 61 |
| Sample tubes | 40 ml |
| Operating voltage | 24 V DC via external power supply |
| Power consumption | 30 W |
| Grid voltage of external power supply | 100 – 240 V, 50 – 60 Hz auto-sensing |
| Dimensions (WxDxH) | 500 mm x 550 mm x 470 mm |

The POC sampler is positioned on the left-hand side of the analyzer and loaded with 1 special cannula.



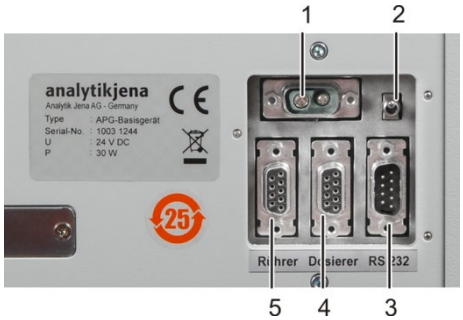

1 Control LED
2 Autosampler arm
3 Cannula holder with cannula
4 POC module
5 Sample tray
6 Wash cup

**Fig. 30 POC sampler with POC module**




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

**Fig. 31 Rear of the POC sampler**

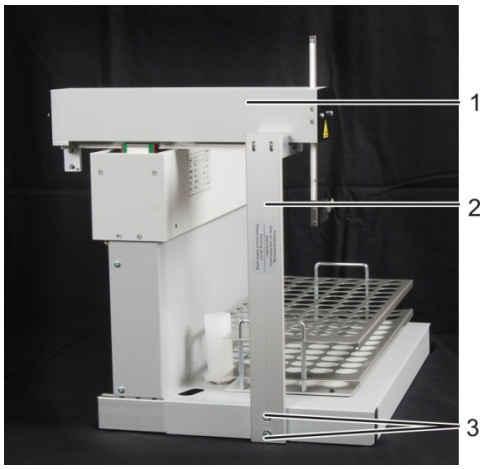



1 Connection to power supply unit
2 Equipment switch
3 Connection to the analyzer
4 Not used
5 Stirrer connection

**Fig. 32 Electrical connections on the rear of the POC sampler**

Setting up POC sampler

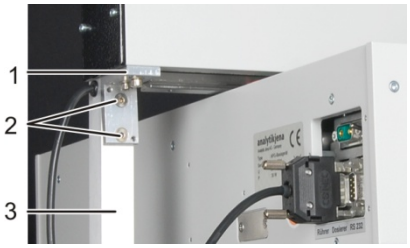
1. Remove the transport lock!
   - Remove the two countersunk screws with the A/F3 hexagon head wrench supplied.
   - Remove the complete transport retaining clip and retain the transport lock well (for transport in case of a service requirement etc.).




1 Autosampler arm
2 Transport retaining clip
3 Screws

**Fig. 33 Transport lock**

2. Fit the stirring arm.
   - Fit the arm to the bracket at the rear end of the sampler arm.
   - Screw on the arm with the countersunk screws supplied (DIN 7991-M4x10) using the A/F2.5 hexagon head wrench.
   - Tighten the screws evenly to allow the arm to be aligned.
   - Connect the stirrer cable to the "Stirrer" port on the rear of the autosampler.




1 Bracket at the autosampler arm
2 Countersunk screws
3 Stirring arm

**Fig. 34 Fitting the stirring arm to the autosampler**

3. Place the autosampler to the left of the analyzer.
4. Connect the table power supply cable on the low voltage side to the rear of the autosampler. Do not connect the power supply to the grid yet.
5. Connect the data cable supplied to the "Sampler" port on the rear of the analyzer.
6. Connect the other end of the data cable to the RS 232 port of the autosampler.
7. Connect the waste hose supplied to the wash cup of the autosampler and to a suitable waste container or drain.
8. Fit the wash cup with waste hose to the autosampler.
9. Place the sample tray onto the space provided.
   Note the positioning of the tray. The label has to be legible if you face the front of the device. The two centering pins (black plastic) on the contact surface of the autosampler have to protrude into the drill holes in the tray floor.

10. Connect the power supply to the grid.
11. Check the configuration in the multiWin program via the INSTRUMENT ▸ SYSTEM INFORMATION menu command in the SET-UP INFO window. If necessary, modify the configuration:
    - Exit multiWin.
    - In the Windows user interface, start the SET-UP TOOL under START ▸ PROGRAM FILES ▸ MULTIWIN ▸ MULTIWIN SET-UP TOOL.
    - Select the sampler type in the SAMPLER list.
    - Exit the SET-UP TOOL with [CREATE].
    - Start the multiWin program and select the CONFIGURATION ▸ EDIT OPTIONS menu command to open the OPTIONS window on the ANALYZER COMPONENTS tab. In the SAMPLER group, select the correct tray and tube size. Exit the OPTIONS window with [OK].

For commissioning the POC sampler with POC module and special cannula, see "POC module" section, p. 62.

### 5.1.5 EPA sampler

**CAUTION**

Always disconnect the power plug before opening the device!

Make sure that no liquid reaches the cable connections or the inside of the equipment or the power supply. There is a danger of electric shock.

Caution in the movement range of the sampler arm and the cannula during operation. There is a risk of injury (crushing etc.)!

Pay attention to the movement range of the autosampler arm when setting up the device. Make sure that there is also sufficient space behind the device.

**Attention**

Do not obstruct the sampler during running operation. The drives might be damaged.

The EPA sampler is a special autosampler with piercing function for sample tubes with septum caps.

Technical data

| | |
|---|---|
| Number of samples | max. 64 |
| Sample tubes | 40 ml |
| Operating voltage | 24 V DC via external power supply |
| Power consumption | 30 W |
| Grid voltage of external power supply | 100 – 240 V, 50 – 60 Hz auto-sensing |
| Dimensions (WxDxH) | 500 mm x 550 mm x 470 mm |

The EPA sampler is positioned on the left of the analyzer. It is loaded with 1 or 2 special cannulas (with ventilation slot).

Layout

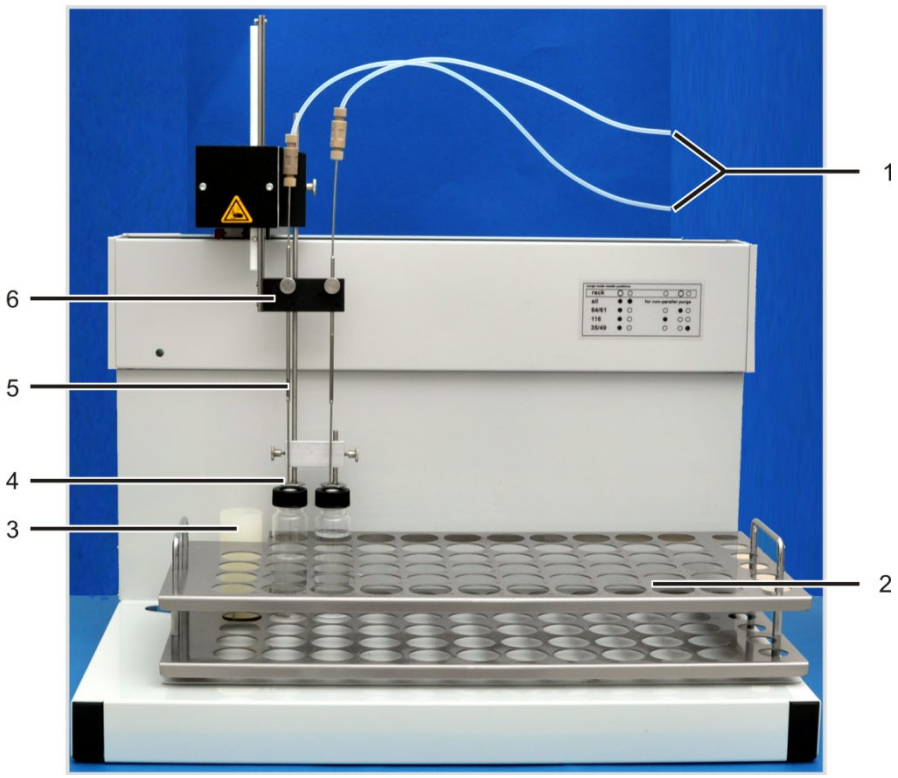
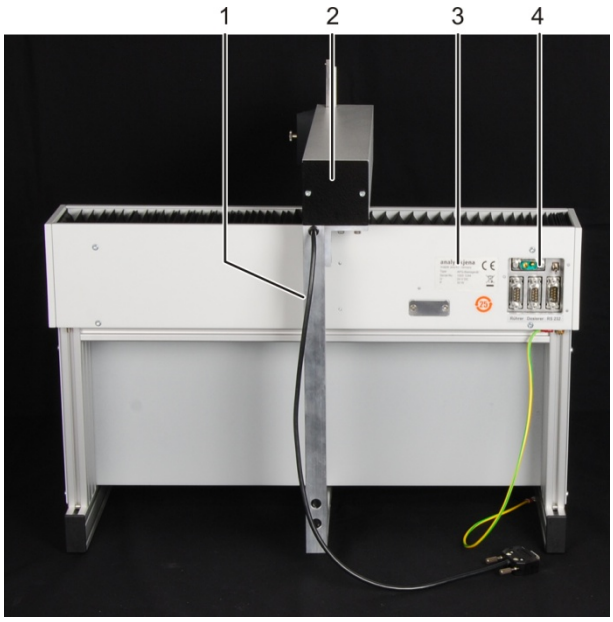


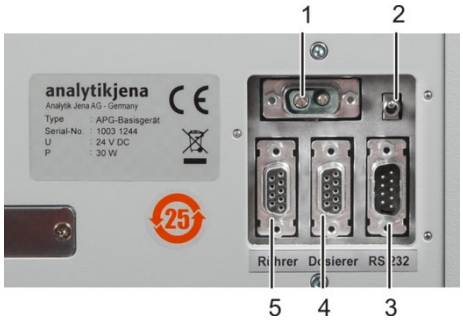

**Fig. 35 EPA sampler**

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




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

**Fig. 36 Rear of the EPA sampler**

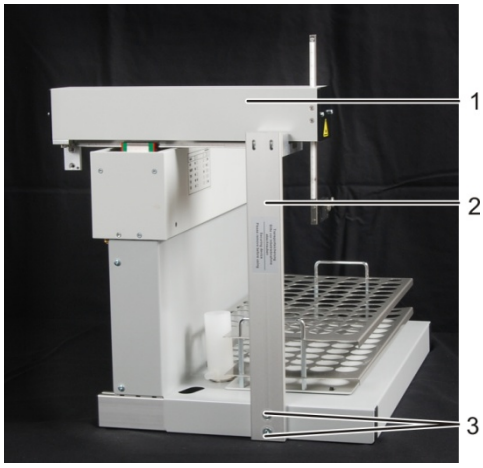



1 Connection to power supply unit
2 Equipment switch
3 Connection to the analyzer
4 Not used
5 Stirrer connection

**Fig. 37 Electrical connections on the rear of the EPA sampler**

Setting up EPA sampler

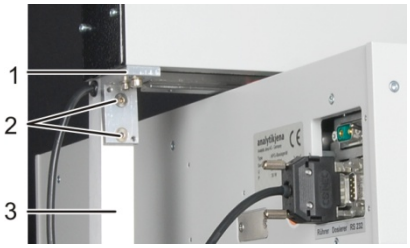
1. Remove the transport lock!
   - Remove the two countersunk screws with the A/F3 hexagon head wrench supplied.
   - Remove the complete transport retaining clip and retain the transport lock well (for transport in case of a service requirement etc.).




1 Autosampler arm
2 Transport retaining clip
3 Screws

**Fig. 38 Transport lock**

2. Fit the stirring arm.
   - Fit the arm to the bracket at the rear end of the sampler arm.
   - Screw on the arm with the countersunk screws supplied (DIN 7991-M4x10) using the A/F2.5 hexagon head wrench.
   - Tighten the screws evenly to allow the arm to be aligned.
   - Connect the stirrer cable to the "Stirrer" port on the rear of the autosampler.

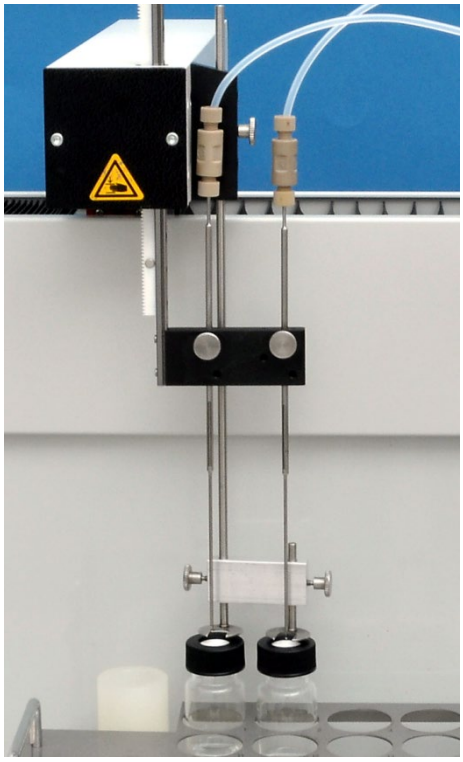



1 Bracket at the autosampler arm
2 Countersunk screws
3 Stirring arm

**Fig. 39 Fitting the stirring arm to the autosampler**

3. Place the sampler to the left of the analyzer.
4. Connect the table power supply cable on the low voltage side to the rear of the autosampler. Do not connect the power supply to the grid yet.
5. Connect the data cable supplied to the "Sampler" port on the rear of the analyzer.

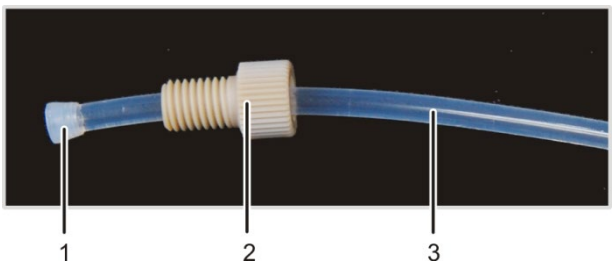
6. Connect the other end of the data cable to the RS 232 port of the autosampler.
7. Connect the waste hose supplied to the wash cup of the autosampler and to a suitable waste container or drain.
8. Fit the wash cup to the autosampler.
9. Place the sample tray onto the space provided.
   Note the positioning of the tray. The label has to be legible if you face the front of the device. The two centering pins (black plastic) on the contact surface of the autosampler have to protrude into the drill holes in the tray floor.
10. Insert piercing cannulas and holding-down clamps into the autosampler arm.



NPOC measurements with parallel purging:
Insert 1 cannula into each of the two positions in the cannula holder

NPOC measurements with non-parallel purging:
Insert both cannulas into the left position in the cannula holder

11. Clamp the two cannulas high enough in the holder to prevent them dipping into the tubes (basic position).
12. Connect the two connection hoses to the analyzer to the cannulas:
    Hose no. AA = sample aspiration hose
    Hose no. 7 = purging hose for NPOC measurements
    - Release the upper Fingertight connection of the cannula.
    - Guide the hose through the banjo bolt.
    - Slide the conical nipple with the conical side towards the banjo bolt onto the hose. The conical nipple and hose must be flush.
    - Retighten the Fingertight connection.




1 Conical nipple
2 Banjo bolt
3 Hose

**Fig. 40 Hose in Fingertight connection**

13. Connect the power supply to the grid.
14. Check the configuration in the multiWin program via the INSTRUMENT ▸ SYSTEM INFORMATION menu command in the SET-UP INFO window. If necessary, modify the configuration:
    - Exit multiWin.
    - In the Windows user interface, start the SET-UP TOOL under START ▸ PROGRAM FILES ▸ MULTIWIN ▸ MULTIWIN SET-UP TOOL.
    - Select the sampler type in the SAMPLER list.
    - Exit the SET-UP TOOL with [CREATE].
    - Start the multiWin program and select the CONFIGURATION ▸ EDIT OPTIONS menu command to open the OPTIONS window on the ANALYZER COMPONENTS tab. In the SAMPLER group, select the correct tray and tube size. Exit the OPTIONS window with [OK].

Before the first start the sampler must be adjusted (see "Adjusting the EPA sampler" section, p. 94).

## 5.2 Chemiluminescence detector (CLD)

### Attention

Before connecting add-on devices switch off the analyzer. Always connect the add-on devices to the multi N/C 3100 when it is switched off!

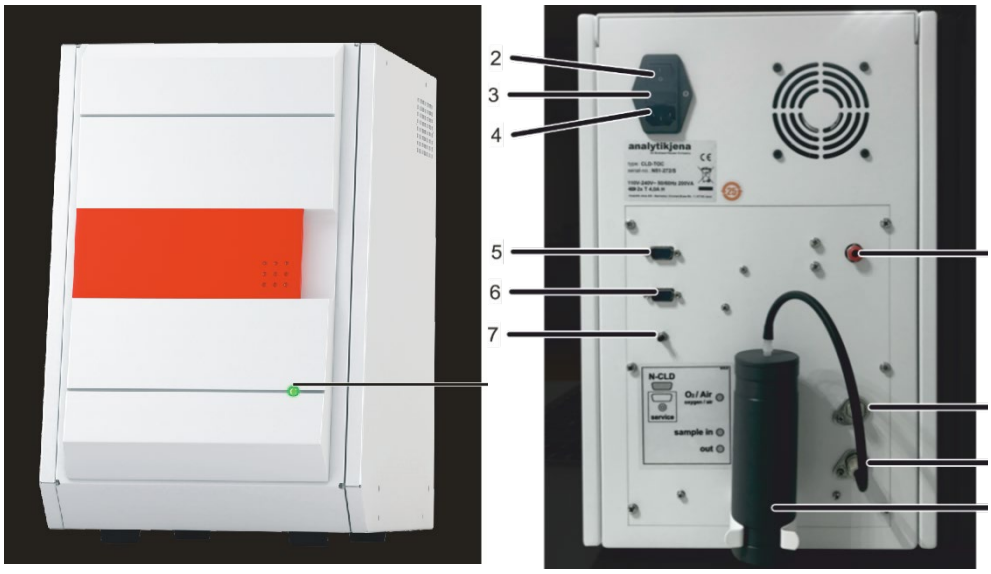



**Fig. 41 CLD – display elements, mains connection and media connections**

1 LED in the front panel
2 Mains switch
3 Fuse holder
4 Mains connection
5 Serial connection to the analyzer
6 Service interface
7 Programming button (for service only)
8 Connection $O_2$, synthetic/purified air
9 Connection to the analyzer "sample in"
10 Gas outlet "out"
11 Adsorption tube (for the removal of $NO_x$)

Technical data

| | |
|---|---|
| Detection principle | Chemiluminescence detector |
| Parameter | $TN_b$ (total bound nitrogen) |
| Measuring range | 0 – 200 mg/l $TN_b$ |
| Detection limit | 0.05 mg/l $TN_b$ |
| Analysis time | 3 - 5 min |
| Gas for ozone generation | Sample gas supply same as multi N/C analyzer, 60 ml/min, 4 – 6 bar |
| Dimensions W x H x D | approx. 300 mm x 460 mm x 550 mm |
| Weight | approx. 12 kg |
| Connection | 110 – 240 V AC 50/60 Hz |
| Protection | 2 x T4.0 A H |
| Typical average power consumption | 200 VA |
| PC interface | RS 232 |
| Interference suppression (electromagnetic compatibility) | in accordance with the rules of EN 55011 Group 1, Class B interference-protected (according to EN 61326-1 suitable for use in basic electromagnetic environments) |

**CAUTION**

The ozone gas ($O_3$) generated from dry carrier gas in the ozone generator is destroyed in the downstream ozone destroyer if the analyzer is used correctly. Furthermore, any potential concentration is harmless: Various safety measures cause the ozone generator to automatically shut down.

If a smell of ozone occurs in the CLD, shut down the device immediately and notify the Service department of Analytik Jena GmbH.

Installation at the analyzer

Connect the chemiluminescence detector to the analyzer as follows:

1. Place the chemiluminescence detector to the right of the analyzer.
2. Connect the connection hose for carrier gas at the gas connection on the equipment backplate (6 in Fig. 41 p. 60).
3. Make the gas connection between CLD and analyzer:
   - CLD-connection 7 in Fig. 41)
   - Analyzer connection 6 in Fig. 17 p. 28.
4. Connect the corresponding serial interface "CLD/HT" on the equipment backplate of the analyzer (13 in Fig. 17) via the serial data cable supplied to the RS 232 interface of the chemiluminescence detector.
5. Switch on the CLD.
   ✓ The lamp on the front panel of the CLD indicates readiness for operation.
6. Check the configuration via the menu command INSTRUMENT ▸ SYSTEM INFORMATION in the window SET-UP INFO. If necessary, modify the configuration:
   - Exit the program multiWin.
   - On the windows user interface start under START ▸ PROGRAM FILES ▸ MULTIWIN ▸ MULTIWIN SET-UP TOOL the SET-UP TOOL.
   - In the DETECTOR list select the option CLD.
   - Exit the SET-UP TOOL with [CREATE].
   - Start the multiWin program and select the CONFIGURATION ▸ EDIT OPTIONS menu command to open the OPTIONS window on the ANALYZER COMPONENTS tab. In the SENSORS group, activate N-measurement. Exit the OPTIONS window with [OK].

## 5.3 External solids module HT 1300

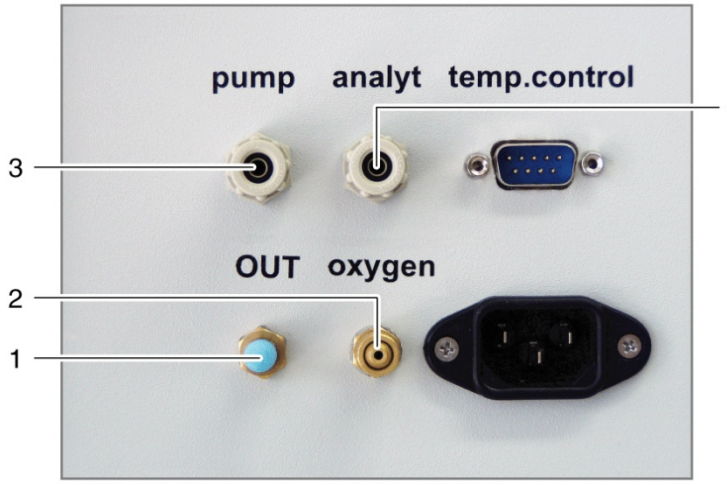
**Attention**

Before connecting add-on devices switch off the analyzer. Always connect the add-on devices to the multi N/C 3100 when it is switched off!

If you connect an external solids module to the analyzer observe the user manual of the solids module in addition to the notes in this user manual!

The connection of an external solids module requires internal valves in the analyzer. Connect the external solids module to the analyzer as follows:

1. Place the external solids module to the right of the analyzer.
2. Connect the solids module and the analyzer to the gas connections:
   - Connect the connection "analyte" on the solids module to the connection "analyte" on the backplate of the analyzer (4 in Fig. 17 p.28).
   - Connect the connection "pump" on the solids module to the connection "pump" on the backplate of the analyzer (5 in Fig. 17 p.28).
3. Connect the serial data cable supplied to the corresponding serial interface (CLD/HT) on the equipment backplate of the analyzer (13 in Fig. 17).
4. Connect the other end of the data cable to the RS 232 interface of the solids module (see user manual of the solids module).
5. Using the menu command CONFIGURATION ▸ EDIT OPTIONS open the window OPTIONS ▸ tab ANALYZER COMPONENTS. Enable the option EXTERNAL SOLIDS MODULE.




1 measuring gas outlet (OUT)
2 carrier gas inlet connection (oxygen)
3 connection to the analyzer "pump" connection
4 connection to the analyzer "analyte" connection

**Fig. 42 Media connections on the backplate of the solids module**

## 5.4 POC module

The POC module is supplied in two versions:

- for manual operation
- for operation with the POC sampler

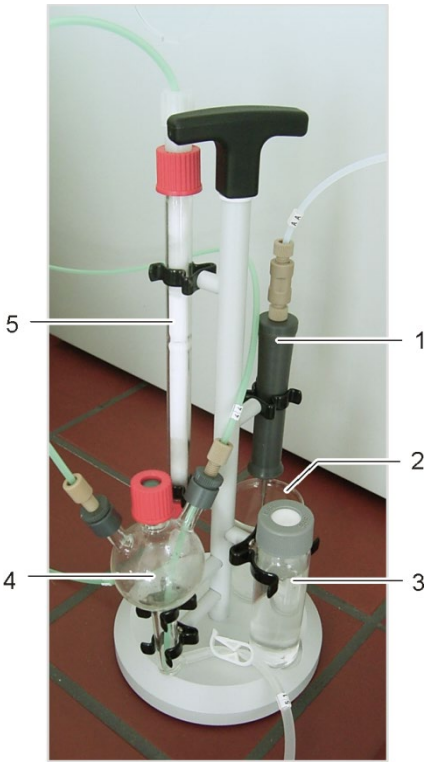
### 5.4.1 Technical data

| Parameter | POC |
|---|---|
| Digestion principle | Purging followed by thermal catalytic oxidation |
| Sample volume | 0.03 fl oz |
| Sample supply | Flow injection – manual or automatic with the POC sampler |

## 5.4.2 Layout of the POC modules

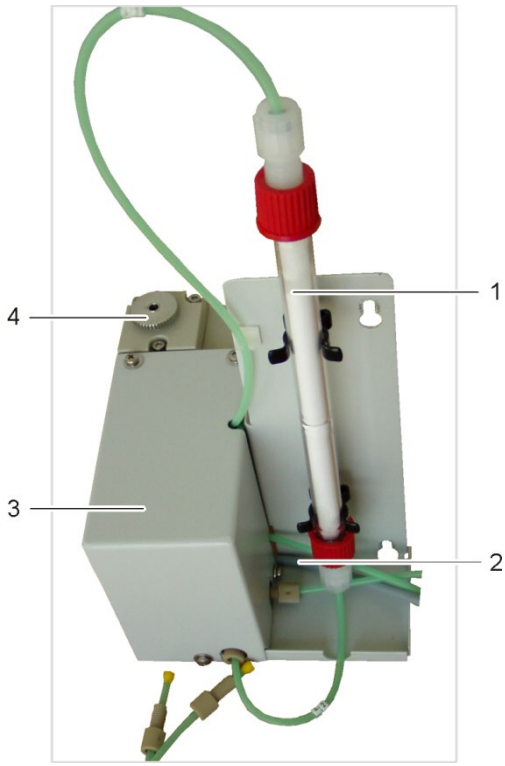
The POC modules consist mainly of:

- POC – reactor with septum
- valve unit for automatic operation with the sampler APG 64
- sample intake cannula specifically for septum vials
- $CO_2$ adsorber with LiOH
- sample tray with 61 positions (included in the delivery of the POC sampler)




1 cannula with handle
2 waste container
3 sample cup with septum
4 POC reactor
5 adsorption tube with LiOH

**Fig. 43 POC module for manual operation**




1 adsorption tube with LiOH
2 electr. connection to the analyzer
3 valve unit
4 POC reactor

**Fig. 44 POC module for automatic operation**

## 5.4.3 Installing the POC modules

The POC module must only be positioned, assembled and installed by the customer service department of Analytik Jena GmbH or by specialist personnel authorized and trained by Analytik Jena GmbH!

Any unauthorized intervention in the POC module reduces or completely invalidates any warranty claims.

### Attention

The analyzer multi N/C 3100 must be switched off before it is connected to the POC module.

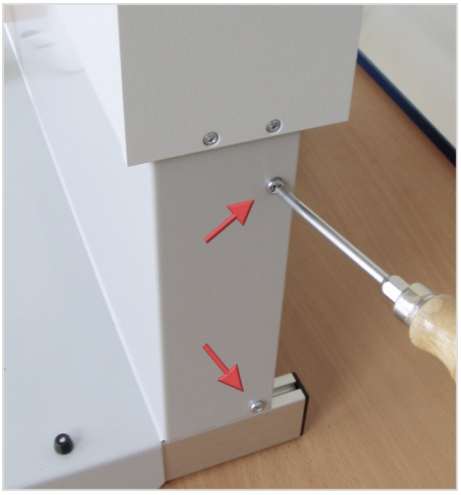
Switch off the main switch at the equipment backplate and disconnect the mains plug from the mains supply.

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

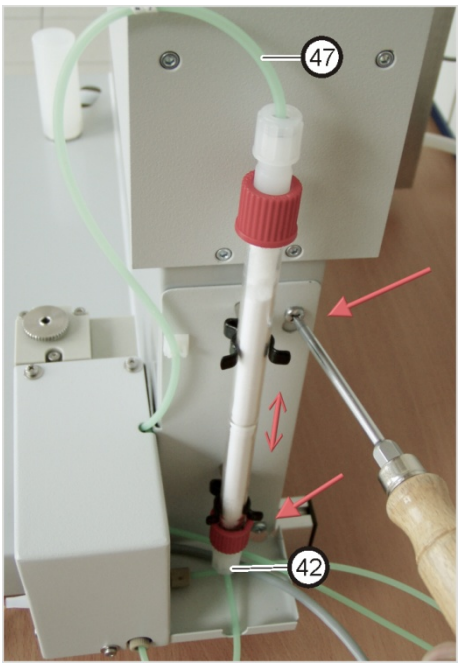
In all other operating modes (TC, NPOC, TIC, TOC, $TN_b$) the carrier gas flow is switched directly to the high temperature reactor.

Connecting the POC module for automatic operation with sampler:

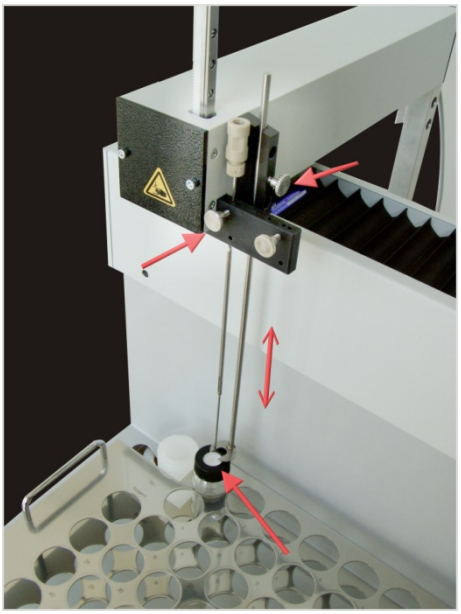
1. Switch off the main switch at the equipment backplate and disconnect the mains plug from the mains supply.
2. Switch off the sampler.



3. Undo the housing screws on the right-hand side of the sampler.

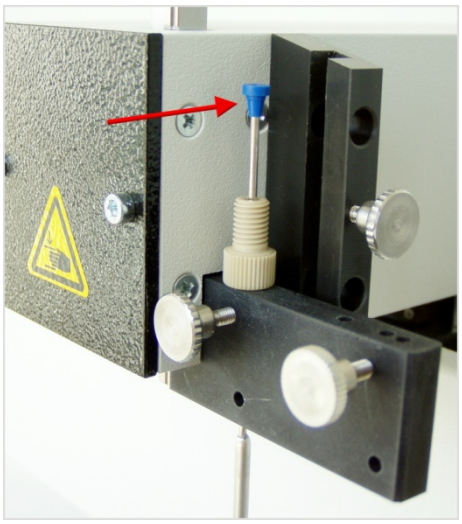



4. Install the filled $CO_2$ adsorber tube between hose no. 47 and hose no. 42.
5. Press the adsorber tube into the clamps of the POC module.
6. Insert the POC module into the housing screws and retighten the screws tightly.



7. Slide the cannula holder of the sampler up.
8. Clamp the special cannula with ventilation slot into the cannula holder of the sampler.
9. Install the holding down clamp.

   With the cannula holder moved up the holding-down clamp and cannulas must be approx. 3mm above the sample cups!



10. At hose AA replace the standard intake cannula with the special cannula.

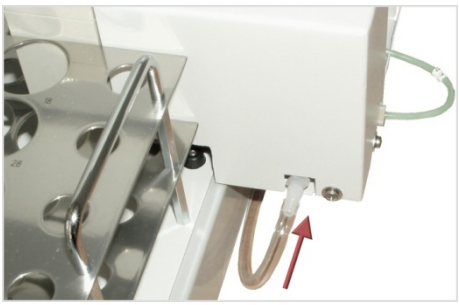
    ATTENTION! Position of the Fingertight connection and ferrules as in the adjacent image!

11. Connect the POC module to the analyzer as follows:

    - Remove the hose protection ramp at the gas connections for the POC module on the backplate of the analyzer (7 in Fig. 17 p. 28).
    - Connect the hose 44 of the POC module to the connection 44 of the analyzer.
    - Connect the hose 45 of the POC module to the connection 45 of the analyzer.

    ATTENTION! Do not swap the gas connections between the POC module and the analyzer!

    - Connect the electrical connection of the POC module with the connection "POC" on the backplate of the analyzer (9 in Fig. 17 p. 28).



12. Attach the waste hose to the waste connection of the POC module and connect it to as suitable waste container or drain.

    ATTENTION! The waste hose must be routed with a continuous down gradient! If necessary, shorten the hose. The hose must not dip into the liquid.

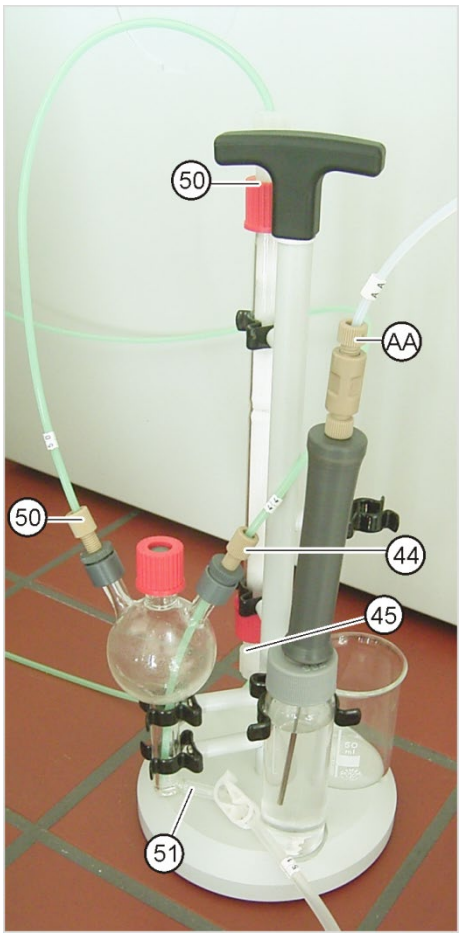
13. Adjust the sampler (see section "Adjusting the sampler with the POC module" p. 91).
14. Check the system for leaks (see section "Inspecting the POC module for leaks" p. 116).

### Connect the POC module for manual operation

After connecting the POC module for manual operation the carrier gas flows always through the POC reactor into the high temperature reactor. We recommend removing the reactor if measurements are carried out in different operating modes (TC, NPOC, TIC, TOC, $TN_b$).

Connect the POC module to the analyzer as follows:

1. Position the POC module to the left of the analyzer.




2. Install the $CO_2$ adsorber tube between hose no. 50 (top end of the adsorber tube) and hose no. 45 (bottom end).
3. Connect hose no. 50 to the POC reactor.
4. Connect the POC module to the analyze:
   - Remove the hose protection ramp at the gas connections for the POC module on the backplate of the analyzer (7 in Fig. 17 p. 28).
   - Connect hose no. 44 to the POC reactor and connection 44 of the analyzer. Hose no. 44 will almost reach into the POC reactor almost down to the bottom.
   - Connect hose no. 45 from the bottom end of the adsorber tube to connection 45 of the analyzer.

   ATTENTION! Do not swap the gas connections between the POC module and the analyzer!
5. Connect the waste hose no. 51 to the bottom end of the TIC reactor. The waste hose is sealed with a hose clamp.
6. Replace the standard intake cannula at hose AA with the special cannula with ventilation slot.

### 5.4.4 POC calibration

It is recommended to carry out a calibration with saccharose.

1. Create a POC method.
2. Load an NPOC method and calibrate it with saccharose standards.
3. Link the measured calibration to the POC method as follows:
   - Negate the query "Link to calibrated method?"
   - Select the POC method in the method list that opens.
   - Using the button [ACCEPT VALUES] transfer the current calibration data to the POC method.

A calibration with dichloromethane standard can also be carried out. However, it should be noted in this case that the high volatility can cause imprecise results. If this calibration is chosen, the POC method is calibrated directly.

# 6 Operation

## 6.1 General notes on working with analyses

When working with analyses observe the following:

- When analyzing strongly acidic saline samples aerosols may form in the TIC condensate container. The capacity of the halogen trap will then exhaust rapidly. The water trap also clogs up quickly. Both components then need to be replaced frequently. If possible, such samples should be diluted before measurement (e.g. 1:10).
- With very strong aerosol formation the analyzer is immediately protected by the integrated aerosol trap (water trap) and the carrier gas supply interrupted. In addition the large water trap must be unplugged from the connection at the TIC condensate container to protect the analyzer.
- For the acidification of samples only hydrochloric acid (HCl) p. a. c = 2 mol/l, created from HCl p. a. (conc.) and TOC water, is to be used.
- For the TIC determination only 10 % orthophosphoric acid ($H_3PO_4$), created from orthophosphoric acid (concentrated) p. a. and TOC water, are to be used.
- When preparing and storing solutions, only clean, non-particulate glass containers (volumetric flasks, sample cups) must be used.
- When preparing and storing solutions in the range of < 1 mg/l it must be noted in particular that the concentrations of the solutions may be slightly modified by components of the laboratory air ($CO_2$, organic vapors).
  You can take the following precautions against this:
- Keep the empty space above the liquids as small as possible. In sampler operation, cover the sample cups on the sample tray with film (differential mode). Remove the source of organic vapors.

## 6.2 Switching on the analyzer (standard commissioning)

**Attention**

Damage to optical and electronic components (detectors, flow sensors) from aggressive combustion products if the copper wool in the halogen trap is used up!

Replace the complete filling of the halogen trap if half of the copper wool is discolored black or the brass wool is discolored!

Always check the following before switching on the analyzer:

- The waste hose is connected to a suitable waste container or drain, clear drainage is ensured and the capacity of the waste container is adequate.
- The gas supply is connected in accordance with regulations and the preliminary pressure is 4 to 6 bar.
- Sufficient phosphoric acid is available in the reagent bottle (0.5 ml for each TIC detection).

- The halogen trap is connected, filled with copper and brass wool and still usable (see safety note above).
- The hoses in the analyzer are connected properly and in good working order.

If applicable, check that additional optional components are connected correctly:

- Sampler
- Chemiluminescence detector (CLD)
- External solids module HT 1300
- POC module

Keep a sample ready and switch on the analyzer as follows:

1. Open the valve at the pressure reducer of the gas supply.
2. Switch on the PC (PC version only).
3. If applicable, switch on any additional components (see user manual of the respective component):
   - Sampler
   - Chemiluminescence detector (CLD)
   - External solids module
4. Switch on the analyzer from the main switch.
   ✓ The LED at the left front door illuminates green.
5. Start the control and analysis software multiWin on the PC and log in with your user name and password.
6. Confirm the query INITIALIZE ANALYZER with [YES] if shown.
   ✓ After successful login the initialization and query of the components starts.

**Attention**

In the window SYSTEM STATE the displays of the components which are not yet ready are shown in read during initialization. During the start-up phase of the analyzer the external communication with the program is blocked.

The individual components have different start-up periods:

- NDIR detector approx. 10 minutes start-up period
- Furnace approx. 10 minutes heating up time
- CLD approx. 30 minutes running-in time

The measuring gas flow reaches the target value (160 ± 10 ml/min) after approx. 1 to 2 minutes.

7. If the analyzer is not ready for measurements after 35 minutes (one or several components are still shown in red in the window SYSTEM STATE), check the hose connections and carry out a fault finding exercise in accordance with the notes in section "Fault Removal" p. 117.
8. If necessary, adjust the NPOC purging flow (see section "Adjust the NPOC purging flow" p. 90).

The NPOC purging flow is set to approx. 100 ml/min and can be increased or reduced dependent on the measuring task.

## 6.3 Switching off the analyzer

### 6.3.1 Switching to standby mode

**Attention**

The analyzer will be damaged if the gas flow is switched off!

The gas supply must not be switched off during standby mode. The required inlet pressure must be available.

Recommendation: Switch to standby mode during pauses of > 30 min between measurements.

1. Click the [EXIT] button on the multiWin user interface.

   The PROGRAM END window opens.

2. Purging the analyzer:

   - For measurements without autosampler:
     Enable the checkbox REVERSE RINSE ANALYZER. Place the sample aspiration cannula into the waste container before starting the backwash.
   - For measurements with the autosampler AS vario, EPA sampler:
     Enable the checkbox REVERSE RINSE ANALYZER. The contents of the sample intake hose is automatically washed back into the purge cup.
   - For measurements with the autosampler AS 10, AS 21:
     Measure an ultrapure water sample at the end of the sequence.
     (The autosamplers AS 10 and AS 21 do not have a purge cup required for the backwash process.)

3. Enable the option STAND-BY ANALYZER and confirm using [OK].

   ✓ The analyzer remains in standby mode.

The sample intake hose is purged with ultrapure water. The rest of the system remains filled.

### 6.3.2 Switching off before longer periods of rest

The analyzer must be completely switched off before extended standstill periods such as weekends or holidays. The analyzer must be purged before it is switched off.

1. Click the [EXIT] button on the multiWin user interface.

   The PROGRAM END window opens.

2. Purging the analyzer:

   - For measurements without autosampler:
     Enable the checkbox REVERSE RINSE ANALYZER. Place the sample aspiration cannula into the waste container before starting the backwash.

- For measurements with the autosampler AS vario, EPA sampler:
  Enable the checkbox REVERSE RINSE ANALYZER. The contents of the sample intake hose is automatically washed back into the purge cup.
- For measurements with the autosampler AS 10, AS 21:
  Measure an ultrapure water sample at the end of the sequence.
  (The autosamplers AS 10 and AS 21 do not have a purge cup required for the backwash process.)

3. Enable the SWITCH OFF ANALYZER option and confirm using [OK].
4. Switch off the analyzer using the power switch.
   ✓ The analyzer is switched off completely.

## 6.4 Carrying out the calibration

### 6.4.1 Preparing and starting the calibration

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

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

Carry out the calibration as follows:

1. In the window SYSTEM STATE select the type of sample supply.
   ✓ This is followed by the initialization of the analyzer.
2. Open the menu command MEASUREMENT ▸ CALIBRATION.
3. In the subsequent query decide whether to select the method to be calibrated or load an already existing calibration table. Then follow the instructions on the screen.
   ✓ After loading the method to be calibrated or after opening an existing calibration table the window CALIBRATION – DATA OF NEW CALIBRATION is opened.

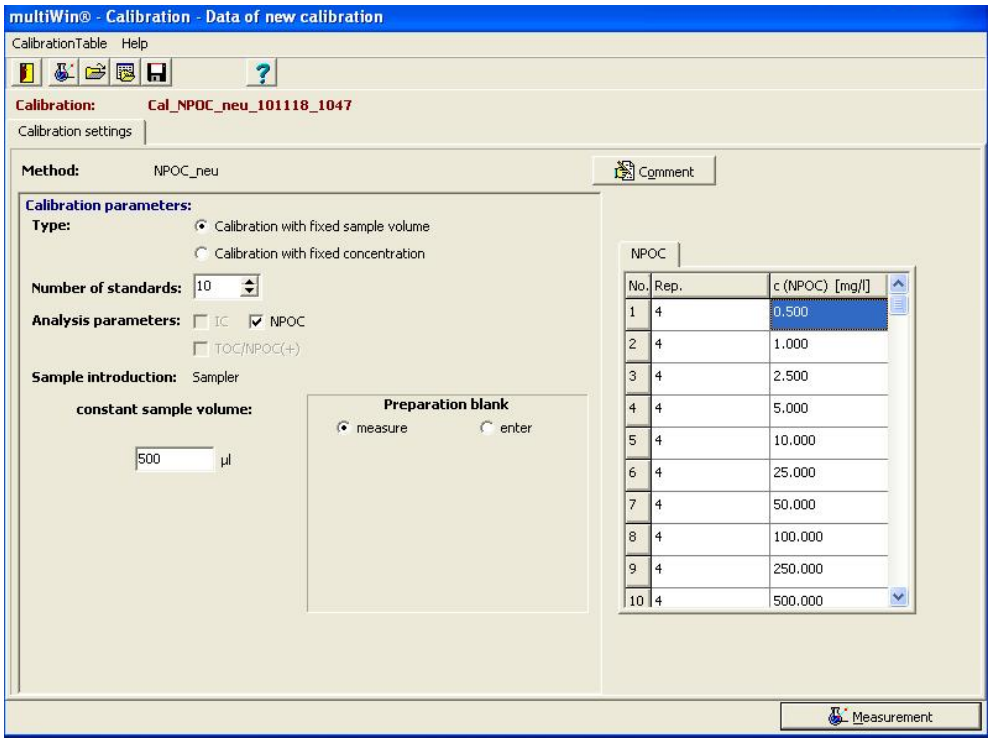



**Fig. 45 Window Calibration – Data of new calibration for new calibration**

4. In the group CALIBRATION PARAMETERS select the calibration type.

   Preferably multipoint calibrations with constant sample volume and variable concentrations should be carried out. In the input field CONSTANT SAMPLE VOLUMES the volume configured in the method is entered automatically. A change is only necessary if the volume differs from the volume configured in the method.

   For CALIBRATION WITH FIXED CONCENTRATION the appropriate concentration of the standard provided must be entered into the input field.

5. In the input field NUMBER OF STANDARDS enter the number of calibration points.

6. Select the ANALYSIS PARAMETERS of the loaded method to be calibrated.

   Not all parameters need necessarily be calibrated. For the calibration of the parameter NPOC plus and concentrations > 0.5 mg/l the parameters IC and TC must be activated individually.

   The calibration for the parameter TOC/NPOC plus must be used when working in a concentration range < 0.5 mg/l. Here a single point calibration is generally sufficient.

7. Under SAMPLE INTRODUCTION the type of sample supply is indicated. The indication is for information only and must not be modified.

8. In the group PREPARATION BLANK select how the preparation water blank value of the standard should be taken into account.

   - Selection field MEASURE:
     The TOC content of the preparation water is measured separately before the calibration. For this, a cup with preparation water must be provided on the first position of the sampler. For manual sample supply the provisioning of the preparation water is first requested.
   - Selection field ENTER:
     The content of the preparation water can be entered as a value.

The preparation water blank value must be specified standardized to 1 ml. If the preparation water blank value is not taken into account enter a 0 in the input field.

9. Complete the calibration table for each parameter to be calibrated in accordance with the standard solutions provided.

   In the column REP. the number of repeat measurements configured in the method is entered automatically. If the outlier selection is enabled in the method, the maximum number is entered. The number of repeat measurements can be manually changed individually for each standard.

10. If necessary, save your calibration table with the menu commands CALIBRATIONTABLE ▸ SAVE CALIBRATIONTABLE or CALIBRATIONTABLE ▸ SAVE CALIBRATIONTABLE AS....

    Calibration tables are automatically given the extension *.kaltab and are saved under ...\Calibration\Tables.

11. Click on the button [MEASUREMENT] and then follow the instructions on the screen.

    Dependent on the method selected and the type of sample supply, additional queries appear or the window CURRENT SAMPLE DATA opens (only for sample supply with sampler).

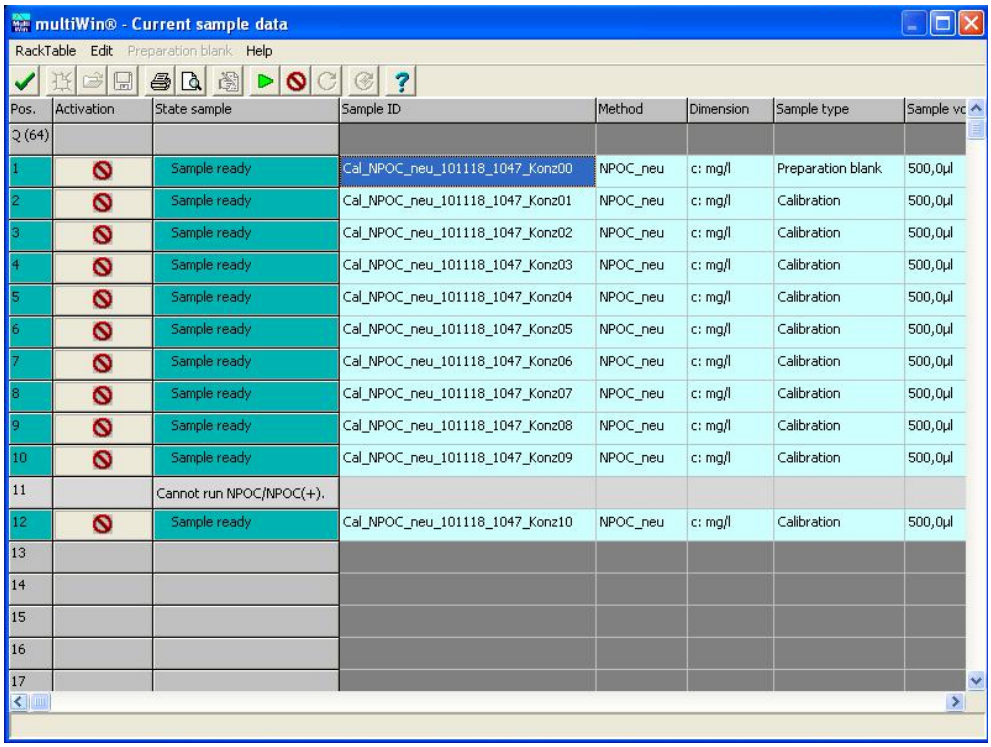



**Fig. 46 Window Current sample data (with sampler operation)**

12. Release the calibration standards in the window CURRENT SAMPLE DATA and exit the window with the button [✓].

13. After opening the window MEASUREMENT click on the button [START F2].

    ✓ The calibration process starts.

## 6.4.2 Display of the calibration results

After completing all calibration measurements the calibration report is automatically opened in the window CALIBRATION – CALIBRATION SETTINGS and can be edited. The calibration report can also be opened later via the menu command DATA EVALUATION ▸ CALIBRATIONREPORT ▸ SELECT CALIBRATIONREPORT.

The window CALIBRATION – CALIBRATION SETTINGS has the tab CALIBRATION DATA and the tab CALIBRATION RESULT.

The CALIBRATION DATA tab displays the calibration settings. Use the [COMMENT] button to enter a note. Use [SIGNATURE] to sign the calibration. In multiWin pharma, only calibrations with the signature status "authorized" can be linked to a method. The tab CALIBRATION RESULT compiles the results for each calibrated parameter.

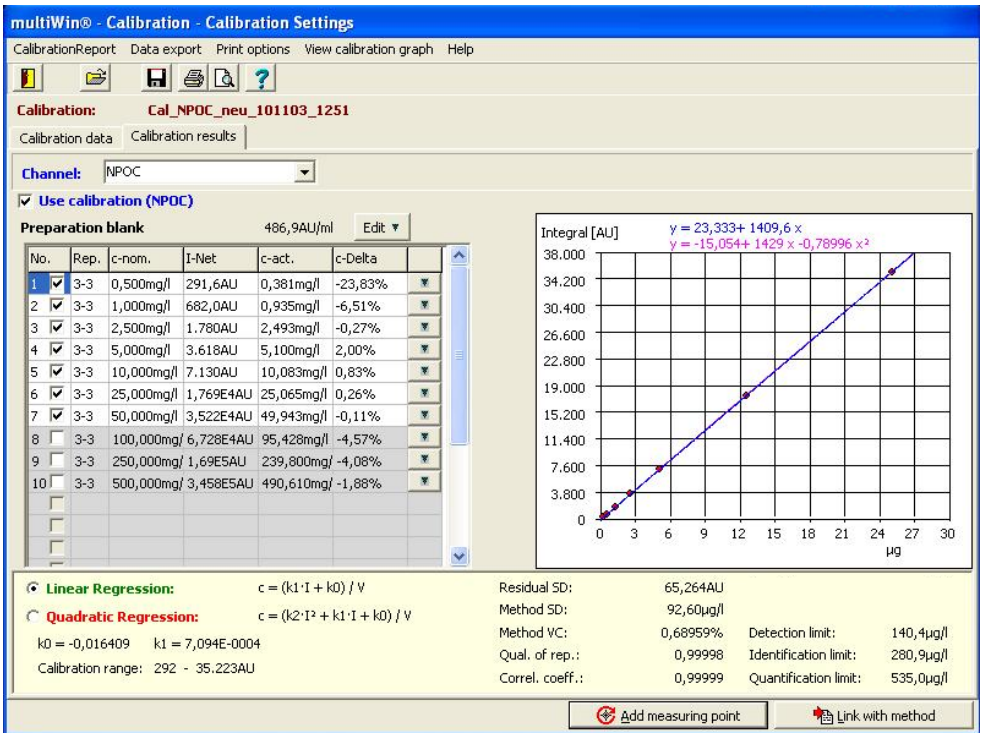



**Fig. 47 Window Calibration – Calibration settings**

Tab Calibration results

| | |
|---|---|
| Result table | The following are displayed:<br>■ number of detections<br>■ target concentration used for constant sample volume<br>■ sample volume used for constant concentration<br>■ average value of the area integers<br>■ average values of the calculated concentrations<br>■ percentage deviation of the calculated concentration and the target concentration |
| LINEAR REGRESSION/ QUADRATIC REGRESSION | Dependent on the methodology selected the regression calculation and determination of the method characteristics is based on individual values or average values of the net integer. For the selected regression type the respective calibration coefficients are displayed. |

| | |
|---|---|
| Calibration diagram | The regression graph can be displayed for the program-internal calibration coefficient determination (x axis - integer, y axis- mass) or for the determination of the method characteristics (x axis - mass; y axis - integer). The view can be switched in the VIEW ▸ CALIBRATION GRAPH menu. |
| Method characteristics | Verification/detection and determination limit:<br>In multiWin the calculation rules of DIN 32645 (calibration function) are used with a significance level of P = 95 %.<br>For the calculation of the determination limit a relative result uncertainty of 33.3 % is being assumed (factor k = 3). Other method characteristics see also section "Method characteristics" p. 36 |

### 6.4.3 Editing an existing calibration

Calibration coefficient, method characteristics and regression graph are recalculated and redisplayed after each change.

The following items can be edited during a calibration:

- Selection of the regression type

  Either linear or quadratic regression can be selected (see Fig. 47). For the selected regression type the respective calibration coefficients and method characteristics are displayed.

- Disabling individual measuring points

  All measuring points enabled by (✓) in the column NO. of the result table are included in the regression calculation. A measuring point can be disabled by removing the (✓) (click in column NO.).

- Disabling individual measured values

  By clicking the button [▼] at the end of each line of the results table you can view the individual measured values (see Fig. 48). Individual values can be disabled by removing the (✓) in the column USE.

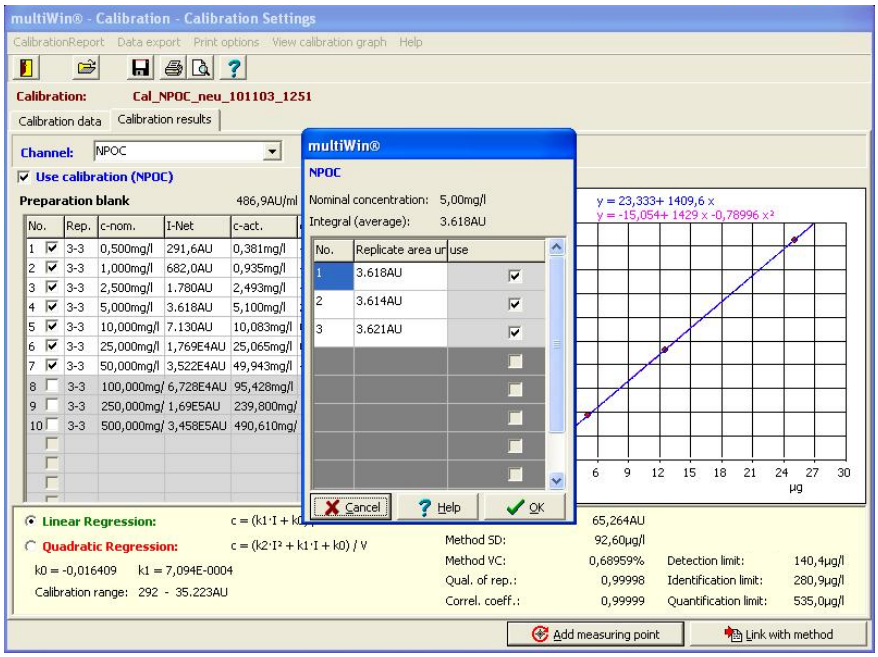



Fig. 48 Disabling individual measured values of a calibration

- Enabling/disabling measured values for preparation water

  The individual values determined for the preparation water can be viewed by clicking on the button [EDIT ▼] and enabled/disabled for the calibration.

- Adding measuring points

  An existing calibration can be extended by additional measuring points. Carry out a measurement with the same method (select CALIBRATION as sample type and enter the target concentration) and select the corresponding analysis report via the button [ADD MEASURING POINT].

  Measuring points can only be added individually.

### 6.4.4 Transferring calibration parameters to a method

Transfer calibration parameters to a method as follows:

1. Select an appropriate calibration range for the respective parameters (e.g. NPOC/TN).

   Up to three linear calibration ranges for each parameter can be stored in a method. It should be ensured that the ranges merge and do not have any gaps! Using calibration function with quadratic regression only one calibration range can be stored in a method.

2. For each selected calibration range and analysis parameter to be transferred enable the field USE CALIBRATION with (✓).

   Not all calibrated parameters need to be transferred to the method.

3. Click on the button [LINK WITH METHOD].

4. Answer the subsequent query "Link to calibrated method?"

   - [YES] the link is made with the calibrated method (default)
   - [NO] the calibration parameters are linked to the selected method

   The method parameters of the calibration and the selected method are not checked! The user must always decide whether such a procedure can be applied to the concrete analytic objective in hand.

5. In the window LINK WITH METHOD: XXX that opens the existing current calibration coefficients (right-hand column) and the newly determined calibration coefficients (left-hand column) are displayed and can be compared.

   The display of the corresponding parameters (e.g. NPOC/TN) can be changed.

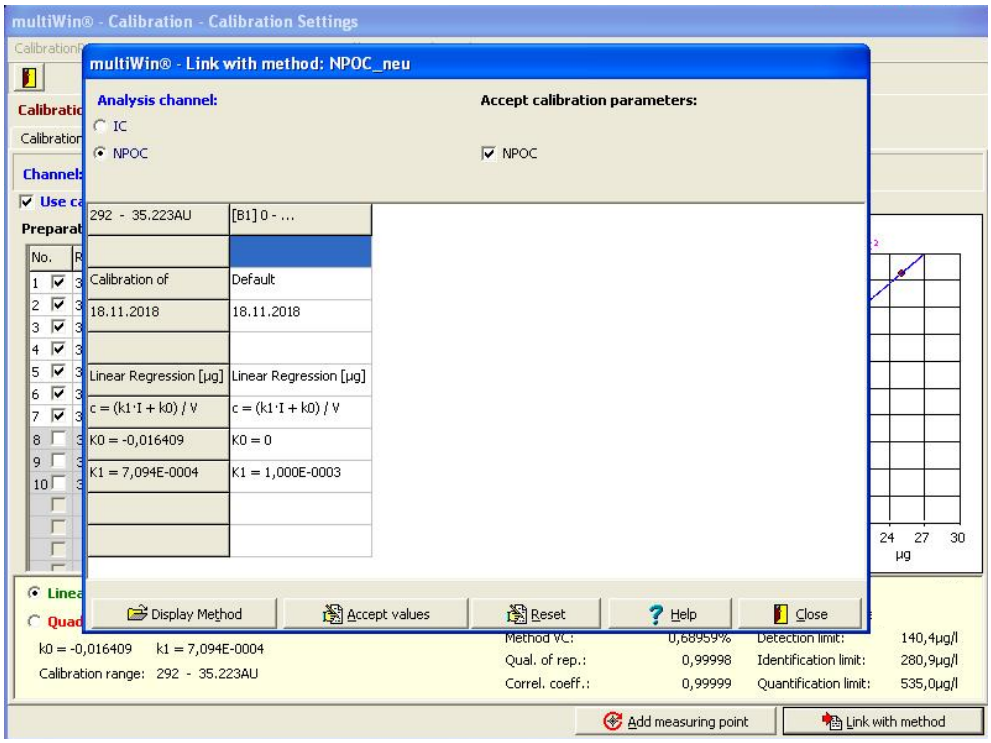



**Fig. 49 Window Link with method**

6. The transfer of the calibration coefficients depends on whether a calibration range or several calibration ranges have been saved in the method:

| | |
|---|---|
| No calibration range exists | Transfer the currently determined calibration data with the button [ACCEPT VALUES].<br>The same calibration coefficients appear in the left-hand and right-hand columns. |
| One or two calibration ranges exist | **Extend the existing calibration range:**<br>Amend the new calibration coefficients with the button [ACCEPT VALUES].<br>Irrespective of the areas the software integrates the new range into the existing ones.<br>Check on the basis of the calibration ranges that a seamless linking of several ranges has taken place.<br>**Replace existing calibration range:**<br>Delete the calibration range.<br>Then proceed as in "Extend existing calibration range". |
| Three calibration ranges exist | A maximum of three calibration ranges can be stored for each parameter in a method. In this case the ranges can only be replaced.<br>Delete the range to be replaced in the right-hand columns using the button [DELETE].<br><br>Transfer the currently determined calibration data with the button [ACCEPT VALUES].<br>Check on the basis of the calibration ranges that a seamless linking of several ranges has taken place. |

The following generally applies:

- Pressing the button [ACCEPT VALUES] causes an automatic allocation of the calibration ranges by the software.
- By pressing the button [DELETE] you first make an initial selection which range should be replaced.
- A **seamless linking** means that the top end of the area of one calibration range corresponds to the bottom end of the area of the next calibration range (see Fig. 50 table, first line)
- The accepted calibration parameters are used for the calculation of all subsequent analyses with this method.

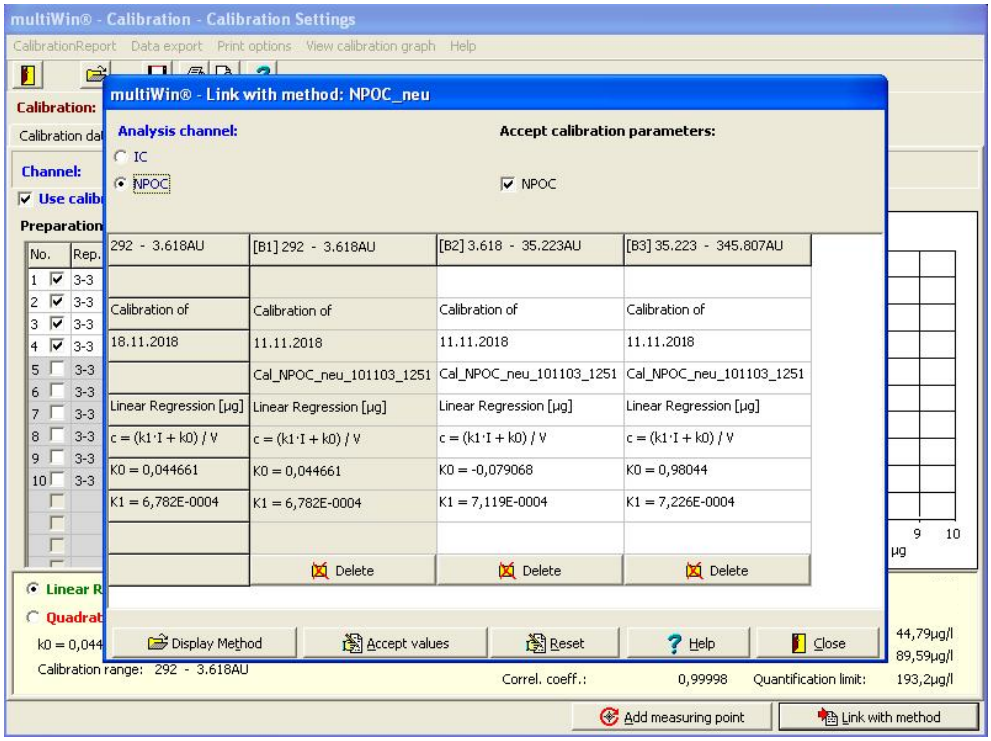



Fig. 50 **Window Link with method with three areas**

## 6.4.5 Managing calibration data

Printing calibration data

Print the calibration report as follows:

1. In the window CALIBRATION – CALIBRATION SETTINGS enable the option USE CALIBRATION.
2. Define the scope of printing under the menu PRINT OPTIONS:
   - Print calibration graph and/or
   - Print replicate area units
3. Start the printout with the menu command CALIBRATIONREPORT ▸ PRINT.

Exporting calibration files

Calibration data are exported via the menu DATA EXPORT in the window CALIBRATION – CALIBRATION SETTINGS. You have the following options for exporting calibration data:

- CalibrationReport into an export file
  The calibrationReport (with the extension *.ajc) is saved in the export directory ..\Calibration.

- Export into a CSV file (*.csv)
  The CSV file is saved in the preconfigured directory (default ...\multiWin\CSV).
  The directory is selected in the window OPTIONS ▸ tab FILES AND DIRECTORIES (main window menu command CONFIGURATION ▸ EDIT OPTIONS).
- Export to clipboard

Reopening a calibration report

1. In the main window open the menu command DATA EVALUATION ▸ CALIBRATIONREPORT.
2. In the window SELECTION CALIBRATIONREPORT select the calibration report.

   In the window SELECTION CALIBRATIONREPORT filters can be set, if necessary, and the records sorted by clicking on the respective header.

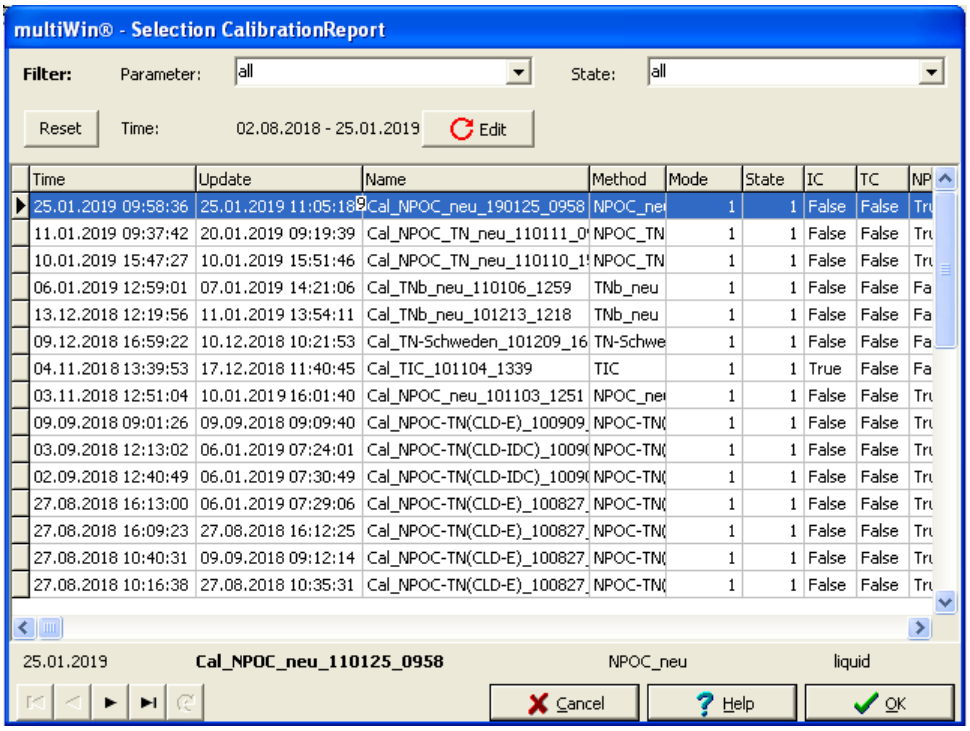



Fig. 51 Window Selection CalibrationReport

3. Highlight the corresponding calibration report and click on the button [OK].

   ✓ The calibration report is displayed.

## 6.5 Carrying out a measurement

**Note for multiWin pharma:** Only methods with the signatures status "authorized" can be used for the measurement.

### 6.5.1 Measurement with manual sample supply

Carry out a measurement with manual sample supply as follows:

1. Insert the sample intake cannula and the purging cannula into the sample.
2. Using the menu command METHOD ▸ NEW create a new method or load an existing method.
   To do so open via the menu command METHOD ▸ LOAD the database window METHOD SELECTION, highlight the desired method and confirm the selection by clicking the button [OK].
3. In the window SYSTEM STATE select the manual sample supply by clicking the button [MANUAL].
   ✓ This is followed by the initialization of the analyzer.
4. In the window SYSTEM STATE check the following entries:
   - Optical bench - OK
   - CLD, if applicable, or, if applicable, ChD - OK
   - Gas flow - OK
   - Temperature - OK
5. If one of the entries is incorrect (shown in red), carry out a fault finding exercise in accordance with the notes in section "Fault Removal" p. 117.
6. Start the measurement.
7. Click on [START MEASUREMENT] or open the menu command MEASUREMENT ▸ START MEASUREMENT.
   ✓ The window MEASUREMENT START opens.
8. Enter the sample ID and, if applicable, a name for the analysis table. You can also enter the dilution, sample type, unit and a remark.
9. With [START ▸] open the window MEASUREMENT.
10. Start the measurement by clicking the button [START F2] and follow the instructions of the control and analysis software.
    ✓ At the end of the measurement, the results appear in the analysis report or in the selected analysis table.

### 6.5.2 Measurement with sampler

**Attention**

After transport or prolonged storage of the analyzer the sampler must be readjusted during recommissioning.

1. Using the menu command METHOD ▸ NEW create a new method or load an existing method.
   To do so open via the menu command METHOD ▸ LOAD the database window METHOD SELECTION, highlight the desired method and confirm the selection by clicking the button [OK].
2. In the window SYSTEM-STATUS select sample supply with sampler by clicking the button [SAMPLER].
   ✓ This is followed by the initialization of the analyzer.
3. In the window SYSTEM STATE check the following entries:
   - Optical bench - OK
   - CLD, if applicable, or, if applicable, ChD - OK
   - Gas flow - OK
   - Temperature - OK
4. If one of the entries is incorrect (shown in red), carry out a fault finding exercise in accordance with the notes in section "Fault Removal" S. 117.
5. Fill the sample cups with the measuring liquid and place them onto the sample tray.
6. **Only for NPOC measurements with AS vario (ER) or EPA/POC sampler:**
   If you wish to use the automatic acidification function of the autosampler, fill the acid cup with HCl (c = 2 mol/l) and place the cup into the acid position of the sample tray:
   For the AS vario (ER) autosampler:
   - Position 28 on sample tray 47 (dilution rack)*
   - Position 42 on sample tray 52
   - Position 55 on sample tray 72
   - Position 85 on sample tray 100
   - Position 85 on sample tray 146

   * Automatic acidification can only be applied if the dilution algorithm is deactivated in the menu OPTIONS ▸ PROCESS CONTROL. If the dilution algorithm is enabled, the original sample needs to be acidified manually

   For the EPA sampler: Position 54 on sample tray 64

   For the POC sampler: Position 51 on sample tray 61
7. Start the measurement.
8. Click on [START MEASUREMENT] or open the menu command MEASUREMENT ▸ START MEASUREMENT.
   ✓ The window MEASUREMENT START opens.

9. In the window MEASUREMENT START enter a name for a new analysis table or select an existing analysis table with [EDIT].
10. With [START ▸] open the window CURRENT SAMPLE DATA.
11. Open an existing rack table or enter the sample name in the column Sample ID in accordance with the assignment of the sample rack. You can also enter the dilution, sample type, unit and a remark.
12. With [▸] release the samples. Confirm the entries with [✓].
    ✓ The rack table will be closed.

    A query follows whether the rack table should be saved. If you want to reuse the entries later, open the default window for saving files with [YES].

    Next the window MEASUREMENT opens. Start the measurement with [START F2] and follow the instructions of the control and analysis software.
    ✓ At the end of the measurement the results appear in the analysis table.

## 6.6 Dilution

### 6.6.1 General

In conjunction with a special sample tray the dilution can be automatic in sampler operation. You will receive the KeyCode with the delivery of the dilution unit (for retrofits). After extending or modifying the configuration of the analyzer a new release using the KeyCode is necessary.

1. Open multiWin and log in as an administrator.
2. Open the menu command CONFIGURATION ▸ KEYCODE.
   ✓ multiWin then quits automatically.
3. After a restart of the program respond to the KeyCode prompt.

### 6.6.2 Automatic TC dilution

Use automatic dilution if samples with very high TC content or in an unknown highly loaded matrix are to be measured:

- to avoid loading the reactor unnecessarily with high contents of inorganic salts and acids (service life),
- to save analysis time, and
- to use the preferred calibration range.

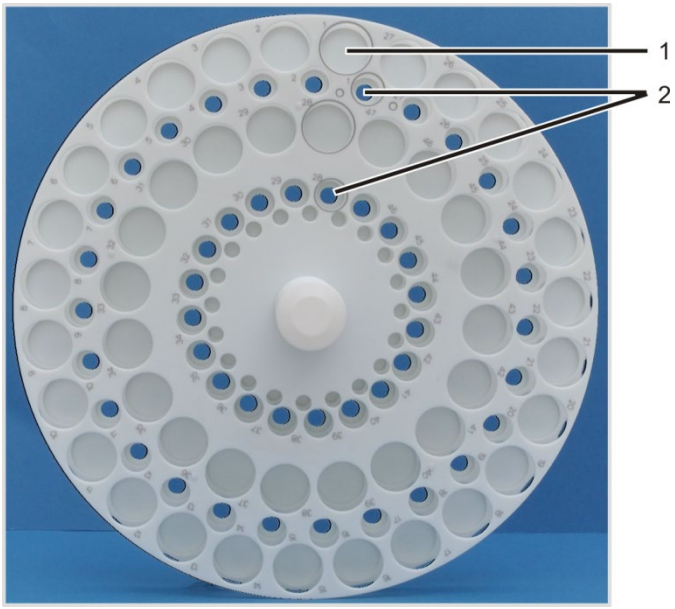
Use automatic dilution as follows:

1. Use a dilution sample tray with suitable cannula holder.
2. With the menu command CONFIGURATION ▸ EDIT OPTIONS open the window OPTIONS tab ANALYZER COMPONENTS.
3. In the SAMPLER group, select tray size 47 DILUTE for the dilution sample tray of the AS vario.
4. On the tab PROCESS CONTROL release the dilution:

- Enable the field USE DILUTION ALGORITHM with [✓].
- Enable the option AUTOMATIC DILUTION.

5. Populate the dilution sample tray with empty 50 ml sample cups.
6. Fill the sample to be diluted into the 12 ml sample cups and populate the dilution sample tray.

   Samples not to be diluted are filled as usual into the 50 ml sample cups.




1 Positions 1-47 for 50 ml tubes
2 Positions 1-47 for 12 ml tubes

**Fig. 52 Dilution sample tray of the AS vario**

7. Fill fresh ultrapure water into the ultrapure water bottle.
8. Using the menu command INSTRUMENT ▸ SAMPLER ALIGNMENT open the window with the same name.
9. Adjust the sample aspiration cannula to the sample tray (see "Adjusting the AS vario (ER) autosampler" section, p. 88).
   Adjust position 1 in a large sample tube (50 ml). Check position 1 in a small sample tube (12 ml).
10. Open the menu command MEASUREMENT ▸ PREPARATION BLANK, if necessary, and determine the blank value of the dilution water.

    The process is defined internally, the water is removed from the ultrapure water bottle.

11. Create or load the rack table. In the field DILUTION select the desired ratio; the following dilutions are possible:

    - 1 in 5
    - 1 in 10
    - 1 in 25
    - 1 in 50
    - 1 in 100

12. Start the measurement.

✓ The original sample is diluted in accordance with the selected dilution ratio in the 50 ml sample cups provided.

When working in the NPOC mode the samples are each diluted in a complete series and then analyzed. The diluted samples are purged.

If the dilution algorithm is activated in the software, the option for automatic acidification is deactivated. For NPOC methods, the user must therefore manually acidify the original samples to pH 2. Alternatively, the user can pipette acid into the 50 ml vials in which the autosampler dilutes the samples. In both cases, check whether the samples actually reach pH 2 so that the TIC is completely removed during purging.

The number of possible multipoint detections of a sample results from the selected method, the injection volume and the number of rinsing cycles. At least three triple detections are possible. An error message appears if the sample volume of the diluted sample is insufficient. In this case, adjust the method accordingly.

In the analysis report, the concentration of the primary sample is shown. The area integers specified are the integers determined for the diluted sample.

### 6.6.3 Intelligent TN dilution

For the nitrogen detection of unknown samples or in unknown sample matrices at higher concentrations the dilution of the sample is recommended to improve the recovery rate for different nitrogen compounds.

In the dilution mode samples with a $TN_b$ content >12 mg/l TNb are diluted automatically. The precise threshold concentration for the dilution depends on the sample matrix, on the nitrogen compounds to be detected, on the sample volume used and on the ageing state of the combustion tube used.

The following threshold values apply to the intelligent dilution:

- from approx. 12 mg/l $TN_b$ automatic dilution 1 : 10
- from approx. 120 mg/l $TN_b$ automatic dilution 1 : 20

For working in the dilution mode calibrate the method up to 15 mg/l $TN_b$. When selecting the calibration function pay particular attention to the calculation of the ACTUAL concentration in addition to the regression coefficient. To obtain exact results later on, the deviation between the ACTUAL and the TARGET concentrations should be max. 5 % over the whole calibration range.

Use the intelligent dilution as follows:

1. Use a dilution sample tray with suitable cannula holder.
2. With the menu command CONFIGURATION ▸ EDIT OPTIONS open the window OPTIONS ▸ tab ANALYZER COMPONENTS.
3. In the SAMPLER group, select tray size 47 DILUTE for the dilution sample tray of the AS vario.
4. On the tab PROCESS CONTROL release the intelligent dilution:
   - Enable the field USE DILUTION with [✓].
   - In the list select the option INTELLIGENT DILUTION.
5. Fill the samples into the 50 ml sample cups and populate the dilution sample tray.
6. Place empty 12 ml sample cups onto the sample tray.

7. Fill fresh ultrapure water into the ultrapure water bottle.
8. Using the menu command INSTRUMENT ▸ SAMPLER ALIGNMENT open the window with the same name.
9. Adjust the sample aspiration cannula to the sample tray (see "Adjusting the AS vario (ER) autosampler" section, p. 88).
   Adjust position 1 in a large sample tube (50 ml). Check position 1 in a small sample tube (12 ml).
10. If necessary, determine the blank value of the dilution water via the menu command MEASUREMENT ▸ PREPARATION BLANK.

    The process is defined internally, the water is removed from the ultrapure water bottle.
11. Create or load the rack table.

    The dilutions entered into the rack table do not relate to the intelligent dilution and have no effect on it. If a different entry than 1 in 1 is made here, this dilution must first take place manually; the calculation is automatic.
12. Start the measurement.

    ✓ The original sample is measured and after the first detection either automatically diluted in the sample cup for this purpose in accordance with the TN content or continued with the repeat measurements configured in the method.

The number of possible multipoint detections of a sample results from the selected method, the injection volume and the number of rinsing cycles. At least triple detections (not in differential mode) are possible. An error message appears if the sample volume of the diluted sample is insufficient. In this case adjust the method accordingly.

In the analysis report the concentration of the primary sample is shown. The area integers specified are the integers determined for the diluted sample.

If a sample has been through automatic intelligent dilution, the diluted sample is given an extension to the originally issued sample ID (_iV_1_10 for dilutions 1:10 or _iV_1_20 for dilutions 1:20) and appears in the analysis table directly after the original sample.

The intelligent dilution is only intended for the $TN_b$ detection and not for the TC detection. A dilution is triggered by the threshold values for TN alone. Even if the carbon detection has been selected in the method, no further detection of the carbon is carried out in the original sample once the nitrogen threshold values have been exceeded. The TC of the diluted samples is generally also detected if selected in the method.

The diluted sample is not purged! For an exact detection of the TC or TOC of these samples a separate analysis cycle for the TOC detection without dilution or with automatic dilution should be carried out.

When using the dilution option for TN with concurrent TOC measurement do not work in the differential mode, because then the sample volume in the dilution cup is not sufficient for a triple detection.

# 7 Maintenance and care

## 7.1 Maintenance intervals

| **Analyzer** | |
|---|---|
| Maintenance task | **Maintenance interval** |
| Clean and service the equipment | weekly |
| Clean drip tray and reagent bottle | weekly and after filling |
| Inspect all hose connections for tight fit | weekly |
| Inspect the fastening screws for tight fit | monthly |
| **Water traps** | |
| **Maintenance task** | **Maintenance interval** |
| Check gas flow | daily |
| Replace water traps | as required, after 6 months at the latest |
| **Halogen trap** | |
| **Maintenance task** | **Maintenance interval** |
| Check copper wool for discoloration | daily |
| Replace used copper/brass wool | if half the copper wool is discolored black or the brass wool is discolored |
| **Combustion tube** | |
| **Maintenance task** | **Maintenance interval** |
| Check for cracks and damage | during catalyst replacement |
| Inspect catalyst and replace, if necessary | as required, after corresponding message in multiWin at the latest |
| Clean combustion tube | during catalyst replacement |
| Renew (replace) combustion tube<br>Replace combustion tube and catalyst | as required, after 12 months at the latest |
| **TIC condensate container** | |
| **Maintenance task** | **Maintenance interval** |
| Check for cracks and damage | 3 months |
| Cleaning the TIC condensate container | as required, after 12 months at the latest |
| **Condensation coil** | |
| **Maintenance task** | **Maintenance interval** |
| Check for cracks and damage | 3 months |
| Clean condensation coil | as required, after 12 months at the latest |
| **Condensate pump** | |
| **Maintenance task** | **Maintenance interval** |
| Check for leaks. | 3 months |
| Replace porous pump hose | as required, after 12 months at the latest |

| **Phosphoric acid pump** | |
|---|---|
| **Maintenance task** | **Maintenance interval** |
| Check for leaks. | 3 months |
| Replace porous pump hose | as required, after 12 months at the latest |
| **Syringe pump** | |
| **Maintenance task** | **Maintenance interval** |
| Check for leaks. | 3 months |
| Clean metering syringe | as required, after 12 months at the latest |
| **POC module** | |
| **Maintenance task** | **Maintenance interval** |
| Checking the operation of the adsorber | monthly, dependent on the organic content of the sample |
| Replace LiOH | as required, after the adsorber material becomes sticky at the latest |
| Check for leaks. | as required, at least monthly |
| Replace septum of the POC port | as required, at least monthly |

**CAUTION**

Make sure that all connections are gas-tight again after maintenance work:

- Do not insert the Fingertight screw connections twisted!
- Tighten all screw connections finger-tight.

Check the system for leaks (see section "Inspecting the system for leaks" p. 116).

To carry out regular inspections and maintenance tasks always ensure that the doors and the left side wall of the analyzer are freely accessible.

## 7.2 Adjustment and setup tasks

### 7.2.1 General notes for adjusting the autosampler

During adjustment, the cannulas to the sample tray are adjusted so that they are optimally immersed into the sample tubes and/or wash cups.

An adjustment of the sampler is necessary:

- before the first start
- after each change in the size of the sample tray
- during recommissioning after transport or storage

Adjusting the AS 10 and AS 21 autosamplers is described in the "Sampler" section, p. 41.

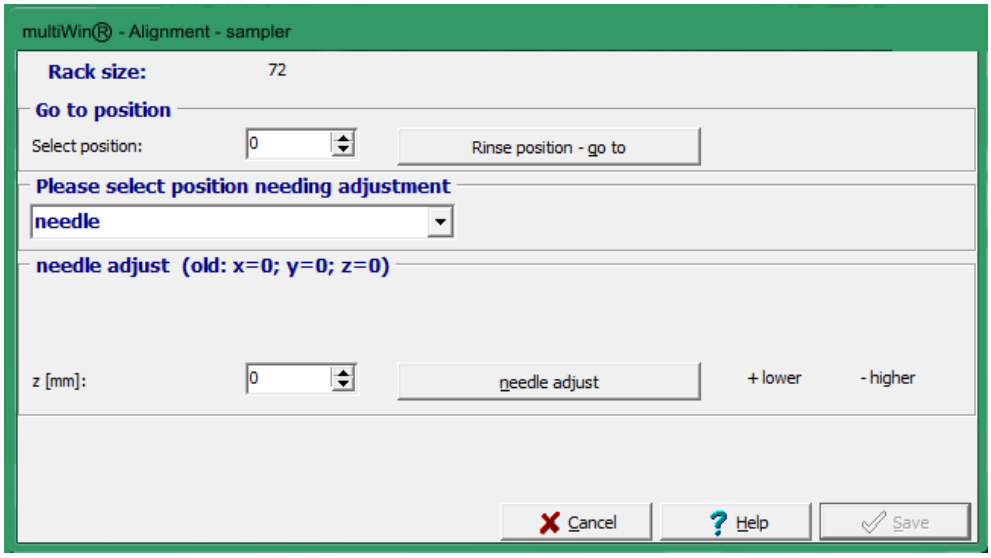
## 7.2.2 Adjusting the AS vario (ER) autosampler

### Attention

The cannulas can bend! Before adjusting the sampler undo the screw connections of the sample aspiration and purging cannulas!

For NPOC measurements, the immersion depth for automatic acidification (z position) depends on the immersion depth in position 1. Adjust the cannula in position 1 and check the adjustment by a test measurement. Make sure the cannula perforates the cover but does not enter the sample liquid when delivering the acid.

1. Start the multiWin software and wait for device initialization.
2. Select the INSTRUMENT ▸ SAMPLER ALIGNMENT menu command to open the window with the same name.
3. In the PLEASE SELECT POSITION NEEDING ADJUSTMENT group, select NEEDLE from the list field.
   The autosampler arm will move over the adjustment points on the sample tray.

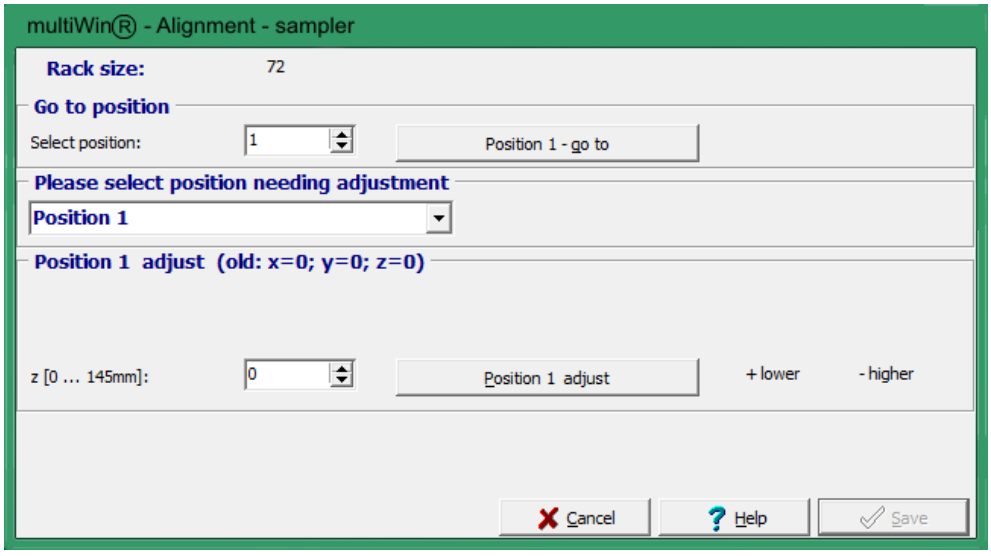



4. Increase or decrease the z values until the cannulas are positioned approx. 2 cm above the adjustment points and click the [NEEDLE ADJUST] button.
5. Align the cannulas with the two adjustment points by carefully bending them.

Fig. 53 Adjustment points on the sample tray

Adjust the immersion depth of the sample aspiration cannula into the wash cup and into a sample tube in position 1 of the sample tray:

6. In the PLEASE SELECT POSITION NEEDING ADJUSTMENT group, select RINSE POSITION or POSITION 1 from the list field.




7. To adjust position 1, place a sample tube with magnetic stirrer onto the sample tray.
8. Increase or reduce the z values to align the rinse position or position 1. Adjust the height of the cannulas to still allow the stirrer free movement.

   Adjust the height of the cannulas in the rinse position so that the cannulas immerse at least 1 cm into the rinse vessel. For AS vario ER set the maximum z-value of z = 145 mm so that the cannulas are sufficiently rinsed from the outside.

   Adjust the height of the cannulas in position 1 to allow the stirrer free movement (distance about 5 mm).
9. Click the [RINSE POSITION ADJUST] or [POSITION 1 ADJUST] button.
   The autosampler will move to the new position. Repeat this step until the cannula position is optimal.
10. Click [SAVE].

✓ The adjustment values will be taken over.

11. Open the ALIGNMENT SAMPLER window again and click the corresponding button to move to the rinse position/position 1 again to check the alignment.

Any position on the sample tray can be moved to for checking.

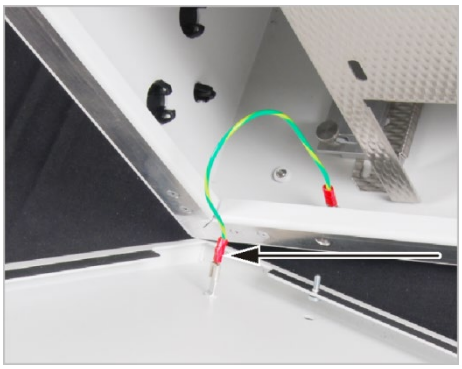
### 7.2.3 Adjust the NPOC purging flow

**CAUTION**

There is a risk of burns at the combustion furnace! Proceed with the greatest care when adjusting the NPOC purging flow via the NPOC needle valve!

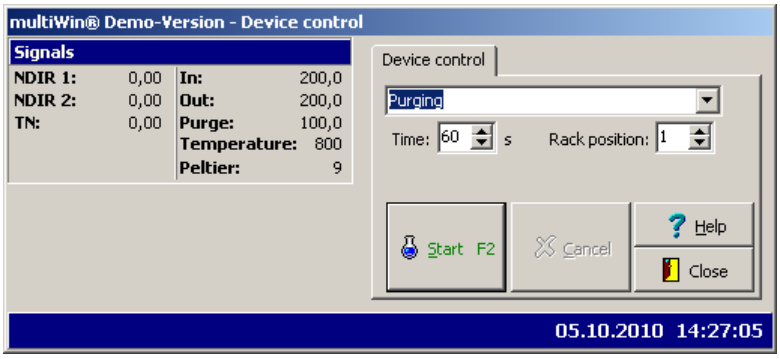
The NPOC purging flow has been preconfigured to approx. 100 ml/min. Dependent on the measuring task you can increase or reduce the NPOC purging flow via the NPOC needle valve. The NPOC needle valve is located behind the left side wall adjacent to the combustion furnace.

Adjust the NPOC purging flow as follows:



1. Open the left side wall of the analyzer. If necessary, move the autosampler to the side. Be careful not to bend the cannulas.
   Unscrew the four fastening screws; the screws are undetachable and remain in the wall.
   Disconnect the grounding conductor and safely put the side wall aside.

2. In multiWin use the menu command INSTRUMENT ▸ DEVICE CONTROL to open the window DEVICE CONTROL.




3. From the list field select the option PURGING.

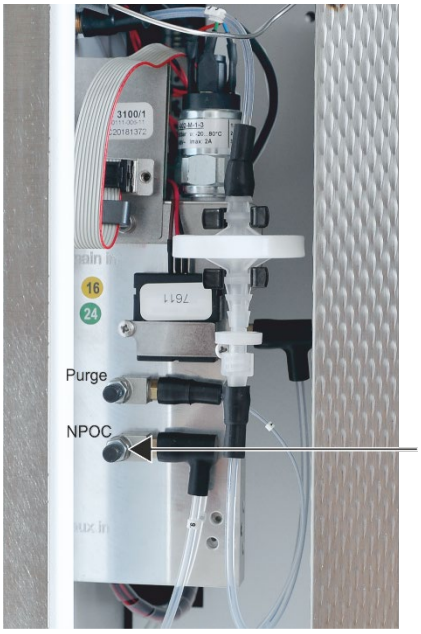
   For sample supplied with sampler:

   - Select the purging time in the field TIME between 1 and 900 seconds.
   - In the RACK POSITION field, select any position on the sample tray in which you want to monitor the purge flow.
   - Place a sample cup with ultrapure water onto this position.

For manual sample supply:

- Select the purging time in the field TIME between 1 and 900 seconds.
- Insert the purging hose into the cup filled with ultrapure water for which the purging flow is to be adjusted.

4. Click on the button [START F2].




5. Undo the adjustment screw at the NPOC needle valve.
6. Regulate the NPOC purging flow:
   - Increasing the NPOC purging flow – turn needle valve to the left
   - Reducing the NPOC purging flow – turn needle valve to the right
   - In the window SYSTEM STATE check the flow indication
7. Relock the adjustment screw at the needle valve.
8. Close the side wall.
   Connect the grounding conductor connection to the left side wall.
   First screw the screws into the bottom and then the top side. Tighten the screws in turn.

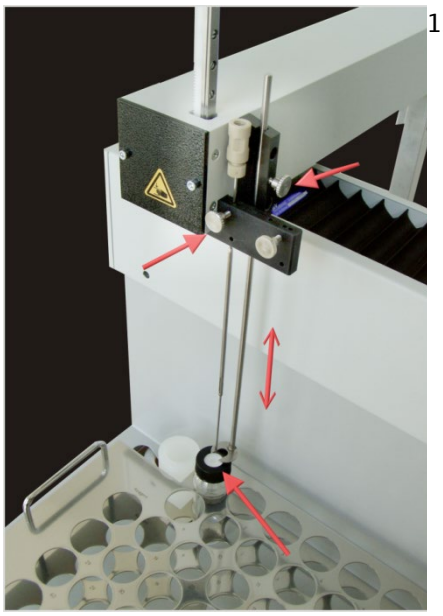
### 7.2.4 Adjusting the sampler with the POC module

An adjustment of the sampler is necessary:

- before the first start
- during recommissioning after transport or storage

During the adjustment the sample intake cannula must be adjusted for the rinse position, the sample position 1 and the POC reactor position on the sample tray. The alignment is carried out by increasing or reducing the x, y and z values.

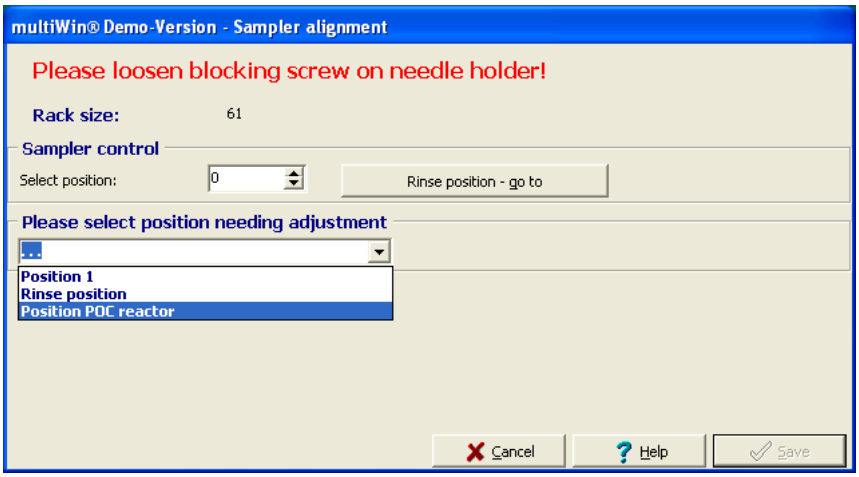
For sample tubes with septum caps, a special sample aspiration cannula with piercing function is required (piercing needle with ventilation slot).



1. Install the special sample intake cannula in the cannula holder.

   ATTENTION! The cannula can bend! Release the fastening screws of the sample aspiration cannula before performing the adjustment. Clamp the cannula into the holder such that the cannula tip does not get immersed in the sample tube.

2. Using the menu command INSTRUMENT ▸ SAMPLER ALIGNMENT open the window with the same name.
3. In the group PLEASE SELECT POSITION NEEDING ADJUSTMENT select in the list field the entry POSITION 1, RINSE POSITION or POSITION POC REACTOR.

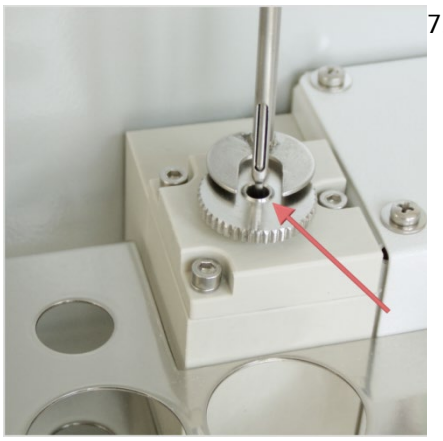



4. Increase or reduce the x, y and z values to align the rinse position or position 1:
   - X direction: forward and back movement
   - Y direction: left or right movement
   - Z direction: up or down movement

5. Adjust position 1
   - To adjust the x and y positions remove the sample cup and index to the position. Place the stirrer onto the sample tray in this position. If it is at the center of the position, it is correctly adjusted.
   - The y value must not be smaller than 33 mm to ensure correct operation.
   - To adjust the z position place the sample cup with screw closure and septum cap (e.g. EPA sample cup) into the sample tray.
     Adjust the special needle in the z direction until approx. 2 mm of the ventilation slot are visible above the septum.

   The ventilation slot must be above and below the septum, otherwise the pressure compensation within the sample cup cannot be guaranteed.

6. Adjust the rinse position.
   - Adjust the x and y positions until the cannula is at the center of the rinsing cup.
   - In the z direction the special cannula may only dip low enough for the ventilation slot to be visible at the top edge of the rinsing cup.



7. Adjust the POC reactor position.
   - Adjust the needle as precisely as possible in the x and y directions.
   - Select the injection depth (z direction) of the special cannula in the POC reactor (septum port) until the whole thick shaft (including ventilation slot) of the needle is above the port.

8. After each change of the x/y/z direction, click the [RINSE POSITION ADJUST], [POSITION 1 ADJUST] or [POSITION POC REACTOR ADJUST] buttons.
   The autosampler will then move to the new coordinates. Repeat this step until the cannula position is optimal.
9. Click [SAVE].
   ✓ The adjustment values will be taken over.
10. Open the ALIGNMENT SAMPLER window again and move to the selected position or any measuring position to check the alignment.

### 7.2.5 Adjusting the EPA sampler

**Attention**

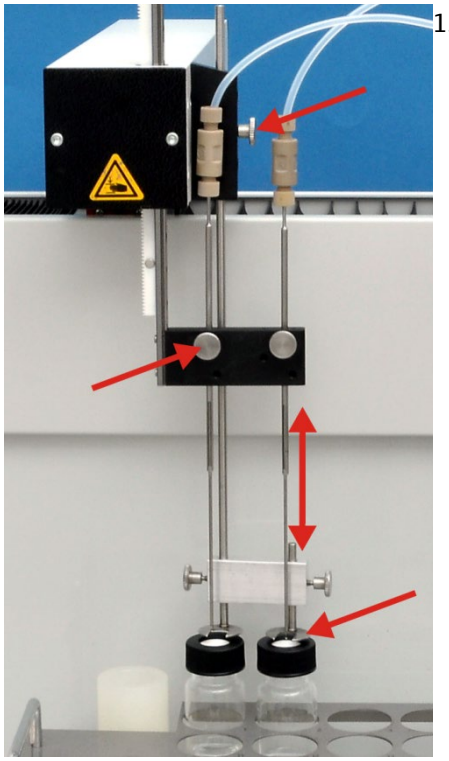
The cannulas can bend! Before adjusting the sampler undo the screw connections of the sample aspiration and purging cannulas!

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

During adjustment, the sample aspiration cannula to the rinse position and to sample position 1 must be adjusted. The alignment is carried out by increasing or reducing the x, y and z values.

For sample tubes with septum caps, special sample aspiration purging cannulas with a piercing function are required (piercing needles with ventilation slot).

For NPOC measurements, the immersion depth for automatic acidification (z position) depends on the immersion depth in position 1. Adjust the cannula in position 1 and check the adjustment by a test measurement. Make sure the cannula perforates the cover but does not enter the sample liquid when delivering the acid.

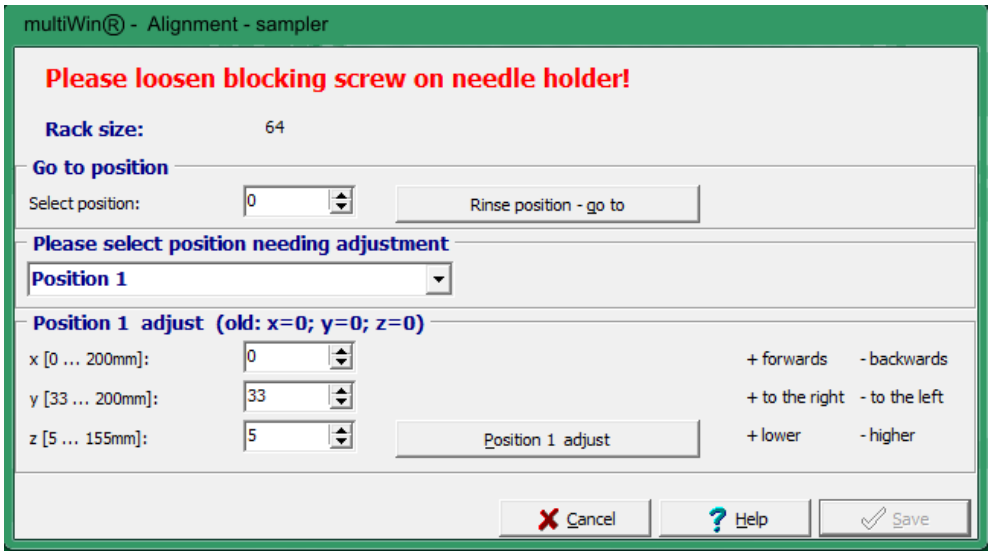


1. Install the holding-down clamps and sample aspiration cannulas in the cannula holder.

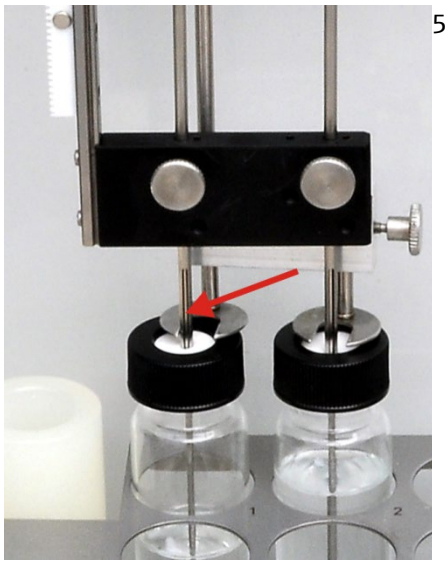
   ATTENTION! The cannula can bend! Release the fastening screws of the cannula before performing the adjustment. Clamp the cannulas into the holder such that the cannula tip does not get immersed in the sample tube.

   The figure shows the installation of 2 cannulas for NPOC measurements with parallel purging.

2. Select the INSTRUMENT ▸ SAMPLER ALIGNMENT menu command to open the window with the same name.
3. In the PLEASE SELECT POSITION NEEDING ADJUSTMENT group, select RINSE POSITION or POSITION 1 from the list field.




4. Increase or reduce the x, y and z values to align the rinse position or position 1:
   - x direction: forward or backward movement
   - y direction: left or right movement
   - z direction: up or down movement



5. Adjust position 1.
   - To adjust the x and y positions, remove the sample tube and move to the position. Place the stirrer onto the sample tray in this position. If it is at the center of the position, the position is correctly adjusted.

   The y value may not be smaller than 33 mm to ensure correct operation.
   - To adjust the z position, place the sample tube with screw closure and septum cap (e.g., EPA sample tube) into the sample tray.

   Adjust the special needle in the z direction until approx. 2 mm of the ventilation slot are visible above the septum.
6. The ventilation slot must be above and below the septum, otherwise the pressure compensation within the sample tube cannot be guaranteed.
7. Adjust the rinse position.

   Adjust the x and y positions until the cannula is at the center of the wash cup.

   In the z direction the special cannula may only dip low enough for the ventilation slot to be visible at the top edge of the wash cup.
8. After each change of the x/y/z direction, click the [RINSE POSITION ADJUST] or [POSITION 1 ADJUST] button.
   The autosampler will then move to the new coordinates. Repeat this step until the cannula position is optimal.

9. Click [SAVE].
   ✓ The adjustment values will be taken over.
10. Open the ALIGNMENT SAMPLER window again and move to the selected position or any measuring position to check the alignment.

## 7.3 Replace the water traps

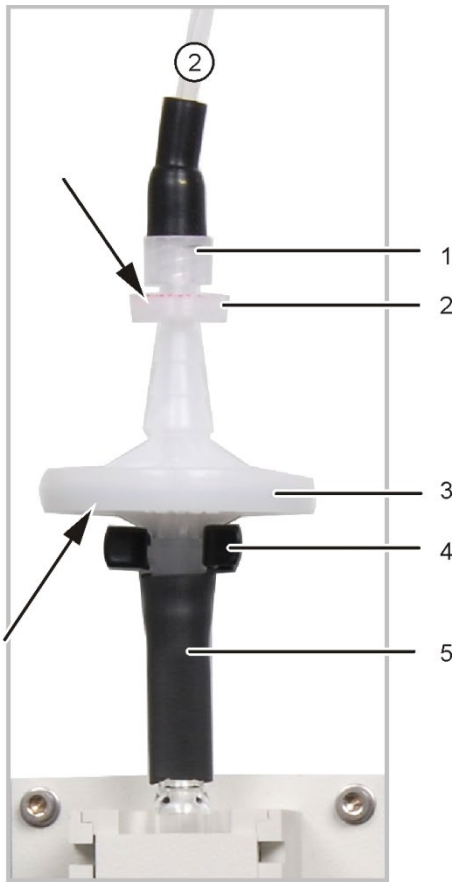
Replace the water traps dependent on the sample matrix, but no later than after 6 months.

Water traps on the front

### Attention

The water traps on the front (TC Pre-filter and disposable retention filter) can be replaced in the switched-on state but not during a measurement. Always replace both water traps!

The water traps only serve their function if they are inserted in the order and installation direction specified!




1 Screw connection
2 Disposable retention filter
3 Aerosol trap
4 Clamp
5 Hose connection

1. Open the doors of the analyzer.
2. Undo the connection (1) to the halogen trap with a single turn.
3. Pull the water trap out of the hose (5) at the TIC container.
4. Assemble the new water traps.

   The label "INLET" on the large water trap (aerosol trap) must point down and the label of the small water trap (disposable retention filter) must point up (arrows in fig. right)
5. Attach the large water trap to the hose at the TIC container (5).
6. Press the water traps into the clamp (4) on the equipment backplate
7. Screw hose no. 2 to the halogen trap to the connection of the small water trap finger-tight.
8. Check the system for leaks (see section "Inspecting the system for leaks" p. 116).
9. Close the front doors.

Water traps at the gasbox

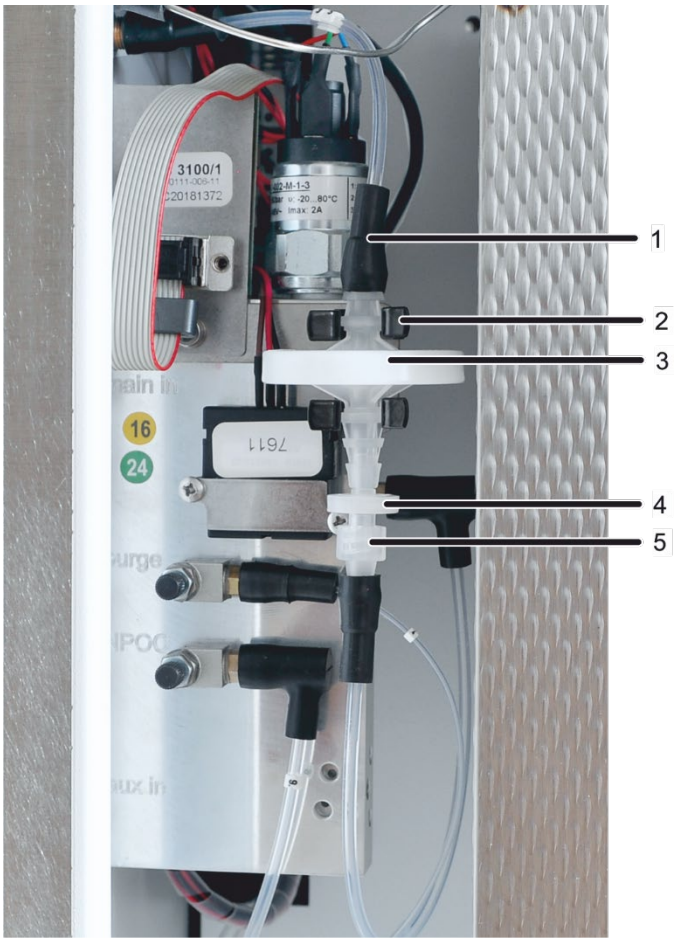
There are two water traps (TC pre-filter and disposable retention filter) installed between the gasbox and the furnace. These two traps protect the gasbox from aerosols or rising water in case of incorrect gas pressures. The left side panel of the analyzer must be opened to replace the water traps.

**WARNING**

Lethal voltages may occur on the inside of the device! Before opening the left side panel, press the main switch to turn off the analyzer and remove the mains plug from the power outlet!

**CAUTION**

There is a risk of burning! The furnace remains hot after switching off the analyzer! Allow the analyzer to cool down for 30 minutes before starting any maintenance work.




1 FAST connector
2 Clamp on the gas box
3 Prefilter (aerosol trap)
4 Disposable retention filter
5 Luer fitting

**Fig. 54 Water traps at the gasbox, left side panel opened**

1. Exit the control and analysis software multiWin.
2. Press the power switch to turn off the analyzer and remove the mains plug from the power outlet.
3. Remove the left side panel from the analyzer:

   Loosen the four fastening screws. Slide the side panel slightly upwards to remove it. Disconnect the grounding conductor connection and put the side panel safely aside.

1. Pull the water traps off the clamp on the gas box (2 in Fig. 54).

2. Pull the FAST connector (1) off the large water trap.
3. Unscrew the Luer fitting (5) from the small water trap.
4. Assemble the new water traps.

   The label "INLET" on the large water trap (aerosol trap) must face upwards.

   The label of the small water trap (disposable retention filter) must be directed downwards.
5. Connect the FAST connector (1) o the large water trap.
6. Screw the Luer fitting (5) to the small water trap.
7. Press the water traps into the clamps on the gasbox.
8. Reinstall the left side panel. Remember to connect the grounding conductor to the side panel.
9. Insert the mains plug into the power outlet and press the main switch to turn on the analyzer.
10. Check the system for leaks (see section "Inspecting the system for leaks" p. 116).

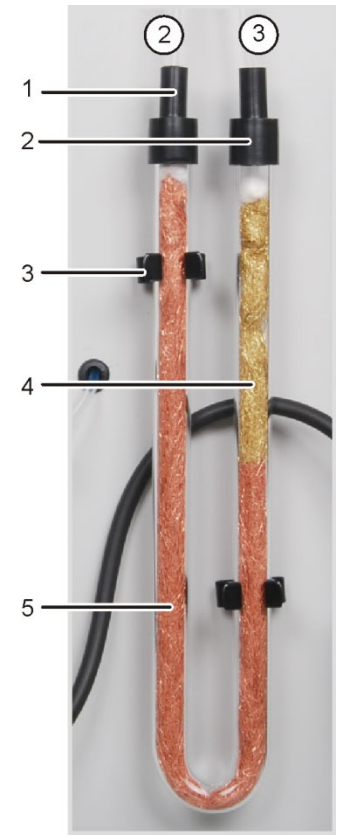
## 7.4 Replacing the halogen trap

**Attention**

Damage to optical and electronic components (detectors, flow sensors) from aggressive combustion products if the copper wool in the halogen trap is used up!

Replace the complete filling of the halogen trap as soon as half of the copper wool is discolored black or the brass wool is discolored!

The analyzer can remain switched on to replace the used copper and brass wool. Replace the halogen trap as follows:




1 FAST connector no. 2 to water trap
2 FAST connector no. 3 to detector
3 Clamp
4 Brass wool
5 Copper wool

1. Open the doors of the analyzer.
2. Pull the FAST connectors (1 & 2) off the halogen trap and pull the U tube out of the clamps (3).
3. Pull the used copper and brass wool out of the U tube using tweezers or a small hook.
4. Check the U tube for cracks.

   Only re-use a completely faultless U-tube!
5. If necessary, rinse the U tube with ultrapure water and allow it to dry fully.
6. Fill the U tube with new copper and brass wool using tweezers or a small hook.

   Replace the total content of the U-tube. When filling the halogen tarp make sure that the copper and brass wool is not stuffed too tightly and there are no larger cavities in the U tube.
7. Cover the copper and brass wool with cotton wool.
8. Press the filled U tube carefully into the clamps.
9. Connect hose no. 2 to the gas inlet branch with copper wool and hose no. 3 to the gas outlet branch with brass wool.
10. Check the system for leaks (see section "Inspecting the system for leaks" p. 116).
11. Close the doors of the analyzer.

## 7.5 Replace catalyst

### 7.5.1 Catalyst service life

If the effectiveness of the catalyst decreases, the combustion tube has to be refilled. An inspection is due after expiry of the maintenance interval (max. 1500 injections). The expiry of the maintenance interval is displayed in multiWin by a message.

### 7.5.2 Removing the combustion tube

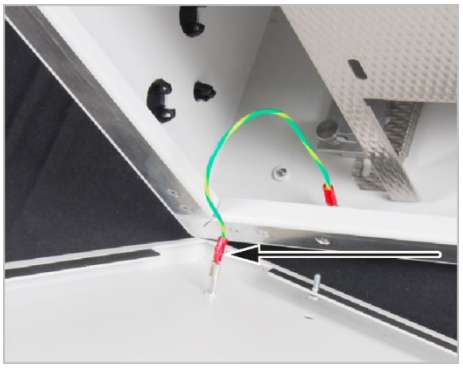
**CAUTION**

There is a risk of burns at the combustion furnace! Only remove the combustion tube when the device is cold or allow the device to cool down sufficiently!

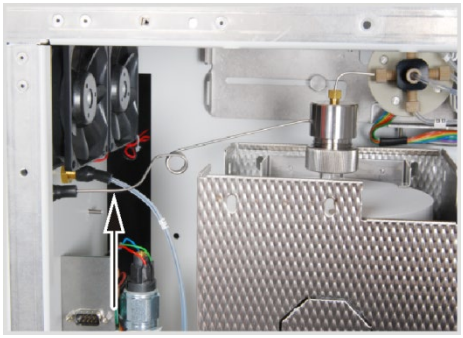
Before switching off set the furnace temperature in multiWin to 20 °C and exit multiWin. Otherwise there is a risk of burns when checking the system for leaks after installation!

Remove the combustion tube as follows:

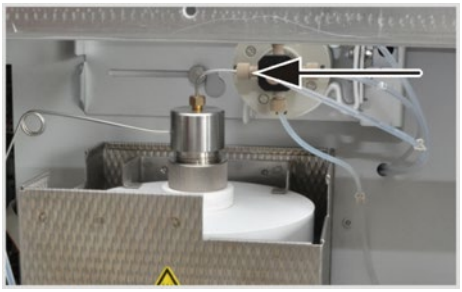
1. Switch off the analyzer from the main switch, remove the mains plug from the mains socket and switch off the gas supply.



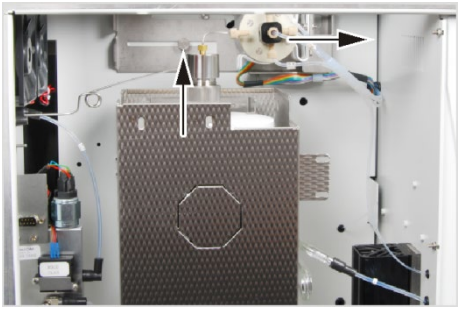
2. Remove the top cover.
   Open the left side wall of the analyzer. Unscrew the four fastening screws; the screws are undetachable and remain in the wall. Disconnect the grounding conductor connection and put the side wall safely aside.



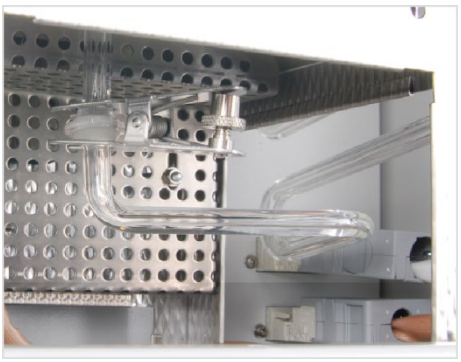
3. Pull the carrier gas cannula out of the FAST connector in the left side wall.



4. Screw the Fingertight connection of the furnace cannula off the change-over valve.



5. Undo the knurled head screw at the holder of the change-over valve.
   Slide the change-over valve to the right. This pulls the furnace cannula out of the change-over valve.



6. Remove the gas clamp below the combustion furnace that connects the outlet of the combustion tube to the condensation coil.

7. Carefully pull the combustion tube upwards out of the combustion furnace.

8. Unscrew the furnace head from the combustion tube and remove the union nut, the pressure ring and the three sealing rings.
9. Remove the used catalyst filling (for notes on disposal see page 193).
10. Check the combustion tube for excessive crystallization, cracks and burst points.

    Only reuse faultless combustion tubes.
11. Rinse the empty combustion tube thoroughly with ultrapure water and allow it to dry fully.

### 7.5.3 Filling the combustion tube

**Attention**

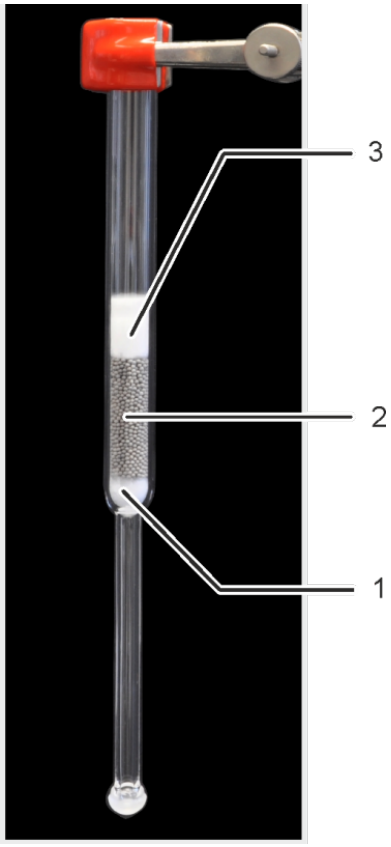
Alkaline salts (hand perspiration) cause crystallizations in the quartz glass when heating the combustion furnace. This shortens the lifetime of the combustion tube.

Where possible do not touch the cleaned combustion tube with your hands for filling. Wear protective gloves to fill the combustion tube.

Only fill fully dried combustion tubes. Dry the combustion tube before filling, if necessary.

Wipe any finger marks on the combustion tube with a cloth wetted with pure alcohol.

Filling the combustion tube for conventional samples




1 Quartz wool, approx. 1 cm

You can secure the combustion tube in a tripod for filling. Fill the combustion tube in accordance with the following instruction from the bottom up:

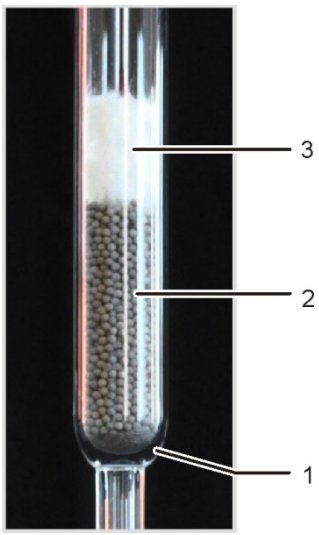
1. Fill the quartz glass wool approx. 1 cm high into the combustion tube; carefully push it down with a glass rod and press it tight.
   Do not apply too much pressure during compaction! The quartz glass wool is used to hold back the catalyst. Make sure that no catalyst can enter the subsequent gas path.
2. Carefully place the platinum catalyst onto the quartz wool (~ 4 cm high).
3. Roll up the high temperature fiber mat (HT mat) of the narrow side. The roll must have a diameter of about 13 mm and a height of 2 cm, so it can easily slide into the combustion tube.
   Insert the rolled HT mat in the combustion tube and slide the HT mat with a glass rod far enough that the

2 Platinum catalyst, approx. 4 cm
3 rolled-up HT mat, approx. 2 cm high

catalyst is covered. Press the mat only slightly onto the catalyst.

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

## Filling the combustion tube for samples with high salt load




1 platinum grid
2 platinum catalyst, approx. 4 cm
3 rolled-up HT mat, approx. 2 cm high

This tube filling should be used for samples with high salt load e.g. sea water, brine, or potassium sulfate extracts of soil.

For samples with high salt load the platinum catalyst is filled on a **platinum** grid.

You can secure the combustion tube in a tripod for filling. Fill the combustion tube in accordance with the following instruction from the bottom up:

1. Put the platinum grid into the combustion tube; carefully push it down with a glass rod.
   The platinum grid is used to hold back the catalyst. Make sure that no catalyst can enter the subsequent gas path.
2. Carefully place the platinum catalyst onto the quartz wool (approx. 4 cm high).
3. Roll up the HT mat from the narrow side. The roll must have a diameter of about 13 mm and a height of 2 cm, so it can easily slide into the combustion tube.

   Insert the rolled HT mat in the combustion tube. Slide the HT mat with a glass rod far enough that the catalyst is covered. Press the mat only slightly onto the catalyst.

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

## 7.5.4 Installing the combustion tube

### Attention

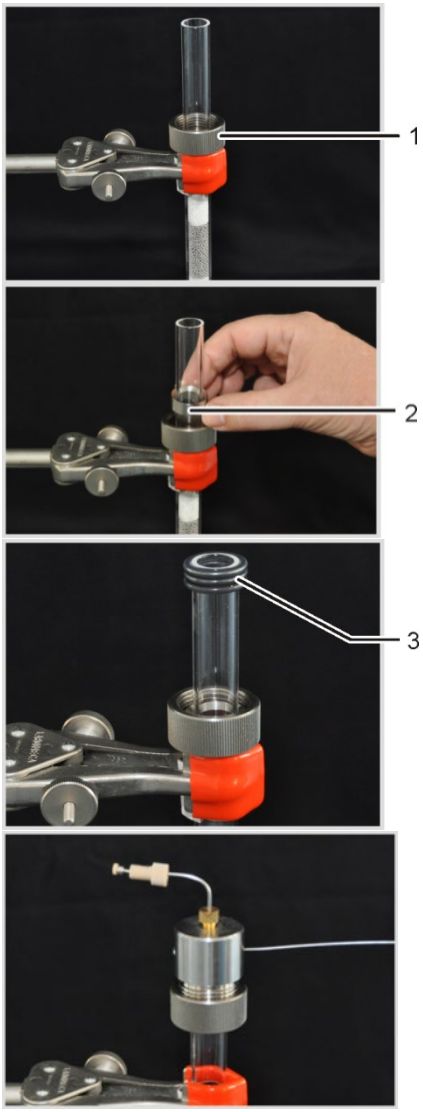
Alkaline salts (hand perspiration) cause crystallizations in the quartz glass when heating the combustion furnace which reduce the service life of the combustion tube.

Where possible do not touch the cleaned combustions tube with your hands. Wear protective gloves to install the lock on the combustion tube.

If necessary, clean the combustion tube externally before installing it in the combustion furnace (e.g. by wiping it with moist cellulose).

It is best to fit the furnace head onto the combustion tube before installing the combustion tube in the combustion furnace.

Install the combustion tube as follows:

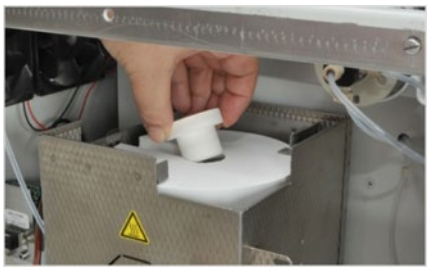



Fit the furnace head to the combustion tube:

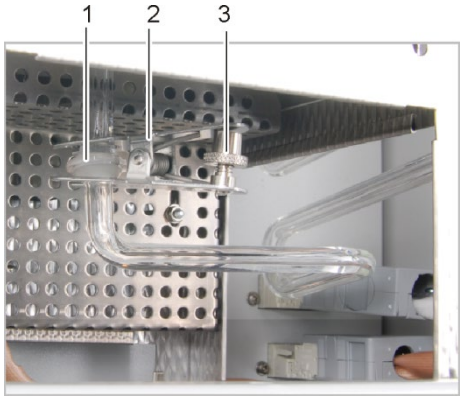
1. Slide the union nut (1) onto the combustion tube.
2. Place the pressure ring (2) into the union nut.

   The conical side of the pressure ring must point up.
3. Slide the three coated sealing rings (3) onto the combustion tube.

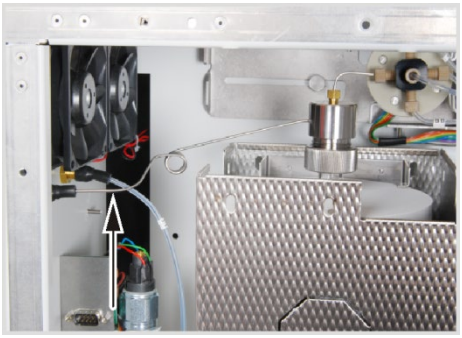
   Make sure that the sealing rings at the edge of the combustion tube are flush.
4. Carefully place the furnace head onto the combustion tube up to the stop; press slightly against it and tighten the union nut finger-tight. The furnace head is fully configured with the furnace cannula and the cannula for the carrier gas.



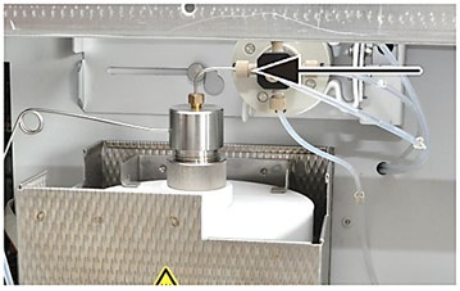
5. Position the ceramic holder in the top opening of the combustion furnace.
6. Insert the combustion tube with the furnace head into the combustion furnace.



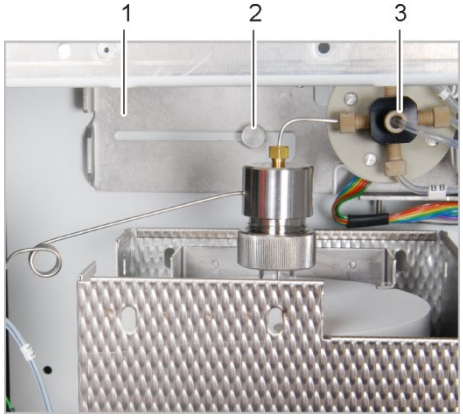

7. Attach the bottom end of the combustion tube and the inlet of the condensation coil (spherical joint (1)).
8. Secure the spherical joint with the fork clamp (2) and tighten the knurled head screw (3) finger-tight.



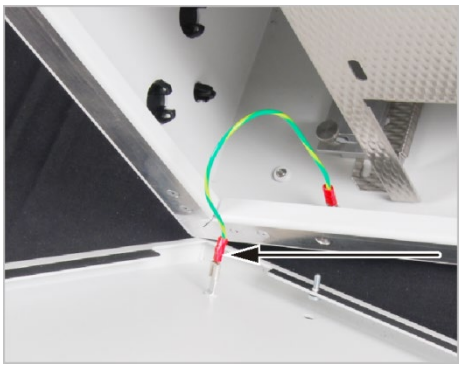
9. Connect the carrier gas connection to the connection in the equipment backplate using the FAST connector.



10. Slide the change-over valve to the left until the change-over valve contacts the connection of the furnace cannula.
11. Screw the furnace cannula with the Fingertight connection finger-tight to the change-over valve.




12. Secure the change-over valve (3) in this position by tightening the knurled head screw (2) at the holder (1) finger-tight.



13. Attach the top cover.
14. Close the side wall.
    Connect the grounding conductor connection to the left side wall.
    First screw the screws into the bottom and then the top side. Tighten the screws in turn.
15. Switch on the gas supply, plug the mains plug into the mains socket and switch on the analyzer from the main switch.
16. Check the system for leaks (see section "Inspecting the system for leaks" p. 116).

### Attention

The catalyst may emit gas during first heating (mist formation in the TIC condensate container). It must therefore be tempered at operating temperature for approx. 30 min. during first heating (until no more mist forms).

During this time remove the water traps from the TIC container to interrupt the gas path to the reactor.

## 7.6 Cleaning the TIC condensate container

### WARNING

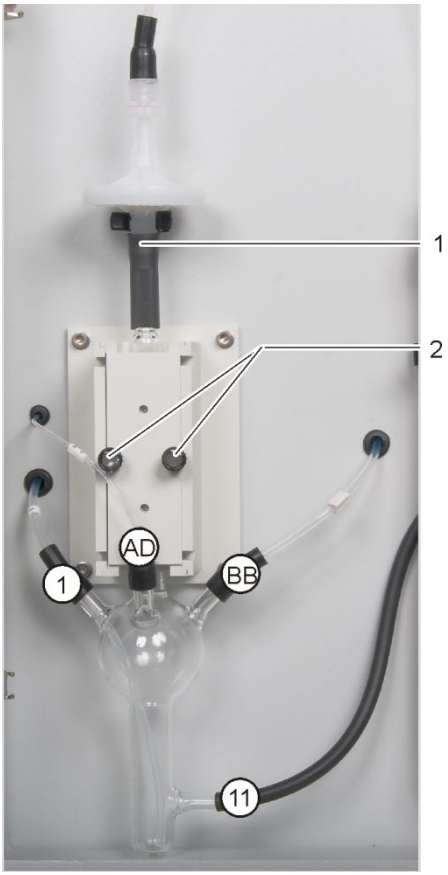
The TIC condensate container contains phosphoric acid! Phosphoric acid irritates the eyes, skin and mucous membranes!

Wear protective gloves and goggles when handling concentrated phosphoric acid! Rinse splashes on the skin immediately with water.

Visually inspect the TIC condensate container regularly for deposits. Cleaning is only required if the purging of the sample is no longer guaranteed.

Remove the TIC condensate container as follows and clean it:

1. Exit the control and analysis software multiWin.
2. Open the doors of the analyzer.




3. Remove connection hose to the water cascades (1) from TIC condensate container.
4. Remove hose No. 1 AD, BB including the quick release connection from the TIC condensate vessel.
5. Disconnect the waste hose no. 11 to from the bottom connection on the TIC condensate vessel.
6. Undo the 2 knurled head screws (2) at the cover of the cooling block and remove the cover and take out the TIC condensate vessel.
7. Pull the quick-release connections off the connectors of the TIC condensate container.
8. Check the TIC condensate vessel for deposits and cracks.
9. If required, rinse the TIC condensate vessel with ultrapure water.
10. Attach the hoses as shown in the adjacent figure:

- Insert waste hose No. 11 at least 1 cm into the lower side connection on the TIC condensate vessel.
- Insert hose No. 1 AD and BB including quick release connector to the connection of the TIC condensate vessel.
  Quick-release connector needs to be inserted at least 1 cm onto the glass support on the TIC condensate vessel.
- Push hose no. 1 almost to the bottom of the TIC condensate container.

## 7.7 Removing and installing the combustion furnace

### 7.7.1 Removing the combustion furnace

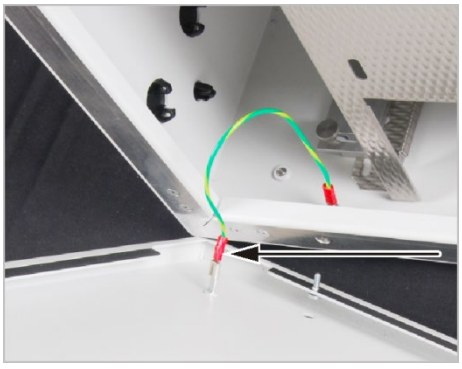
**CAUTION**

There is a risk of burns at the combustion furnace! Only remove the combustion tube when the device is cold or allow the device to cool down sufficiently!

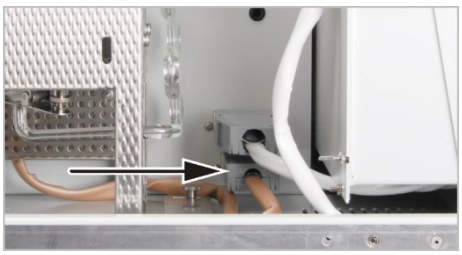
Before switching off set the furnace temperature in multiWin to 20 °C and exit multiWin. Otherwise there is a risk of burns when checking the system for leaks after installation!

Remove the combustion furnace as follows:

1. Switch off the analyzer from the main switch, remove the mains plug from the mains socket and switch off the gas supply.
2. Remove the top cover.



3. Open the left side wall of the analyzer. Unscrew the four fastening screws; the screws are undetachable and remain in the wall.
   Disconnect the grounding conductor connection and put the side wall safely aside.
4. Remove the combustion tube (see section "Removing the combustion tube" p. 99). Slide the change-over valve to the right to prevent it from obstructing the removal.
5. Remove the condensation coil (see section "Removing and cleaning the condensation coil" p. 110).

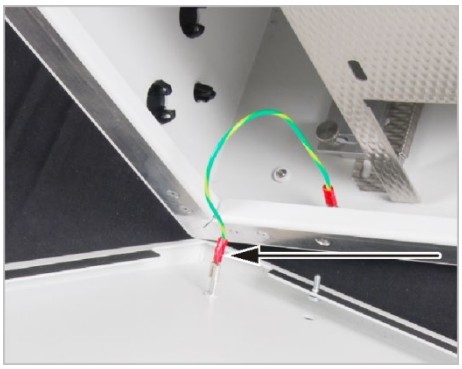


6. Pull the plug-in connector for the combustion furnace out of its socket.

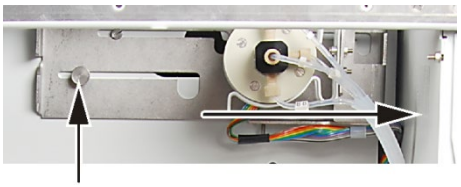
7. Undo the knurled head screws at the retention plates of the furnace at the device floor.
   Slide the plates to the outside.
8. Lift the combustion furnace out of the analyzer.

### 7.7.2 Installing the combustion furnace

Install the combustion furnace as follows:

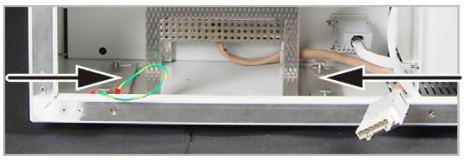


1. Remove the top cover.
2. Open the left side wall of the analyzer. Unscrew the four fastening screws; the screws are undetachable and remain in the wall.
   Disconnect the grounding conductor connection and put the side wall safely aside.

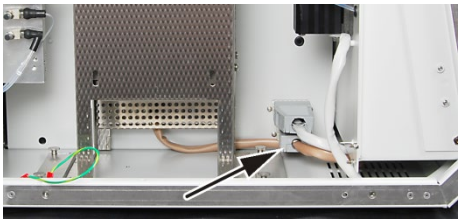


3. Undo the knurled head screw at the holder of the change-over valve and slide the change-over valve to the right.

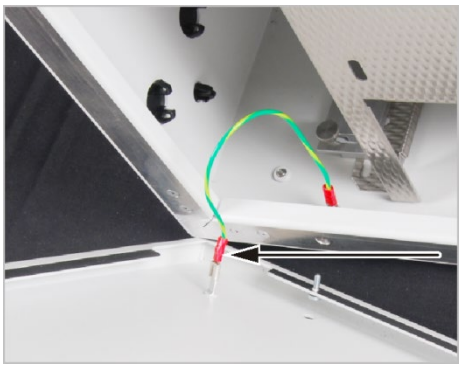
4. Undo the knurled head screws at the retention plates on the floor of the analyzer and slide the retention plate outwards.



5. Place the furnace centered between the retention plates and align its front parallel to the equipment wall.
   Slide the retention plates over the furnace feet and tighten the knurled head screws finger-tight.



6. Plug the plug-in connector for the combustion furnace into the socket at the bottom right of the rear equipment wall.
7. Insert the combustion tube (see section "Filling the combustion tube" p. 102).
8. Install the condensation coil (see section "Removing and cleaning the condensation coil" S. 110).



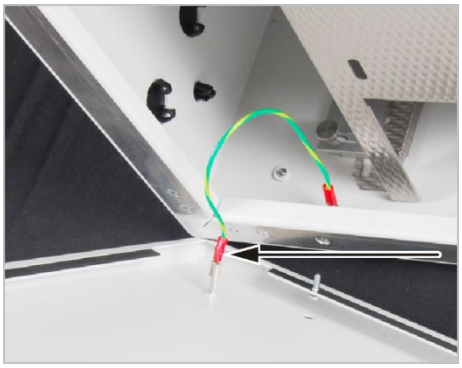
9. Push the sample intake hose and the purging hose through the top aperture. Attach the cover.
10. Close the side wall.
    Connect the grounding conductor connection to the left side wall.
    First screw the screws into the bottom and then the top side. Tighten the screws in turn.
11. Switch on the gas supply, plug the mains plug into the mains socket and switch on the analyzer from the main switch.
12. Check the system for leaks (see section "Inspecting the system for leaks" p. 116).

## 7.8 Removing and installing the condensation coil

### 7.8.1 Removing and cleaning the condensation coil

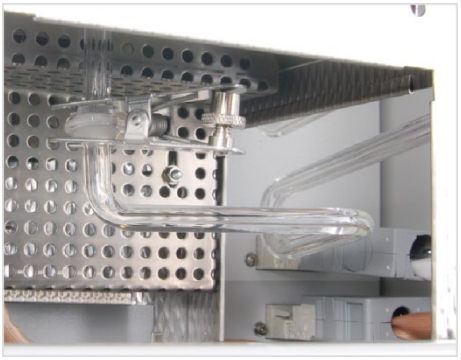
Remove the condensation coil as follows:

1. Switch off the analyzer from the main switch, remove the mains plug from the mains socket and switch off the gas supply.



2. Open the left side wall of the analyzer.
   Unscrew the four fastening screws; the screws are undetachable and remain in the wall.
   Disconnect the grounding conductor connection and put the side wall safely aside.

3. Pull hose 1 off the quick-release connector of the condensation coil.
4. Then pull the quick-release connector off the glass adapter of the condensation coil.

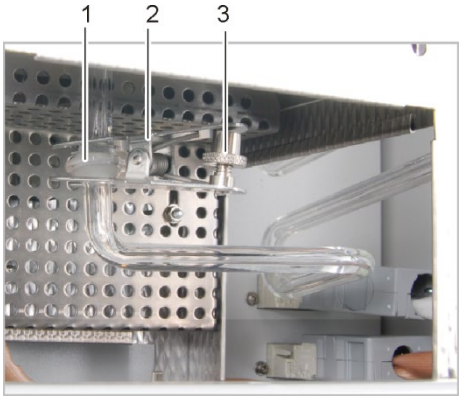


5. Undo the knurled screw on the fork type clamp and remove the fork type clamp that connects the exit of the combustion tube with the condensation coil.
6. Disconnect the spherical joint and allow the condensation coil to slide down by approx. 1 cm.
7. Carefully pull the bottom portion of the condensation coil out of the cut-out in the combustion furnace.
8. Inspect the condensation coil for deposits and cracks.
9. If necessary, rinse the condensation coil with ultrapure water and allow it to dry.

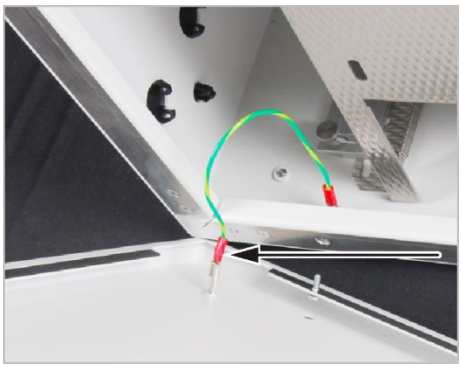
### 7.8.2 Inserting the condensation coil

Insert the condensation coil as follows:

1. Push hose 1 into the quick-release connector.
2. Then push the quick-release connector onto the adapter of the condensation coil.
3. Hold the condensation coil against the right-hand side of the combustion furnace. The spherical joint of the coil protrudes into the lower cut-out of the furnace.




4. Attach the bottom end of the combustion tube and the inlet of the condensation coil (spherical joint (1)).
5. Secure the spherical joint with the fork clamp (2) and tighten the knurled head screw (3) finger-tight.



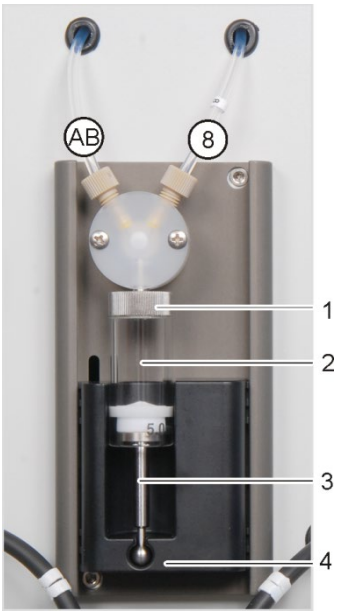
6. Close the side wall.
   Connect the grounding conductor connection to the left side wall.
   First screw the screws into the bottom and then the top side. Tighten the screws in turn.

7. Switch on the gas supply, plug the mains plug into the mains socket and switch on the analyzer from the main switch.
8. Check the system for leaks (see section "Inspecting the system for leaks" p. 116).

## 7.9 Cleaning and replacing the metering syringe

Replace or clean the metering syringe as follows:

1. Open the doors of the analyzer.




2. Unscrew the metering syringe from the valve (1) and remove it from the drive (4).
3. Dismantle and clean the glass cylinder (2) and piston (3).
4. Insert the piston rod of the new metering syringe into the drive.
5. Screw the glass cylinder to the valve.

## 7.10 Removing and replacing the pump hose

**CAUTION**

The pump hose contains phosphoric acid! Phosphoric acid irritates the eyes, skin and mucous membranes!

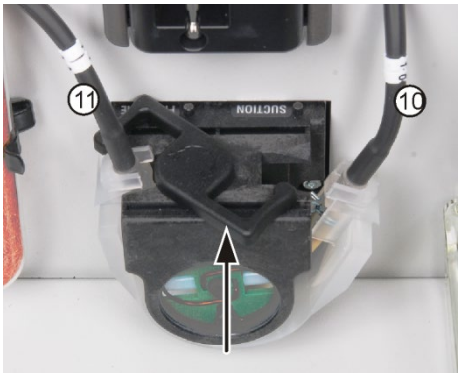
Wear protective gloves and goggles when handling concentrated phosphoric acid! Rinse splashes on the skin immediately with water.

Inspect the pump hoses of the condensate pump and the phosphoric acid pump every 3 months or after every catalyst replacement for leaks.

Condensate pump

Remove the pump hose of the condensate pump as follows and inspect it for leaks:

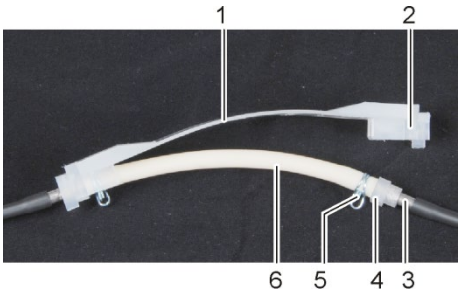
1. Open the doors of the analyzer.




2. Press the bracket at the condensate pump to the left.
3. Pull the hoses no. 10 and no. 11 off the connections.
4. Remove the conveyor belt with the pump hose off the pump body.
5. Inspect the pump hose and connections for excessive wear and cracks.

   If moisture escapes from the pump hose or the connections, the pump hose must be replaced.
6. Wipe the pump body and roller carrier.
7. Inspect the pump body and the roller carrier for wear.

   If the pump body and roller carrier are heavily corroded, please contact Service.



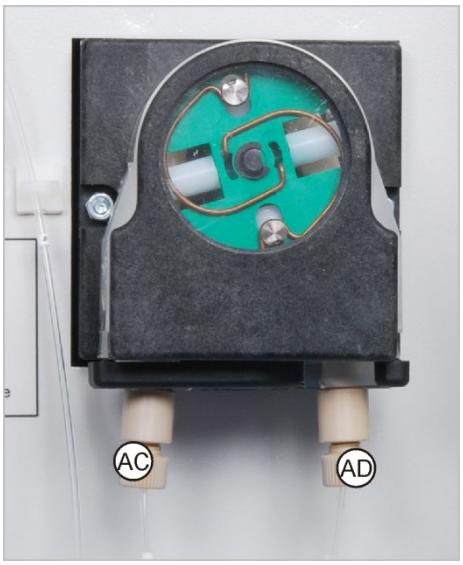

| | | | |
|---|---|---|---|
| 1 | Conveyor belt | 4 | Tube guide |
| 2 | Groove | 5 | Hose clamp |
| 3 | Metal adapter | 6 | Pump hose |

8. Press the intact or new pump hose into the conveyor belt.

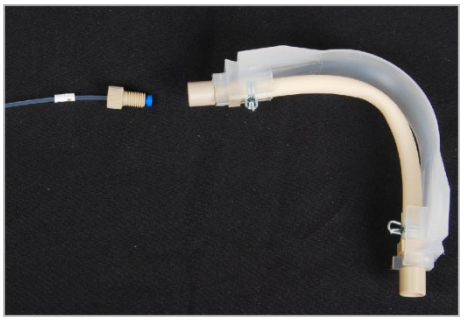
   The hose clamps have to face downwards during installation. Push the hose guide into the groove of the conveyor belt.


9. Place the conveyor belt around the pump body.
10. Press the conveyor belt upwards with one hand and rotate the bracket with the other hand to the right until it engages.
11. Push hose **10** and hose **11** back onto their adapters.
12. Check the system for leaks (see section "Inspecting the system for leaks" p. 116).

Phosphoric acid pump




Remove the pump hose analog to the removal for the condensate pump and inspect it for leaks.

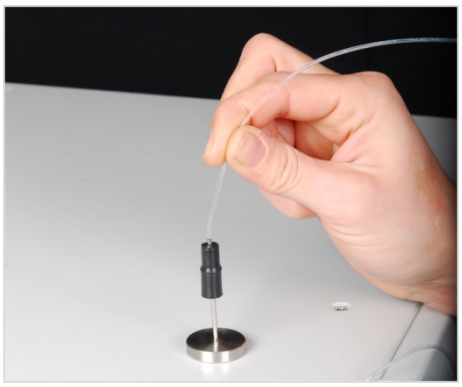


The hoses **AC** and **AD** are connected to the pump using Fingertight connections. Unscrew the connections from the connectors during removal and tighten them again after installing the pump hose.

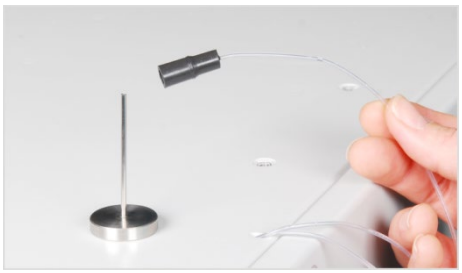
## 7.11 Replacing the hose connections

The analyzer uses mainly FAST connectors to connect the hoses to the glass components. Use the threading aid to feed think hoses into the FAST connectors. It is included with the analyzer tools.

1. Slide the FAST connector onto the cannula of the threading aid. The narrow hole of the connector points upwards.

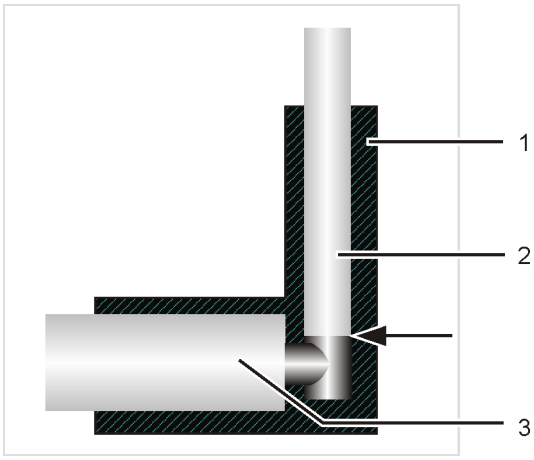


2. Thread the hose into the cannula of the threading aid.



3. Slide the FAST connector from the cannula onto the hose.
4. Pull the hose off the cannula.
5. Pull the hose far out of the FAST connector until it no longer reaches into the wider hole.

For angled FAST connectors make sure not to slide the hose ends beyond the branch length of the connector in order to guarantee an unimpeded gas flow.

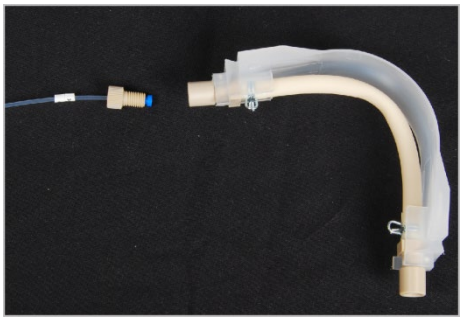



1 angled FAST connector
2 hose
3 connection adapter

**Fig. 55 Angled FAST connector with connected hose**

Fingertight screw connections are e.g. found at the cannula and the syringe pump.

When replacing damaged Fingertight screw connections only use straight cut, round and unpinched hose ends for the connection. Slide the conical nipple with the conical side towards the banjo bolt onto the hose. The conical nipple and hose end must be flush.



1 conical nipple
2 banjo bolt
3 Hose type

**Fig. 56 Replacing the Fingertight connection**

## 7.12 Inspecting the system for leaks

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

1. Switch on the analyzer multi N/C 3100.
2. Open the carrier gas supply at the pressure reducer.
3. Start the control and analysis software multiWin.
4. Check the flow indication in the window System-Status:
   - In (inlet flow): 160 ml/min
   - Out (outlet flow): 160 ml/min (± 10 ml/min)

**CAUTION**

If the outlet flow is significantly below the inlet flow of 160 ml/min, all connection points must be reinspected.

## 7.13 Servicing the POC module

### 7.13.1 Checking the operation of the adsorber

The adsorber must be checked at least once a month. A shorter test interval is indicated for high inorganic carbon content.

1. Prepare a TIC mixed standard from carbonate/hydrocarbonate with a concentration of 100 mg/l.
2. Load a POC method. Measure the carbonate/hydrocarbonate standard solution.
3. If the result is greater than 0.1 mg/l, the adsorber is exhausted. In this case replace the adsorber.

### 7.13.2 Inspecting the POC module for leaks

1. Switch on the multi N/C 3100.
2. Open the gas supply at the pressure reducer.
3. Start the multiWin software.
4. Check the flow indication in the window SYSTEM-STATUS:
   - In (inlet flow): 160 ml/min
   - Out (outlet flow): 160 ml/min (± 10 ml/min)
5. With the menu command INSTRUMENT ▸ COMPONENT TEST open the window COMPONENT TEST ▸ tab VALVES.
6. Switch on valve 3 and check the flow indication in the window SYSTEM-STATUS:
   - In (inlet flow): 160 ml/min , Out (outlet flow): 160 ml/min (± 10 ml/min)

   ATTENTION! Check all connections and septums if the flow is significantly below 160 ml/min.
7. Close the window COMPONENT TEST.

# 8 Fault Removal

## 8.1 General information

The following chapter describes a number of possible problems that the user can partially remedy independently. If such problems occur frequently, the Service department of Analytik Jena GmbH must always be informed.

As soon as the multi N/C 3100 is switched on, system monitoring takes place. Any errors occurring are displayed in a window after start-up. Starting a measurement is not possible.

The user must acknowledge the error messages by clicking the button [OK]. Next a message text opens in the main window and possibly the button [INITIALIZE ANALYZER].

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

For fault analysis it is possible to record log files. The recording of log files should be enabled after consultation with the Analytik Jena GmbH service department for specific faults. The log files are stored in the directory ...\multiWin\LOG.

The following files can be generated and saved:

- multiWin_LOG.*:

Log file for error messages, always generated automatically

- multiWin_ADU.*:

Log file to monitor the NDIR detector, generated automatically

- With the menu command INSTRUMENT ▸ COMPONENT TEST open the window COMPONENT TEST ▸ tab OPTICAL BENCH and enable the option SAVE VALUES with (✓).

### Attention

If the errors below cannot be remedied using the corresponding fault removal notes, the Service department of Analytik Jena GmbH must always be informed. This also applies for the repeated occurrence of individual faults.

- For fault diagnosis the complete directory multiWin\LOG must be emailed to the Service department of Analytik Jena GmbH (see Service address on the inside front cover).
- To copy the directory multiWin\LOG use the command COPY ..\MULTIWIN\LOG*.* in the menu INSTRUMENT / SYSTEM PARAMETERS / tab ERROR ANALYSIS.

## 8.2 Error messages in multiWin

| 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 program |
| VERS1 | **Communication error – analyzer** | |
| | **Cause** | **Remedy** |
| | analyzer not switched on | switch on analyzer |
| | multiWin started too early | only start multiWin after 30 sec |
| | analyzer not connected to PC | check connection between analyzer and PC |
| | incorrect COM port set at the external computer | check set interface at the external computer, if necessary select different interface in multiWin with menu command CONFIGURATION ▸ INTERFACE |
| -6 | **Analyzer is busy** | |
| | **Cause** | **Remedy** |
| | analyzer > 10 min in the busy state | initialize analyzer |
| -5 | **communication error – analyzer STAT, MESS, STEP or INIT** | |
| | **Cause** | **Remedy** |
| | communication error | initialize analyzer |
| -4 | **Communication error – analyzer** | |
| | **Cause** | **Remedy** |
| | communication error | check interface cable<br>initialize analyzer |
| -3 | **command from the analyzer CRC error CRC error invalid command from the analyzer** | |
| -2<br>-1 | **Cause** | Remedy |
| | communication error | initialize analyzer |
| 1<br>2<br>3<br>4<br>5<br>6 | **Incomplete command from the PC**<br>**PC command without STX**<br>**PC command without ***<br>**PC command CRC error**<br>**PC command invalid command**<br>**PC command invalid MESS command** | |
| | **Cause** | **Remedy** |
| | faulty connection between internal and external program | initialize analyzer |

| Error code | Error message | |
|---|---|---|
| 7<br>8<br>9 | **COM 2 not found**<br>**COM 3 not found**<br>**COM 4 not found** | |
| | **Cause** | **Remedy** |
| | problems with internal hardware | switch analyzer off/on |
| 10 | **Gas pressure error** | |
| | **Cause** | Remedy |
| | counterpressure in the analyzer system too great: carrier gas supply is automatically interrupted to protect the analyzer; flow indication MFC approx. 0 ml/min | search for and replace component causing the gas pressure error |
| | water trap clogged | undo connection upstream of the water traps and reinitialize the analyzer<br>check if gas pressure error occurs again, if not, replace water traps |
| | no gas flow at the measuring outlet (gas supply hose for sample gas supply kinked) | check gas supply hose, remove kink if necessary |
| | condensation coil clogged with catalyst balls | interrupt measuring gas flow between the combustion tube and condensation coil ⇒ check if "gas pressure error" occurs again, if not - rinse condensation coil clear with ultrapure water<br>during catalyst replacement always make sure that sufficient quartz glass wool is filled as a first layer |
| | combustion tube "salidified" (by analysis of highly saline samples accumulation of salt in the combustion tube)<br>HT mat used up by analysis of highly saline samples | replace HT mat in the combustion tube or replace catalyst (dependent on the number of measurements with the current catalyst filling and activity of the catalyst) |
| | gas supply to the furnace head clogged | clean gas supply to the furnace head |
| 11 | **Change-over valve timing error** | |
| | **Cause** | **Remedy** |
| | change-over valve does not rotate<br>change-over valve does not stop rotating | initialize analyzer<br>check individual valve positions in multiWin with menu command INSTRUMENT / COMPONENT TEST on tab VALVES |
| 12 | **Incorrect version number** | |
| | **Cause** | **Remedy** |
| | the versions of multiWin and the software of the internal computer do not match | update as appropriate |
| 13 | **No connection to sampler** | |
| | **Cause** | **Remedy** |
| | sample not switched on<br>connection cable not connected or faulty | switch on sampler and initialize analyzer<br>check connection cable |

| Error code | Error message | |
|---|---|---|
| 15 | **no $O_2$ pressure at HT furnace** | |
| | **Cause** | **Remedy** |
| | Carrier gas connection not present or faulty | connect carrier gas to HT furnace (check 4 – 6 bar preliminary pressure) |
| 20<br>21<br>22<br>26 | **no connection to optics (NDIR)**<br>**CRC error optics**<br>**status error optics**<br>**optics error; incorrect command return** | |
| | **Cause** | **Remedy** |
| | communication error | initialize analyzer |
| | NDIR detector faulty | inform Service |
| 24 | **Optics error, analog values outside of range** | |
| | **Cause** | **Remedy** |
| | the analog values of the detector are outside the working range | check the quality of the carrier gas<br>initialize analyzer and check analog values via component test |
| 27 | **Optics error, analog values outside of range** | |
| | **Cause** | **Remedy** |
| | the analog values of the detector are outside the working range | check quality of the carrier gas<br>for solids methods and connection of HT 1300 carrier gas flow > intake flow<br>initialize analyzer and check analog values via component test |
| 30 | **No connection to N sensor** | |
| | **Cause** | **Remedy** |
| | CLD is not switched on<br>connection cable not connected or faulty<br>incorrect connection | switch on CLD<br>check connection cable<br>check connection |
| 40 | **no connection to the syringe pump** | |
| | **Cause** | **Remedy** |
| | no communication between analyzer and syringe pump | initialize analyzer<br>switch off PC, switch back on and initialize analyzer |
| 80 | **No connection to temperature controller** | |
| | **Cause** | **Remedy** |
| | no connection to the solids module<br>not switched on<br>incorrect connection | switch on solids module<br>check connection cable<br>check connection |
| 81 | **Thermocouple HT furnace interruption** | |
| | **Cause** | **Remedy** |
| | faulty thermocouple | inform Service |

| Error code | Error message | |
|---|---|---|
| 84 | **Communication error HT furnace temperature controller** | |
| | **Cause** | **Remedy** |
| | communication error | inform Service |
| 86 | **No external furnace found** | |
| | **Cause** | **Remedy** |
| | no connection to the solids module | check connection cable |
| 111 | **Rotator error** | |
| | **Cause** | **Remedy** |
| | Drive incorrectly positioned, e.g. jammed<br>Drive faulty | initialize analyzer<br>if the error cannot be corrected, contact Service |
| 112 | **Swivel drive error** | |
| | **Cause** | **Remedy** |
| | Drive incorrectly positioned, e.g. jammed<br>Drive faulty | initialize analyzer<br>if the error cannot be corrected, contact Service |
| 113 | **Lifting drive error** | |
| | **Cause** | **Remedy** |
| | Drive incorrectly positioned, e.g. jammed<br>Drive faulty | Initialize analyzer<br>If the error cannot be corrected, contact Service |
| 114 | **Rack detection error** | |
| | **Cause** | **Remedy** |
| | Sample tray not positioned correctly | position the sample tray again and make sure it clicks into place<br>initialize analyzer |
| 115 | **Wrong rack** | |
| | **Cause** | **Remedy** |
| | Wrong sample tray set in the software | check settings in the software (see section 5.1); if necessary, set a different sample tray |
| 116 | **Unknown sampler command** | |
| | **Cause** | **Remedy** |
| | Communication error | contact Service |
| 200 | **Restart computer in the analyzer** | |
| | **Cause** | **Remedy** |
| | internal computer reset<br>overvoltage<br>short-term power failure | if the front LED indication (Lockin) illuminates, initialize the analyzer<br>for repeated occurrence monitor precisely at which time the error occurs (note status line) |
| 201 | **Restart the internal program** | |
| | **Cause** | **Remedy** |
| | internal program error | initialize analyzer<br>for repeated occurrence monitor precisely at which time the error occurs (note status line) |

| Error code | Error message | |
|---|---|---|
| 202<br>203 | **File method.txt not found**<br>**File init.cnf not found** | |
| | **Cause** | **Remedy** |
| | program error | inform Service |
| 401<br>402<br>403<br>404 | **Syringe pump: Initialization**<br>**syringe pump: invalid command**<br>**syringe pump: invalid operand**<br>**syringe pump: faulty command sequence** | |
| | **Cause** | **Remedy** |
| | communication error | initialize analyzer |
| | syringe pump faulty | inform Service |
| 407 | **Syringe pump: Syringe pump not initialized** | |
| | **Cause** | **Remedy** |
| | analyzer not yet initialized after switching on<br>reset the syringe pump | initialize analyzer |
| 409 | **Syringe pump: pump sluggish** | |
| | **Cause** | **Remedy** |
| | clogging of a hose line (8) or (AB) | search for cause, remedy fault<br>replace hose line – if necessary remove hose line and rinse with ultrapure water, then reinstall<br>initialize analyzer |
| | syringe pump faulty | inform Service |
| 410 | **Syringe pump: valve sluggish** | |
| | **Cause** | **Remedy** |
| | syringe pump faulty<br>valve broken | inform Service |
| 411<br>415 | **Syringe pump: pump step not permitted**<br>**Syringe pump: Invalid command** | |
| | **Cause** | **Remedy** |
| | communication error | initialize analyzer |
| | syringe pump faulty | inform Service |
| MESSx | **Analyzer error: MESSx measurement is cancelled** | |
| | **Cause** | **Remedy** |
| | equipment fault | initialize analyzer<br>check window *System state* for error removal<br>search for equipment fault and remove error |
| | **Peltier temperature outside range** | |
| | **Cause** | **Remedy** |
| | Peltier cooling insufficient | inform Service. After successful repair, a replacement of the water traps is recommended. |

| Error code | Error message | |
|---|---|---|
| | **Minimum sample volume > cup volume** | |
| | **Cause** | **Remedy** |
| | for sample supply with sampler:<br>sample volume selected too large<br>number of detections too high | check configuration in the method:<br>sample volume/rinsing volume<br>adjust number of detections (repeat measurements) to the cup volume |

## 8.3 Status errors – indications in the window System state

Status errors are shown in the window SYSTEM STATE in red or yellow.

| **Error indication** | |
|---|---|
| Flow indication MFC: 160 ml/min<br>Flow indication MFM: < 150 ml/min | |
| **Cause** | **Remedy** |
| ■ union nut at the combustion tube not tightened correctly (after catalyst replacement)<br>■ carrier gas supply to the furnace head not connected properly (after catalyst replacement)<br>■ sealing rings at the combustion tube faulty (severely deformed) or not attached (after catalyst replacement)<br>■ TIC condensate container – FAST connector leaking<br>■ Luer connection at the water trap system loose (after installing water traps, installing halogen trap) | ■ check screw connections for completeness, deformation, tighten if necessary<br>■ check carrier gas supply (FAST connector at the analyzer wall and screw connection at the furnace head)<br>■ check all connection points (water traps) and replace if necessary |
| ■ connection between combustion tube and condensation coil or screw connections leaking | ■ check connection between combustion tube and condensation coil (tight fit of the fork clamp) |
| ■ combustion tube faulty (cracks, fractures at the edge)<br>■ TIC condensate faulty (fractures at the connections) | ■ check glass components, replace if faulty |
| ■ Water traps clogged | ■ replace the water traps |
| ■ condensate pump hose leaking | ■ check condensate pump, replace hose if necessary |
| **Flow indication MFC: 160 ml/min**<br>**Flow indication MFM 1: < 150 ml/min or > 170 ml/min** | |
| **Cause** | **Remedy** |
| ■ MFM (mass flow sensor) faulty | ■ check flow with external mass flow sensor to confirm error if possible<br>■ inform Service |
| ■ halogen trap filling used up | ■ check halogen trap |

| **Flow indication MFC: < 160 ml/min or fluctuating<br>flow indication MFM1 : < 150 ml/min** | |
|---|---|
| **Cause** | **Remedy** |
| ■ no carrier gas<br>■ hose line leaking | ■ turn on carrier gas at the pressure reducer<br>■ search for and remedy leak |
| ■ preliminary pressure at the carrier gas supply too low | ■ set carrier gas preliminary pressure to 4 to 6 bar |
| ■ pressure switch in the analyzer has tripped – simultaneous error message in multiWin "gas pressure error | ■ see gas pressure error (error code 10) on page 119 |
| ■ MFC faulty | ■ inform Service |
| **Flow indication MFC: 160 ml/min<br>Flow indication MFM 1: > 170 ml/min** | |
| **Cause** | **Remedy** |
| ■ Peltier cooling insufficient (simultaneous error message temperature out of range below Peltier temperature) | ■ check from the top at the TIC condensate container whether cooling takes place (formation of condensate at the cooling block indicates that the cooling is working) |
| ■ MFC faulty | ■ inform Service |
| **Flow indication MFC: 0 ml/min<br>Flow indication MFM 1: 0 ml/min** | |
| **Cause** | **Remedy** |
| ■ a hose line is clogged up | ■ replace clogged hose line<br>■ if necessary, remove and rinse clogged hose line, then reinstall |
| ■ no method loaded | ■ load method |
| **Values of the NDIR detector below opt. bank indicated in yellow** | |
| **Cause** | **Remedy** |
| ■ Analogue values of the detector are located at the border of operating range.<br>The ADU values can be viewed in the control and analysis software multiWin with the menu command INSTRUMENT ▸ COMPONENT TEST on the tab NDIR. | ■ check halogen trap and replace if necessary<br>■ consult regarding the application with Analytik Jena GmbH about specific application rules for difficult sample matrix<br>Measurements are still possible, but it should be pointed out to the user that the ADU values of the detector are leaving the optimum range |

The ADU values of the NDIR detector are changing slowly due to normal ageing. If the values change intense within a few analyses, this indicates damage to the detector by components of the analysis gas!

## 8.4 Equipment faults and analytical problems

Other problems not detected by the system monitoring can also occur. Starting a measurement is possible. Such errors are usually detected on the basis of implausible measuring results (analytical problems) or are clearly visible in the equipment technology.

If the suggested solutions are not successful, inform Service.

| Error | |
|---|---|
| **Water traps clogged** | |
| **Cause** | **Remedy** |
| ■ Service life expired (replacement recommended after 6 months, dependent on matrix)<br>■ Measuring of samples with strong aerosol generation | ■ Replacement of the water traps (see section "Replace the water traps" p. 96) |
| **Dispersing measured values** | |
| **Cause** | **Remedy** |
| ■ filling of combustion tube used up | ■ replace catalyst |
| ■ metering faulty | ■ check metering |
| ■ inhomogeneous sample matrix | ■ temper cold samples prior to analysis<br>■ filter samples prior to analysis |
| ■ stirring insufficient | ■ stir particulate samples, for measurements with sampler adjust stirring speed in multiWin under METHOD ▸ EDIT ▸ PROCESS PARAMETERS ▸ STIR |
| ■ sensitive samples | ■ prevent introduction of $CO_2$ or organic vapors from the ambient air<br>■ cover sample cups on sampler with aluminum foil<br>■ for manual measurement introduce gas to the head room of the sample cup<br>■ check ambient conditions<br>■ remove interference |
| **Dispersing measured values** | |
| **Cause** | **Remedy** |
| drift of NDIR base<br>■ unfavorable integration criteria<br>■ measurement was stopped too early: | ■ check configurations<br>■ extend maximum integration time |
| **Cannula faulty** | |
| Cause | **Remedy** |
| ■ Injection cannula corroded during injection due to sample matrix and temperature<br>■ cannula clogged | ■ replace cannula<br>Misting up of the cannula is normal. Replacement is required if the sample is no longer metered as a cohesive beam but is sprayed. |
| **Sample is not drawn in without air bubbles** | |
| **Cause** | **Remedy** |
| ■ leaks in the sample intake path | ■ check connections and tighten any loose connections:<br>cannula - hose<br>hose - syringe pump valve |
| ■ sample intake cannula clogged | ■ remove cannula and clean in ultrasonic bath<br>■ replace cannula |
| ■ metering syringe leaking<br>■ PTFE sealing lips of the plunger are damaged | ■ remove and check metering syringe<br>■ replace metering syringe |

| Error | |
|---|---|
| **Incomplete metering in reactors** | |
| **Cause** | **Remedy** |
| ■ leaks in the metering path | ■ check connections and tighten any loose connections:<br>syringe pump – change-over valve<br>change-over valve – Injection cannula<br>change-over valve – TIC condensate container |
| **Carry-over** | |
| **Cause** | **Remedy** |
| ■ insufficient syringe rinse | ■ rinse the metering syringe with sample before the next injection: under METHOD ▸ EDIT in the tab METHOD enter 3 for rinse cycles for the first measurement, for all other measurements rinsing is generally not required, enter 0 |
| **Low results; all areas** | |
| **Cause** | **Remedy** |
| ■ catalyst used up | ■ replace catalyst |
| ■ system leaking | ■ inspect system for leaks |
| ■ faulty metering | ■ check metering |
| ■ particulate samples not or insufficiently stirred | ■ stir particulate samples |
| **Low results for analyses through combustion (TC, TOC, NPOC, $TN_b$)**<br>**TIC measurements are ok** | |
| **Cause** | **Remedy** |
| ■ catalyst used up | ■ replace catalyst<br>After a catalyst replacement a calibration must be carried out. |
| **Low results for TIC measurements**<br>**Analyses through combustion (TC, TOC, NPOC) are ok** | |
| **Cause** | **Remedy** |
| ■ no phosphoric acid in the reagent bottle for phosphoric acid | ■ refill phosphoric acid |
| ■ faulty metering of the sample | ■ check metering |
| **Low results for $TN_b$** | |
| **Cause** | **Remedy** |
| ■ catalyst used up | ■ replace catalyst |
| ■ measurement outside the calibrated range | ■ observe the calibrated range<br>■ use quadratic calibration<br>■ calibrate dependent on matrix when possible<br>■ when analyzing unknown substances use low concentration where possible (dilute sample if possible) |
| **Unusual peak form (TC and $TN_b$-Measurement)** | |
| **Cause** | **Remedy** |
| ■ catalyst used up | ■ if low results simultaneously, replace catalyst |
| ■ unfavorable integration criteria selected | ■ check integration criteria |

| | |
|---|---|
| ■ exceeding the measuring range for $TN_b$ measurement with CLD (peak height > 1000 ppm NO in the measuring gas) | ■ dilute sample |
| **$TN_b$ measurements with CLD faulty (TC measurements are ok)** | |
| **Cause** | **Remedy** |
| ■ gas connection between multi N/C 3100 and CLD faulty<br>■ ozone generator faulty | ■ check gas connection between multi N/C 3100 and CLD<br>■ inform Service |
| **Condensate pump/phosphoric acid pump leaking** | |
| **Cause** | **Remedy** |
| ■ hose connections leaking<br>■ pump hose faulty | ■ replace pump hose |
| **Control lamps at the analyzer do not illuminate: 5 V, 24 V** | |
| **Cause** | **Remedy** |
| ■ error in the power supply or in the electronics | ■ check the electrical connections<br>■ check the power supply of the lab |
| ■ equipment fuse faulty | ■ inform Service |
| **Front LED display on the analyzer does not illuminate: Lockin** | |
| **Cause** | **Remedy** |
| ■ internal program has not been started | ■ switch analyzer on again (switch off/on from main switch) |
| **Control lamp at the analyzer does not illuminate: Heating** | |
| **Cause** | **Remedy** |
| ■ incorrect temperature configuration in multiWin | ■ check temperature configuration in multiWin under CONFIGURATION ▸ EDIT OPTIONS on the tab ANALYZER COMPONENTS (list field FURNACE TEMPERATURE) |
| ■ faulty thermocouple (furnace)<br>A faulty thermocouple can be detected by an indication in the LED strip in the analyzer | ■ inform Service |
| ■ faulty electronics component | ■ inform Service |
| ■ combustion furnace not connected correctly | ■ check correct contact of the combustion furnace |

# 9 Transport and storage

## 9.1 Transport

### 9.1.1 Preparing the analyzer for transport

**CAUTION**

There is a risk of burns at the combustion furnace! Only remove the combustion furnace when the device is cold or allow the device to cool down sufficiently!

When removing the glass components there is a risk of injury from glass breakage! Remove all glass components carefully from the analyzer!

**Attention**

Unsuitable packaging material and residue of measuring solution and chemicals can damage individual components of the analyzer!

Only transport the analyzer in its original packaging! Ensure that the analyzer is fully drained and all transport locks have been fitted!

The cannulas can bend! Package the cannulas in the original packaging!

Prepare the analyzer for transport as follows:

1. Rinse the phosphoric acid pump and corresponding hoses with ultrapure water and then drain these components.
2. Switch off the analyzer from the main switch and allow the equipment to cool down.
3. Disconnect the gas supply and unplug the mains plug from the mains outlet.
4. Undo all connections on the back of the analyzer.
5. Open the doors of the analyzer and remove the reagent bottle and drip tray and any other loose accessory components.
6. Pull the hoses off the connections at the halogen trap and press the halogen trap out of the clamps.
7. Remove and drain the TIC condensate container (see section "Cleaning the TIC condensate container" p. 107).
8. Pack open hose ends in protective bags and secure them e.g. using adhesive tape.

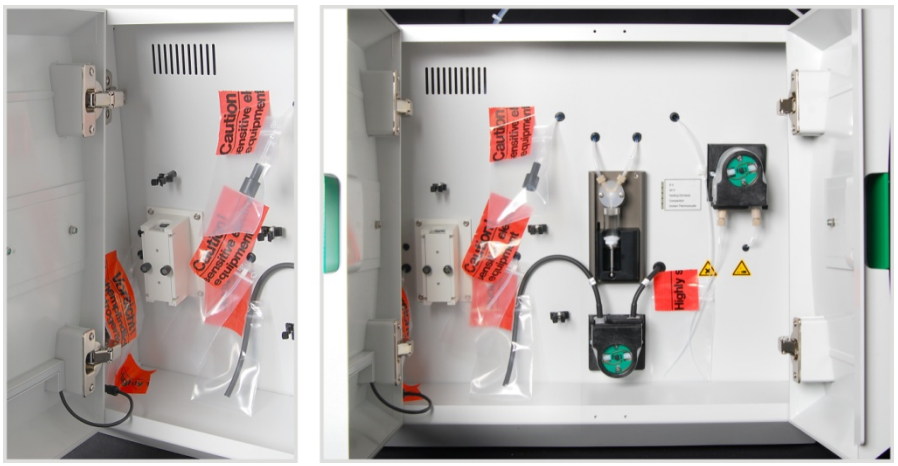


Fig. 57 Components behind the front door secured for transport

9. Open the left side wall,
   - Unscrew the four fastening screws; the screws are undetachable and remain in the wall.
   - Disconnect the grounding conductor connection and put the side wall aside.
10. Carefully remove the condensation coil from the holder, drain the condensation coil and safely put it aside (see section "Removing and cleaning the condensation coil" p. 110).
11. Remove the combustion tube (see section "Removing the combustion furnace" p. 108).
12. Remove the combustion furnace (see section "Removing the combustion furnace" p. 108).
13. Package the hose end of the condensation coil in a protective bag and secure it with adhesive tape.

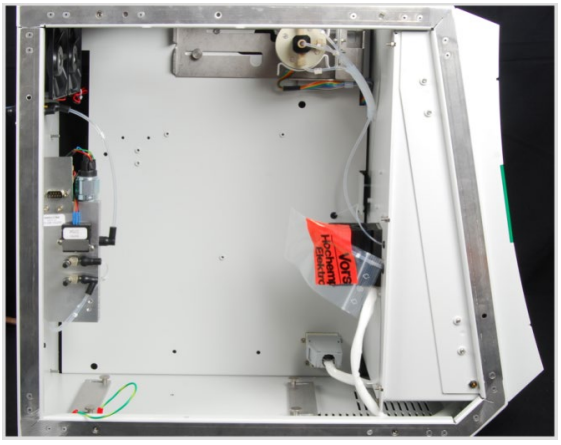


Fig. 58 Combustion furnace removed, loose hose ends secured

14. Remove the cannulas from the hoses and insert the cannulas into the cannula packaging.
15. Close the left side wall of the analyzer:
   - Connect the grounding conductor connection to the side wall.
   - First screw in the screws at the bottom and then the top side. Tighten the screws in turn.
16. Close the doors of the analyzer.

17. Attach the top cover and secure it with adhesive tape.
18. Carefully package the accessories, in particular protect glass components against breakage.

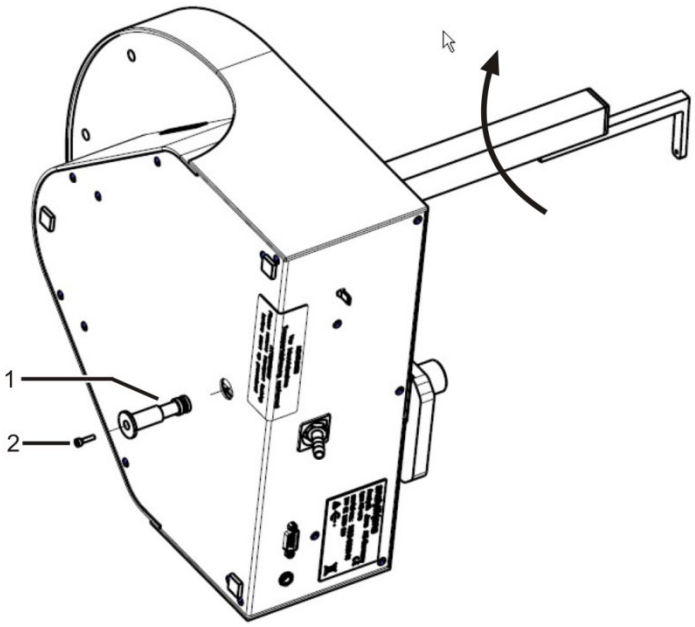
### 9.1.2 Transport notes

Observe the safety instructions in section "Safety instructions, transport and installation" p. 14. Transport the analyzer very carefully to prevent damage from impact or vibration. The analyzer should be transported in such a way that major temperature fluctuations are avoided and the formation of condensate is thus prevented.

### 9.1.3 Preparing the autosampler AS vario (ER) for transport

**Attention**

Before transporting the autosampler the transport lock has to be installed, otherwise the drives might be damaged.




1 Transport lock
2 Screw M3x12

**Fig. 59 Transport lock on autosampler AS vario (ER)**

1. Place the autosampler on the side as shown in Fig. 59.
2. Turn the autosampler arm clockwise to the stop.
   Then the drives are in the right position.
3. Push the red transport lock into the opening on the bottom of the autosampler as far as possible.
4. Bolt the screw (2 in Fig. 59) with the Allen wrench.

### 9.1.4 Moving the analyzer in the laboratory

**CAUTION**

Unintentional dropping of the analyzer poses a risk of injury and the analyzer will be damaged! Move the analyzer with great care! 2 persons are required to lift and carry the analyzer!

Observe the following when moving the analyzer within the laboratory:

- Insufficiently secured components pose a risk of injury! Before moving the analyzer remove all loose components, in particular the reagent bottle with phosphoric acid.
- Disconnect all supply connections and any add-on devices from the analyzer.
- To prevent health damage the following must be observed when moving the analyzer in the laboratory (lifting and carrying):

For reasons of safety 2 persons are required to transport the analyzer and must position themselves on both sides of the equipment.

Because the analyzer does not feature any handles, firmly grip the device from the bottom and make sure prior to simultaneous lifting the device that the sensitive components at the front are protected by the closed doors.

- Observe the guide values and adhere to the legally mandated limits for lifting and carrying without auxiliary means!
- For the setup at the new location observe the notes in section "Location requirements" p. 39.

## 9.2 Storage

**Attention**

Environmental influences and condensate formation can destroy individual components of the analyzer!

The analyzer must only be stored in acclimatized rooms. The atmosphere must be low in dust and free from aggressive vapors.

If the analyzer and add-on devices are not positioned immediately after delivery or are not required for a prolonged period of time, they should best be stored in their original packaging. A suitable desiccant should be placed in the packaging or the equipment to prevent damage from moisture.

The following requirements are placed on the climatic conditions in the storage room of the analyzer:

- Temperature range: +5 °C to +55 °C
- Max. humidity: +10 °C to +30 °C
- Air pressure: 0.7 bar to 1.06 bar

## 9.3 Recommissioning after transport or storage

### 9.3.1 Assembling the analyzer after transport or storage

When positioning the analyzer observe the notes in section "Location requirements" p. 39.

Assemble the components of the analyzer as follows:

1. Carefully remove the basic device, accessories and any add-on devices from the transport packaging. Do not damage the transport packaging!
2. Position the analyzer at the intended location.
3. Remove the adhesive tape at the top cover and the side walls, remove the top cover, put it safely aside, and open the doors.
4. Open the left side wall,
   - Unscrew the four fastening screws; the screws are undetachable and remain in the wall.
   - Disconnect the grounding conductor connection and put the side wall safely aside.
5. Remove all remaining adhesive tape and protective bags.
6. Install the combustion furnace (see section "Installing the combustion furnace" p. 109).
7. Install the halogen trap and the water traps (the water traps are connected to the halogen trap).
8. Install the TIC condensate container (see section "Cleaning the TIC condensate container" p. 107).
9. Install the condensation coil (see section "Inserting the condensation coil" p. 111).
10. Fill the combustion tube and install the combustion tube in the combustion furnace (see sections "Filling the combustion tube" p. 102 and "Installing the combustion tube" p. 104)
11. Close the left side wall of the analyzer:
    - Connect the grounding conductor connection to the side wall.
    - First screw in the screws at the bottom and then the top side. Tighten the screws in turn.
12. Place the reagent bottle with the drip tray into the analyzer.
13. Connect the cannulas to the hoses no. 7 and AA and tighten the Fingertight connections finger-tight.
14. Close the doors of the analyzer.
15. Position any add-on devices at the intended location and connect them. Observe in this regard the user manuals of the add-on devices.

## 9.3.2 Connecting the analyzer

The mains connection and media connections are on the analyzer backplate:

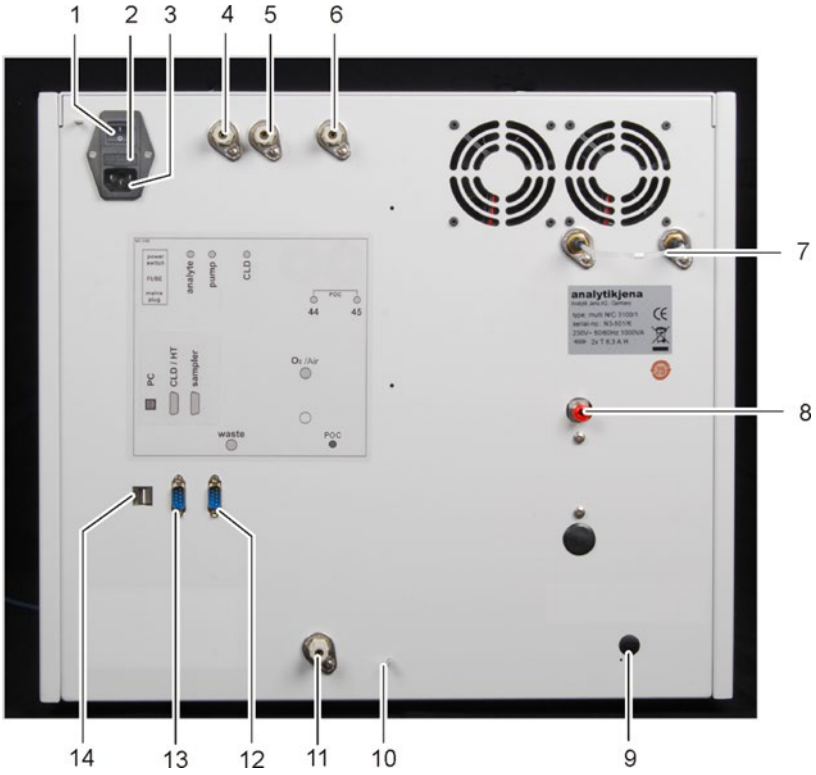



**Fig. 60 Mains connection and gas connections at the multi N/C 3100**

1 Main switch to switch the analyzer on and off "power switch"
2 Holder for mains fuse "FUSE"
3 Mains connection "main plug"
4 Gas connection "analyte"
5 Gas connection "pump"
6 Connection "CLD"
7 Bridge for the gas connection of the POC module
8 Carrier gas connection "$O_2$ /Air"
9 Connection for POC module (optional)
10 Connection of neutral conductor at the sampler
11 Waste connection "waste"
12 RS 232 interface for the sampler "sampler"
13 RS 232 interface for CLD and HT module "CLD/HT"
14 USB port for PC

Connect the mains cable

### CAUTION

Always connect the system components to the multi N/C 3100 when it is switched off!

Before connecting the mains cable ensure that the main switch on the back of the equipment is set to "0"!

Only use the low-heat connection cable supplied (VDE mark, 1.5 m long) for the mains connection. Extensions of the supply cable are not permitted!

### Attention

Settled condensation and temperature differences can damage individual components of the analyzer during recommissioning.

Allow the analyzer multi N/C 3100 to acclimatize for at least one hour before commissioning after positioning it in the operating room.

Make the mains connection as follows:

1. Connect the low-heat connection cable to the mains connection at the rear of the analyzer (3 in Fig. 60 p. 133).
2. Connect the mains plug of the low-heat connection cable to a grounded socket.

Connecting the gas supply

The operator is responsible for providing the necessary gas connection. Make sure that the preliminary pressure at the pressure reducer is set to between 4 and 6 bar.

Make the carrier gas connection as follows:

1. Connect the connection hose supplied to the pressure reducer of the gas supply and the gas connection "$O_2$/Air" on the equipment backplate (8 in Fig. 60 p. 133).
2. Set the preliminary pressure at the pressure reducer to between 4 and 6 bar.

The carrier gas connection at the equipment is a quick-release connection:

- The hose is inserted into the connection and thereby attached.
- To undo the hose the red ring must be pressed back and the hose pulled off the connection.

Connecting accessories

Connect the reagent bottle and accessory components as follows:

1. Connect the waste hose to the "waste" connection at the analyzer backplate and lead the loose end into a suitable waste container or drain.
2. Open the right front door at the analyzer and place the reagent bottle filled with 10 % phosphoric acid and the drip tray into the analyzer.
3. Connect the hoses no. 4 and no. AC to the reagent bottle with phosphoric acid.

# 10 Waste disposal

## 10.1 Waste water

Waste water arises during the ongoing analysis operation of the multi N/C 3100. Dependent on the measuring mode this contains hydrochloric acid, diluted phosphoric acid and sample matter.

Any neutralized waste must be brought to the appropriate waste disposal center for correct disposal according to the appropriate legal guidelines.

## 10.2 Halogen trap

The halogen trap contains copper. Contact the responsible agency (authority or waste disposal contractor). They will provide information about recycling or disposal.

## 10.3 Catalyst

The disposal of the depleted catalyst should be according to the local regulations (Waste name: used catalyst, CeO2). Analytik Jena GmbH accepts the special catalyst back for disposal. Please contact the customer service department (see inside front cover).

## 10.4 Analyzer

At the end of its service life the multi N/C 3100 and all its electronic components must be disposed of as electronic waste in accordance with the applicable regulations.

# 11 Specifications

## 11.1 Technical data

| | |
|---|---|
| General characteristics | |
| Designation/type | multi N/C 3100<br>multi N/C 3100 pharma |
| Dimensions | Basic device (W x H x D) (513 x 464 x 550) mm |
| Mass | approx. 28 kg |
| Procedural data | |
| Digestion principle | Thermocatalytic oxidation |
| Digestion temperature | up to 950 °C, depending on the catalyst |
| Catalyst | Platinum catalyst for multi N/C Pt($Al_2O_3$) |
| Measuring method | TC, TIC, TOC (differential method), NPOC, $TN_b$ (optional), POC (optional) |
| Carbon detection | NDIR (coupled with VITA method) |
| Nitrogen detection (optional) | CLD<br>ChD (not for multi N/C 3100 pharma) |
| Sample volume | 100 – 1000 µl |
| Particle handling capacity | according to DIN EN 1484 |
| Sample Feed | Flow injection |
| Gas supply | Synthetic air (free of HC, free of $CO_2$)<br>Synthetic/Purified air can be supplied from gas cylinders or after clean-up of pressurized air by a TOC gas generator.<br>Purity specifications to be met: $CO_2$ < 1 ppm<br>Hydrocarbons < 0,5 ppm (as $CH_4$)<br>Supply pressure: min. 5 bar (72 psi)<br>Provided flow rate: min. 300 ml/min<br>or oxygen (at least 4.5)<br>Preliminary pressure 4 – 6 bar |
| Gas consumption: | |
| in total | approx.15 l/h, dependent on the measuring mode |
| Analyte gas flow | 160 ± 10 ml/min approx. |
| Purge flow | 50 – 160 ml/min |
| Control/analysis (control and analysis software multiWin) | Real-time graphics, status indication during analysis, graphical display of the measured results, result print-out<br>Data integrity and compliance with FDA 21 CFR Part 11 and EudraLex Vol. 4 Annex 11 (in pharma version) |

| **Electrical variables** | |
|---|---|
| Connection | 230 V AC, optionally 115 V AC, 50/60 Hz |
| Protection | 230 V: T6,3 A H 115 V: T6,3 A H<br>(Use only original fuses of Analytik Jena GmbH) |
| Typical average power consumption | 400 VA |
| PC interface | USB 2.0 |
| Environmental conditions | |
| Temperature during storage | 5 – 55 °C |
| Temperature during operation | 10 – 35 °C |
| Humidity during operation | Max. 90 % at +30 °C |
| Humidity during storage | 10 – 30 % (use desiccant) |
| Air pressure | 0.7 – 1.06 bar |

Minimum equipment for the PC

| | |
|---|---|
| Operating system | Windows 7 Professional or better |
| Processor | 3,2 GHz or better |
| Working memory | 4 GB min. |
| Free hard disk space | 40 GB min. |
| Drive | CD/DVD drive (for software installation) |
| Monitor resolution | 1024 x 768 min |
| Interfaces | 1 USB 2.0 interface min. (for connecting the multi N/C) |

## 11.2 Standards and directives

Safety class, safety type

The analyzer belongs to protection class I. The casing has protection class IP 20.

Device safety

The analyzer conforms to the safety standards

- EN 61010-1
- EN 61010-2-081
- EN 61010-2-010
- EN 61010-2-051 (for operation with sampler)

EMC compatibility

The analyzer has been checked for interference emission and resistance.

It meets the requirements for interference emission of

- EN 61326-1 (EN 55011 Group 1, Class B)

It meets the requirements of interference resistance of

- EN 61326-1 (requirements for use in basic electromagnetic environments)

Environmental compatibility

The analyzer has been tested for environmental compatibility and meets the requirements of

- ISO 9022-3
- ISO 9022-2

EU directives

The analyzer is built and tested according to standards that meet the requirements of EU directives 2014/35/EU and 2014/30/EU. The analyzer leaves the factory in a sound condition as far as technical safety is concerned. To maintain this condition and to ensure safe operation, the operator must strictly observe the safety and operating instructions contained in this manual. For accessories which have also been supplied, and system components from other manufacturers, their operating instructions should be referred to.

Chinese directives

The analyzer contains regulated substances (according to directive "Management Methods for the Restriction of the Use of Hazardous Substances in Electrical and Electronic Products"). Analytik Jena GmbH guarantees that these substances will not leak out during the next 25 years if the analyzer is used in accordance with its intended use and thus do not pose a threat to the environment or health within this period.