

**Operating Manual** 

multi N/C 3300 HS TOC/TNb Analyzer



#### Manufacturer

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

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## 1 Basic information

### 1.1 About this user manual

Content

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

multi N/C 3300 HS

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

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

Conventions

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

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

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

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

Symbols and signal words used in this manual

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



### WARNING

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



### CAUTION

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

### NOTICE

Provides information on potential material or environmental damage.

### 1.2 Analyzer area of application

The analyzer is designed especially for pure and ultrapure water analysis. It is suitable for analysis of water for injection purposes (WFI) and water for pharmaceutical purposes.

The analyzer can optionally be equipped with an integrated solids module (Swab Test module). This permits carbon detection in solids in the lower concentration range, e.g., during cleaning verification (swab test).

The optional FDA software upgrade provides complete data integrity and conforms to the pharmaceutical guidelines 21 CFR Part 11 and EudraLex Volume 4 Annex 11.

### 1.3 Intended use

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

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

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

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

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

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

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

# 2 Safety

### 2.1 Safety labeling on the device

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

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

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

Warning symbol	Meaning	Comment
	Warning against hot surface	<ul> <li>On the furnace, on the furnace cover:</li> <li>On the left side wall: Risk of burns from the hot furnace</li> </ul>
	Warning against corro- sive substances	<ul> <li>On the front side, next to the phosphoric acid bottle: Warning against phosphoric acid</li> </ul>
	Warning against harm- ful or irritating sub- stances	<ul> <li>On the front side: Warning against phosphoric acid</li> </ul>
	Warning against crush- ing	On the autosampler: There is a risk of injury in the movement range of the autosampler.
	Warning of sharp ob- jects	On the autosampler: There is a risk of puncture injuries on the autosampler canula

Hazardous substances are used during operation:

GHS labeling	Meaning	Comment
	Corrosivity warning	On the phosphoric acid bottle: Phos- phoric acid is corrosive
Mandatory signs/ information sym- bols	Meaning	Comment
	Disconnect the power supply before opening the device cover	On the side parts and the rear of the device: Before opening the device cover, switch off the device and dis- connect the mains plug from the mains socket.
	Observe the operating manual	On the side parts and the rear of the device: Before starting work, read the operating manual.

Mandatory signs/ information sym- bols	Meaning	Comment
25)	For People's Republic of China only	The device contains controlled sub- stances. Analytik Jena warrants that these substances will not be released from the device within the next 25 years provided the device is em- ployed as intended.

### 2.2 Requirements for the operating personnel

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

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

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

### 2.3 Safety instructions, transport and commissioning

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

- Only the Analytik Jena customer service or specialist personnel trained and authorized by them is allowed to install and commission the device and its system components.
- Unauthorized assembly and installation is not permitted.
- Insufficiently secured components pose a risk of injury.
- During transport, secure the device components as specified in these operating instructions.
- Loose parts must be removed from the system components and packed separately.

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

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

### 2.4 Safety instructions: during operation

### 2.4.1 Summary of safety instructions

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

Observe the following:

- The device may only be operated if all items of protective equipment (e.g. covers in front of electronic components) are in place, properly installed and fully operational.
- The sound condition of the protection and safety equipment must be checked regularly. Any defects must be corrected as soon as they occur.
- Protective and safety equipment must never be removed, modified or switched off during operation.
- Always ensure free access to the main switch and to the emergency shutdown switches and locks during operation.
- The ventilation equipment on the device must be in good working condition. Covered ventilation grilles or slots etc. may cause the device to break down or may cause damage to it.
- Modifications, conversions and extensions to the device are only permitted after consultation with Analytik Jena. Unauthorized modifications can jeopardize the device's operational safety and may lead to limitations regarding the warranty and access to customer service.
- Keep all combustible materials away from the device.
- The furnace operates at temperatures of 700 to 950 °C. Do not touch the hot components (furnace, condensation coil) during or directly after operation.
- Caution when handling glass components. Risk of broken glass and therefore risk of injury!
- Ensure that no liquid enters the interior of the device, for example at cable connections. There is a danger of electric shock.
- There is a risk of injury in the movement range of the autosampler. For example, hands or fingers may be crushed. Maintain a safety distance from the autosampler during operation.
- The optional chemiluminescence detector (CLD) contains an ozone generator which produces ozone (O<sub>3</sub>). When used in accordance with the intended use, the downstream ozone decomposer destroys the toxic gas. Various safety measures result in the automatic shut-down of the ozone generator. However, the following still applies: if you notice a strong smell of ozone, switch the device off immediately and inform customer service. In order to guarantee perfect and safe operation, Analytik Jena recommends an annual inspection and maintenance of the detector by customer service.

### 2.4.2 Safety instructions – protection against explosion and fire

The device may not be operated in an explosive environment.

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

### 2.4.3 Electrical system safety instructions

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

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

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

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

### 2.4.5 Handling of auxiliary and operating materials

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

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

 Special care must be taken when handling concentrated acids. The regulations and notes in the safety data sheets for the handling of orthophosphoric acid (H<sub>3</sub>PO<sub>4</sub>) or hydrochloric acid (HCI) must be observed.

The combustion tube is filled with platinum or  $CeO_2$  catalyst and glass wool and ceramic glass wool.

Observe the following when handling operating materials that can form dust:

- Only store hazardous substances in closed containers.
- Avoid the formation of dust! Inhaled dust may cause irritation to respiratory pathways.
- Wear personal protective equipment (laboratory coat, protective gloves, safety goggles). Work under an extractor or wear a respiratory mask.
- Collect waste in sealed containers and dispose of it in accordance with applicable legal regulations.

Observe the following:

- The operator is responsible for carrying out suitable decontamination should the device become contaminated externally or internally with dangerous substances.
- Splashes, drops or larger liquid spillages should be removed using an absorbent material such as cotton wool, laboratory wipes or cellulose.
- For biological contamination, wipe the affected area with a suitable disinfectant, such as an Incidin Plus solution. Then wipe the cleaned areas so that they are dry.
- The only suitable cleaning method for the housing is wipe disinfection. If the disinfectant has a spray nozzle, apply disinfectant to a suitable cloth before using it on the device.

Work particularly carefully and cleanly with infectious material because the device cannot be decontaminated as a whole.

 Before using a cleaning or decontamination procedure other than that prescribed by the manufacturer, the user is required to check with the manufacturer that the intended procedure will not damage the device. Safety labels attached to the device must not have methanol applied.

#### 2.4.6 Safety instructions – maintenance and repair

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

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

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

#### See also

B Maintenance and care [▶ 73]

### 2.5 Behavior during emergencies

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

# 3 Function and design

### 3.1 Layout

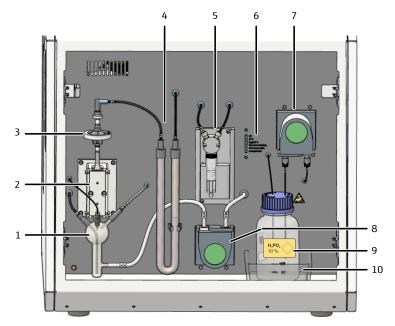
The analyzer is a compact table-top device with permanently installed main components. Further accessories and reagents are required for the measurement process.

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

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

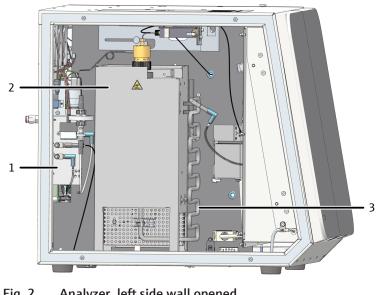
The analyzer consists of the following main components:

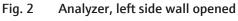
- Sample supply system
- Gas box and hose system
- Combustion system
- Measuring gas drying and cleaning
- Detector
- Indicator and control elements, connections
- Electronics
- Accessories



#### Fig. 1 Analyzer, front doors opened

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



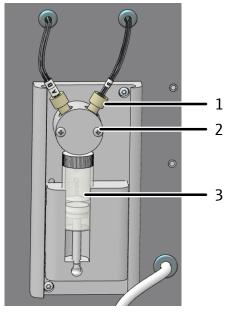


- Gas box
   Condensation coil
- 2 Combustion system

### 3.1.1 Sample supply system

Sample supply is carried out as flow injection via syringe pump with 2-port valve. The injection volume is 50 to 3000  $\mu$ l.

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



#### Fig. 3 Syringe pump

3 Dosing syringe

1 Fingertight connection

2 2-port valve

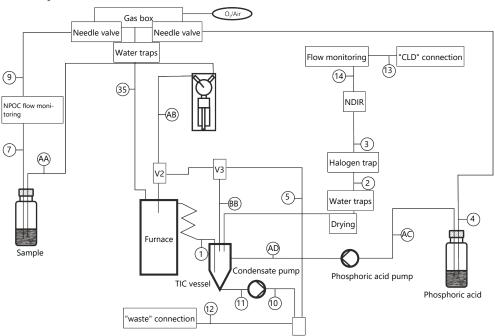
The hoses on the 2-port valve are connected to the following components:

- Hose AA to the sample
- Hose AB to the change-over valve

### 3.1.2 Hose system

Hose diagram

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



#### Fig. 4 Hose diagram

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

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

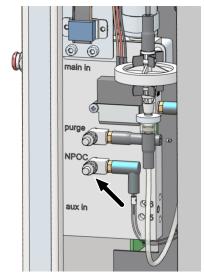


Fig. 5 Setting the NPOC purge flow

#### Condensate pump

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

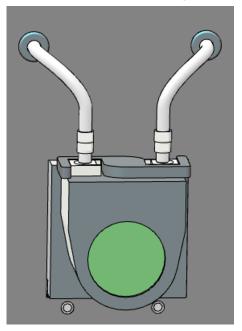


Fig. 6 Condensate pump

Phosphoric acid pump

The phosphoric acid pump transports phosphoric acid (10 %) to the TIC condensate container.

During this process, the phosphoric acid is permanently outgassed.

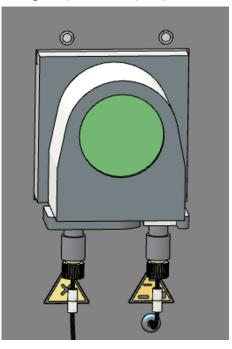
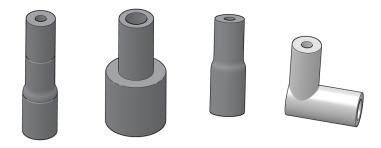


Fig. 7 Phosphoric acid pump

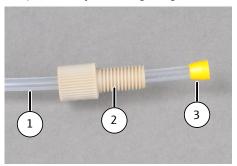
Connection method

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



#### Fig. 8 FAST connector

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





1 Hose

3 Conical nipple

2 Banjo bolt

#### 3.1.3 Combustion system

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

The combustion furnace is a resistor-heated vertical furnace for digestion temperatures of up to 950  $^{\circ}$ C.

A combined combustion furnace for vertical and horizontal operation can optionally be installed for operation with the Swab Test module for the analysis of solid samples.

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

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

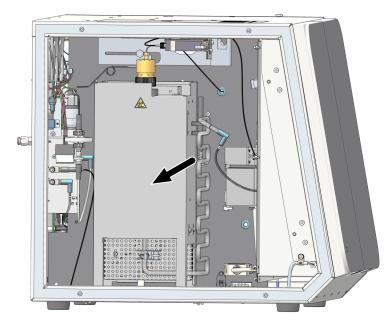


Fig. 10 Combustion furnace

### 3.1.4 Measuring gas drying and cleaning

Condensation coil

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

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

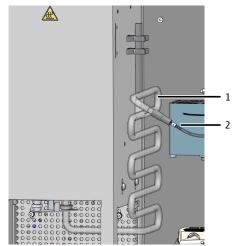


Fig. 11 Condensation coil

1 Condensation coil

TIC condensation module

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

2 Hose 1 to the TIC condensate container

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

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

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

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

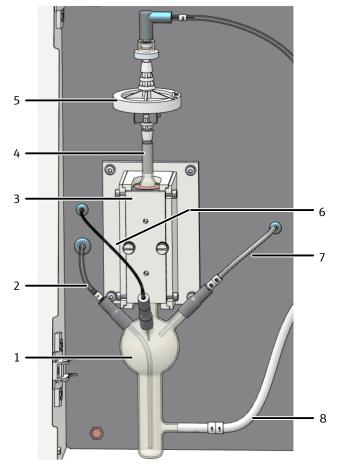
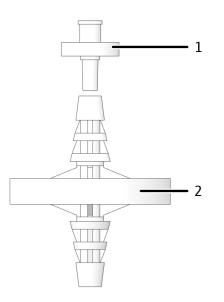


Fig. 12 TIC condensation module

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

Water traps

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



#### Fig. 13 Water traps

1 Disposable retention filter

2 TC prefilter

Halogen trap

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

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

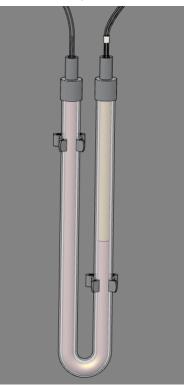
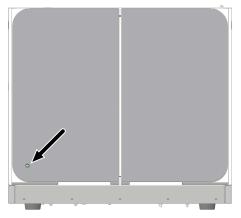


Fig. 14 Halogen trap

#### Indicator and control elements, connections 3.1.5

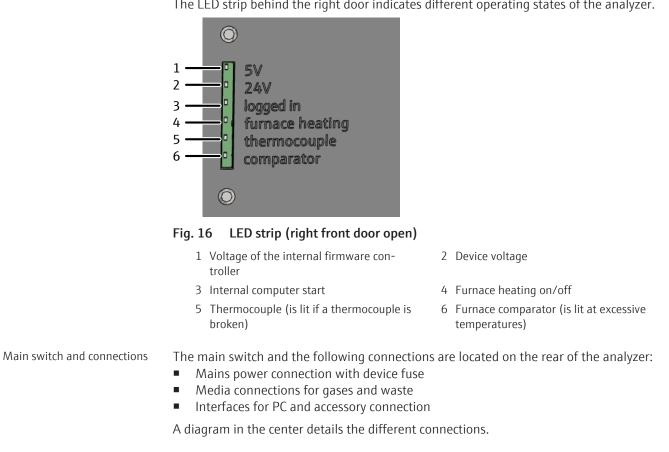
LED display

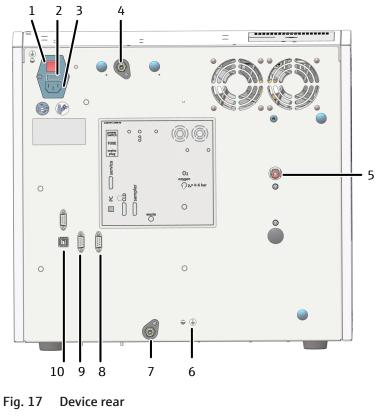
A green LED is installed on the left door of the analyzer. The LED is lit when the analyzer is switched on, indicating operational readiness.



#### Fig. 15 Status LED

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





- 1 "Power switch" main switch
- 3 "Main plug" mains connection
- 5 "O<sub>2</sub>/Air" carrier gas connection
- 7 "waste" connection
- 9 RS 232 interface for "CLD" CLD

- 2 "FUSE" mains fuse holder
- 4 "CLD" gas connection
- 6 Connection of the neutral conductor on the autosampler
- 8 RS 232 interface for "sampler" autosampler
- 10 USB 2.0 "PC" interface

Type plate

The type plate is attached to the device rear.

The type plate contains the following information:

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

### 3.1.6 Accessories

The following accessories are required for measurements with the analyzer:

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

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

### 3.2 Additional options for the analyzer

#### Autosampler

The following autosamplers are available for the analyzer:

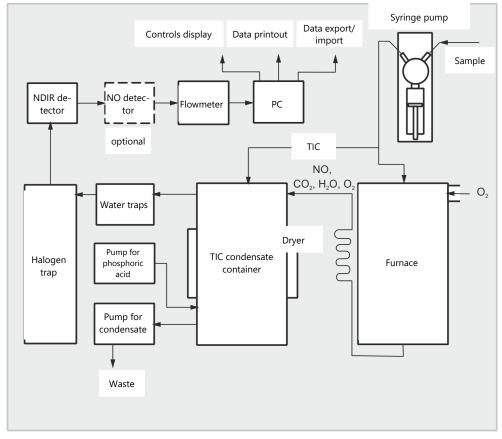
- AS vario with various tray sizes
- AS 10e for 10 samples
- AS 21hp for 21 samples
- EPA Sampler with piercing function

Integrated solids module The analyzer can be equipped with a Swab Test module to analyze small amounts of solid samples.

The module consists of a special reactor and a lock with manual feed. The model is inserted into the combustion furnace. During digestion of solid samples, temperatures of up to 950  $^{\circ}$ C are achieved. The digestion is supported by catalysts.

### 3.3 Function and measuring principle

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



#### Fig. 18 Principle of operation

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

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

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

 $\text{R-N+O}_2 \rightarrow \text{NO} + \text{CO}_2 + \text{H}_2\text{O} \text{ (2)}$ 

 $\text{R-CI+O}_2 \rightarrow \text{HCI} + \text{CO}_2 + \text{H}_2\text{O} \text{ (3)}$ 

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 gases, the  $CO_2$  measuring gas is added to the NDIR detector or NO detector.

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

The  $CO_2$  or NO concentration is detected several times every second. An integral over time is calculated from this signal sequence. The integral is proportional to the concentration of the carbon or nitrogen in the measurement solution. Afterwards, the calculation of the carbon or nitrogen content in the sample is performed via a previously determined calibration function.

### 3.4 Measuring methods

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

#### 3.4.1 TC analysis

TC: Total Carbon

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

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

 $TN_{b}$  detection is possible in parallel to TC detection.

#### 3.4.2 TOC analysis

TOC: Total Organic Carbon

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

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

TOC = TC - TIC

TOC - total organic carbon

TC - total carbon

TIC - total inorganic carbon

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

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

 $TN_{b}$  detection is possible in parallel to TOC detection.

### 3.4.3 TIC analysis

TIC: Total Inorganic Carbon

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

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

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

### 3.4.4 NPOC analysis

NPOC: Non-purgeable Organic Carbon

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

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

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

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

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

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

- Acidify the sample outside the analyzer (pH <2).
- Purge most of the carbon dioxide formed externally immediately before analyzing.
- Prepare an NPOC plus method and analyze the samples.
- The analyzer determines the TC and TIC content of the prepared samples and calculates the NPOC content from the difference.

Since you have purged most of the inorganically bound carbon externally, the TIC value determined using this method is only a calculated value and has no analytical relevance.

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

 $TN_{b}$  detection is possible in parallel to the NPOC and NPOC plus detection.

### 3.4.5 DOC analysis

DOC: Dissolved Organic Carbon

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

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

### 3.4.6 TN<sub>b</sub> analysis

TN<sub>b</sub>: Total Nitrogen bound

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

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

### 3.4.7 Additional sum parameters

In the control and analysis software, you can activate the calculation of additional sum parameters in the method settings.

CSB	CSB (COD): Chemical Oxygen Demand
	For TOC and NPOC methods, you can activate the calculation of the COD based on the TOC or NPOC.
	Formula: $c(COD) = A \times c(TOC) + B$
	You can define the rise (A) and intercept (B) for the calculation of the COD, default setting: $A = 3.000$ , $B = 0.000$ .
BOD5	BOD <sub>5</sub> : Biochemical Oxygen Demand
	For TOC and NPOC methods, you can activate the calculation of the BOD5 based on the TOC or NPOC.
	Formula: $c(BOD_5) = A \times c(TOC) + B$
	You can define the rise (A) and intercept (B) for the calculation of the $BOD_5$ , default setting: A = 3.000, B = 0.000.
CO2	For TIC methods and liquid measurements, you can activate the calculation of the car- bon dioxide concentration based on the TIC.
	Formula: $c(CO_2) = 2.833 \times c(TIC)$
TP	TP: Total Protein
	For TN methods you can activate the calculation of the total protein content based on the TN.
	Formula: c(Total Protein) = A x c(TN)
	You can set the factor for calculating the total protein content between 0 and 10, de- fault setting: A = 6.250 (Comparative substance: BSA – Bovine Serum Albumin).

### 3.5 Catalysts

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

The platinum catalyst can be used universally over the entire working range for carbon and nitrogen determination. Its optimal function is at a reaction temperature of 750 °C. Because of its very low individual blank value, it allows safe and precise analysis of low carbon and nitrogen contents. The catalyst also works effectively during analysis of highly contaminated waters.

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

Alternatively, a CeO<sub>2</sub> catalyst can be used at a reaction temperature of 850 °C.

### 3.6 Calibration

### 3.6.1 Calibration strategies

#### Multiple point calibration with constant sample volume

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

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

#### Multiple point calibration with constant concentration

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

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

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

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

### Single point calibration

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

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

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

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

### 3.6.2 Daily factor

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

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

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

### 3.6.3 Calibration method

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

You can define up to three linear calibration functions for different concentration ranges for each parameter. The software automatically assigns the measurement results to the correct calibration range.

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

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

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

c: target concentration of the standards

V: Sample volume

I<sub>Net</sub>: Net integral

 $k_0, k_1, k_2$ : calibration coefficient

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

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

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

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

The TIC channel is calibrated.

The following applies:  $c_{TIC} = f(I_{TIC})$ 

TOC The TOC is determined with the differential method (TOC Diff). Generally, separate calibration functions are determined for the TC and TIC channels.

The calculation of the analysis results is based on the calculated calibration functions for TC and TIC. The TOC content results from the following formula:

 $c_{TOC} = c_{TC} - c_{TIC}$ 

The TC and TIC parameters can be calibrated simultaneously. The use of mixed standard solutions such as carbonate/hydrogen carbonate and potassium hydrogen phthalate or sucrose is recommended for this.

The TIC and TC channels can also be calibrated consecutively with separate standard solutions. This is useful if different ranges are to be calibrated for the TC and TIC channels.

TC/NPOC

TIC

NPOC plus The calibration of the NPOC plus method is the same as the calibration of the TOC (Diff) method. Before analysis, the TIC must be sufficiently purged for the use of the differential method to be practical. Method process: Separate calibration of TIC and TC channels Measurement of samples and calculation of the analysis results via the software - Purging of the acidified sample (3 to 5 min) - Determination of the remaining TIC with the calibration curve - Determination of the TC with the calibration curve - Calculation of the TOC from the difference of TC and TIC The matrix-dependent calibration is as close to real samples as possible. To do this, add carbonate to the standard solutions until you get a TIC content similar to that of the samples. TNb The TN channel is calibrated. The following applies for the determined calibration function:  $c_{TN} = f(I_{TN})$ .

### 3.6.4 Method characteristics

Coefficient of determination	The coefficient of determination allows the quality of fit of the regression model to be assessed. The coefficient of determination is calculated as the square of the correlation coefficient. The correlation coefficient compares the dispersion of the calibration measuring points of the regression function with the total dispersion of the calibration.
Limit of verification	The verification limit of the calibration specifies the lowest concentration that can be differentiated qualitatively from the zero point with a given probability. The verification limit should always be smaller than the lowest calibration measuring point.
Limit of determination	The determination limit of the calibration specifies the lowest concentration that can be differentiated quantitatively from the zero point with a given probability.

### 3.6.5 Other calculations

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

If carbon and nitrogen are detected in parallel, the outlier selection takes place separately for each parameter.

Average value

The average value of the final result is calculated from the concentrations determined for the individual detections after eliminating the outliers.

### 3.7 Blank values

### 3.7.1 Water blank values

Preparation water blank value	Especially for measurements with low TOC concentrations ( $\mu$ g/l range), the TOC content of the water used to prepare the standard solutions must be taken into account. The concentration of the standard solution and the TOC blank value of the preparation water are often within the same range. This blank value can be taken into account during cali- bration.	
	The TOC content of the preparation water is measured separately before the calibration. The software then subtracts the average integral determined for the preparation water for each measuring point of the calibration from the determined gross integral.	
	$I_{\text{Net}} = I_{\text{Gross}} - I_{\text{Preparation water}}$	
	The software determines the calibration function from the net integrals. Mathemati- cally, this corresponds to a parallel movement of the calibration line.	
	The software also takes the preparation water blank value into account when determin- ing the daily factor.	
Diluent blank value	If the sample is diluted, the blank value for the diluent is of interest. This value can be determined separately or entered manually in the software. The software takes the diluent blank value into account when calculating the concentration of diluted samples.	
	The diluent blank value can change over time and must therefore be determined again before beginning a measurement series. Otherwise, the software will use the last value.	
	The diluent blank value is always indicated in the software normalized to a volume of $1 \text{ ml.}$	
Diluent blank value use	The software calculates the actual diluent integral $(I_{DiBV})$ for each measurement based on the diluent blank value, the sample volume used and the dilution ratio. The software then subtracts the diluent integral $(I_{DiBV})$ from the experimentally determined raw integral $(I_{Raw})$ .	
	$I_{DiBV} = V_{DiBV} \times (V_{Sample} - N_{P}/N_{D} \times V_{Sample})$	
	$I_{eff} = I_{Raw} - I_{DiBV}$	
	V <sub>DiBV</sub> : Diluent blank value	
	V <sub>Sample</sub> Sample volume	
	l <sub>eff</sub> : Effective integral	
	$N_{P}$ : Number of units of the primary sample	
	$N_{D}$ : Number of units of the diluent	
	I <sub>Raw</sub> : Raw integral	
	I <sub>DIBV</sub> : Diluent integral	

Diluent indication	Proportions of the primary probe: in total proportions (e.g., 10 parts in 100 parts)
	This means that 10 ml of the primary sample is filled to a total volume of 100 ml with dilution water.
	A 1:1 dilution ratio equals $I_{DiBV} = 0$ .
Calculation of the sample con- centration	To calculate the sample concentration c, the sample volume and the dilution ratio are used:
	$c = m/V_{sample} \times N_D/N_P$
	The following equation applies for the linear calibration function:
	$c = (k_1 \times I_{eff} + k_0) / V_{Sample} \times N_D / N_P$
	If the user dilutes a sample and enters the dilution ratio in the software, the software automatically calculates the concentration of the undiluted primary sample and outputs it to the analysis report.

#### 3.7.2 Eluate blank value

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

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

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

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

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

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

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

I<sub>eff</sub>: Effective integral

I<sub>Raw</sub>: Raw integral

I<sub>Eluate blank value</sub>: Eluate blank value

#### 3.7.3 Boat blank value

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

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

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

### 3.8 System suitability test

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

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

The standards solutions and their concentrations are defined in the respective pharmacopeia, e.g., in the European Pharmacopeia or in the USP (United States Pharmacopeia). These define sucrose as an easily oxidizable compound, and p-benzoquinone as a poorly oxidizable one. The ratio of the recovery rate of p-benzoquinone to the recovery rate of sucrose must be within the range of 85 to 115 %. Only then is the selected method suitable.

Procedure:

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

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

 $E = (r_{ss} - r_{W})/(r_{s} - r_{W}) \times 100$ 

E: System efficiency in %

- r<sub>s</sub>: TOC of the reference solution (sucrose)
- r<sub>ss</sub>: TOC of the system suitability solution (p-benzoquinone)
- r<sub>w</sub>: TOC of the TOC water used (preparation water blank value)

# 4 Installation and commissioning

### 4.1 Installation conditions

### 4.1.1 Ambient conditions

- This laboratory device is designed for inside use.
- Avoid direct sunlight and radiation from heaters onto the device. If necessary, provide air conditioning.
- The installation site must be free of drafts, dust and caustic fumes.
- The room air must be as low in TOC and NO<sub>x</sub> as possible.
- Avoid mechanical shocks and vibrations.
- Do not locate the device near sources of electromagnetic interference.
- Place the device on a heat-resistant and acid-resistant surface.
- The device must be positioned in such a way that allows easy access from all sides.
- Keep the ventilation slits free and do not obstruct them with other devices.

The following climate requirements apply in the room of operation:

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

### 4.1.2 Device layout and space requirements

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

The AS 10e and AS 21hp liquid autosamplers are attached to the right side wall of the basic device. Alternatively, the autosamplers can be placed next to the device.

There must be a clearance of at least 10 cm between the device system and any cabinet/shelf located above it.

Further components of the measuring station:

- The PC, monitor and printer may be placed on a separate side table.
- An acid-resistant waste container can be placed on or under the bench.
- With the multi N/C 3300 HS model, the AS vario and EPA Sampler autosamplers must be placed next to the basic device on the right.
- The CLD-300 nitrogen detector is set up to the right of the basic device.
- With the multi N/C 3300 HS model, the CLD-300 nitrogen detector must be placed next to the basic device on the left.
- The integrated solids module (Swab-Test Module) is attached to the left side panel of the basic device.

Component	Dimensions (Width x Depth x Height)	Weight
Basic device	513 x 547 x 464 mm	21 kg
AS 10e autosampler	260 x 320 x 390 mm	4.5 kg
AS 21hp autosampler	260 x 320 x 390 mm	4.5 kg
AS vario autosampler	350 x 400 x 470 mm	15 kg
EPA Sampler	500 x 540 x 550 mm	15 kg
CLD-300 nitrogen detector	296 x 581 x 462 mm	12.5 kg
Swab-Test Module	300 x 80 x 80 mm	3 kg

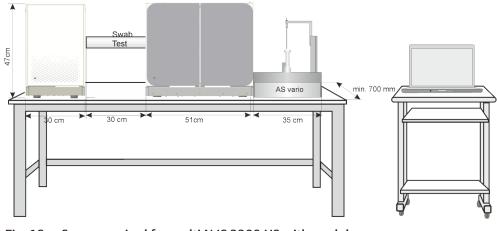


Fig. 19 Space required for multi N/C 3300 HS with modules

### 4.1.3 Power supply



### WARNING

#### Danger due to electrical voltage

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

The device operates on single-phase alternating current.

Before connecting the device to a power outlet, check its voltage rating to ensure that the required voltage and frequency match the available power source.

### 4.1.4 Gas supply

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

The connection hose is supplied:

- Outer diameter 6 mm
- Inner diameter 4 mm

## 4.2 Unpacking and setting up the device

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

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

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

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

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

### 4.2.1 Installing and commissioning the analyzer

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

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

- Close the doors of the analyzer.
  - $\checkmark$  The device has been installed.

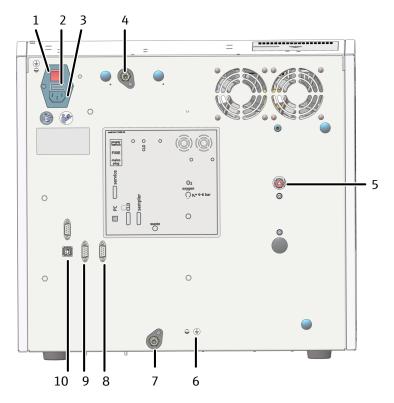
#### See also

■ Maintenance and care [▶ 73]

### 4.2.1.1 Connecting the analyzer

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

A diagram in the center details the different connections.



#### Fig. 20 Device rear

- 1 "Power switch" main switch
- 3 "Main plug" mains connection
- 5 "O<sub>2</sub>/Air" carrier gas connection

7 "waste" connection

9 RS 232 interface for "CLD" CLD

- 2 "FUSE" mains fuse holder
- 4 "CLD" gas connection
- 6 Connection of the neutral conductor on the autosampler
- 8 RS 232 interface for "sampler" autosampler
- 10 USB 2.0 "PC" interface



## Connecting the power

## NOTICE

### Risk of damage to the sensitive electronics

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



## NOTICE

### Damage to the electronics due to condensation

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

- After long-term storage or transport in a colder environment, allow the device to acclimatize at room temperature for at least one hour before switching it on.
- Connect the connection cable to the mains power connection on the rear of the analyzer.
- Connect the power plug to a grounded power outlet.
- Do not switch the device on yet.

### Connecting the gases

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

- Connect the carrier gas. To do this, connect the supplied connection hose to the pressure reducer of the gas supply.
- Connect the carrier gas hose to the  $"O_2/Air"$  gas connection on the rear of the device.
  - To do this, insert the hose in the quick-release connector.
  - To release the hose again later, press the red ring back and pull the hose out of the connection.

### **Connecting accessories**



## WARNING

### Risk of chemical burns from concentrated acids

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

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

Connect the reagent bottle and accessory components as follows:

- Connect the waste hose to the "waste" connection on the rear of the analyzer. Put the free hose end in a suitable waste container.
- Open the front doors on the analyzer.

- Fill the reagent bottle with phosphoric acid (10 %). Put the bottle with the drip tray into the analyzer.
- Connect hoses 4 and AC to the reagent bottle with phosphoric acid.
  - ✓ The analyzer has been commissioned.

## 4.3 Connecting accessories



### NOTICE

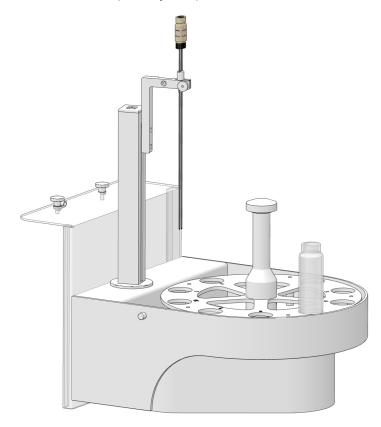
### Risk of damage to the sensitive electronics

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

### 4.3.1 The AS 10e and AS 21hp autosamplers

AS 10e autosampler

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



### Fig. 21 AS 10e autosampler

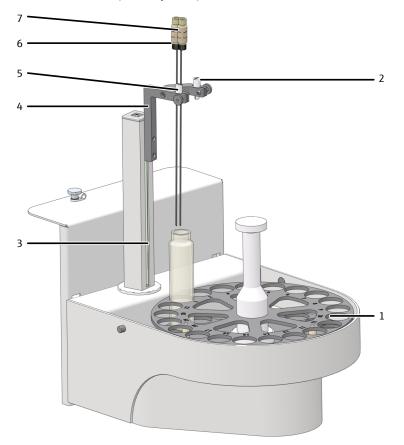
The autosampler can be equipped with two canulas. This allows the autosampler to automatically purge samples for NPOC analysis.

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

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

- First, the autosampler purges volatile organic compounds and CO<sub>2</sub> from the sample.
- In a second step, the autosampler picks up the prepared sample and transfers it via the intake hose to the analyzer.

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



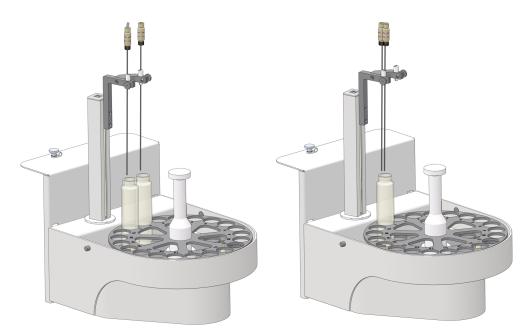
### Fig. 22 AS 21hp autosampler

- 1 Sleeve (with 1 bore) as a purging canula holder
- 3 Autosampler arm with Z-drive
- 5 Sleeve (with 2 bores)

- 2 Sample tray (rotatable, 21 samples)
- 4 Canula holder
- 6 Sample intake canula with screw connection
- 7 Purging canula with screw connection

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

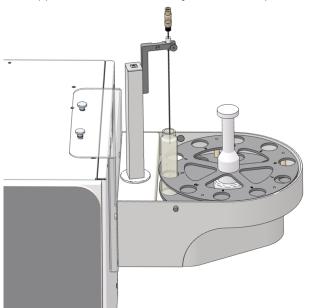
The autosampler comes with a canula holder for two canulas. The holder keeps the two canulas at a distance. This allows the autosampler to aspirate a sample and to purge a second sample in parallel (**parallel purging**). During NPOC analysis, the autosampler can also work sequentially (option).



### Fig. 23 Parallel purging (left) and sequential purging (right)

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

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



#### Fig. 24 Autosampler attached to the analyzer by means of the holder

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

Cover (optional)

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

### 4.3.1.1 Installing and commissioning the sampler



## CAUTION

### Risk of injury from moving parts

There is a risk of injury in the movement range of the sampler arm. For example, hands or fingers may be crushed. The canula can cause puncture injuries.

• Maintain a safety distance from the autosampler during operation.



## NOTICE

### Risk of device damage

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

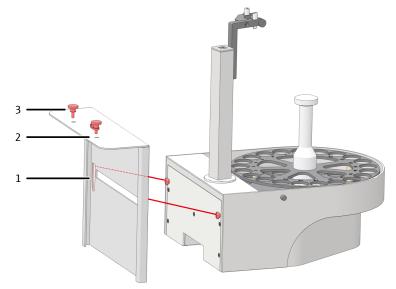
- Do not touch the sampler arm during operation.
- Only carry out manual adjustment when the device is switched off.
- Switch off the analyzer before installing the autosampler.
- Plug the grounding conductor into the connection on the rear of the analyzer. Connect the grounding conductor to the connection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



### Fig. 25 Connections on the bottom of the autosampler

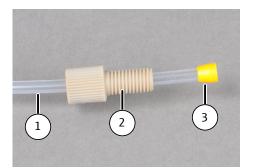
- 1 Connection for equipotential bonding 2 Power cable connection cable (grounding cable)
- 3 Analyzer interface
- Attach the autosampler to the side of the analyzer with the holder.

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



#### Fig. 26 Attaching the AS 21hp autosampler to the holder

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



#### **Fingertight connection** Fig. 27

1 Hose

2 Banjo bolt

3 Conical nipple

- Connect the power supply unit to the mains network.

Switch on the components of the analysis system. Start the software.

- Check the device configuration with the **Instrument** | **Manage instruments** menu option in the Manage instruments window.
- If necessary, change the device configuration or create a new device configuration:
  - Click on the **Add** button to create a new device configuration.
  - Edit the device configuration in the detail view **Instrument configuration**.
  - Select autosampler in the dropdown menu under **Sampler type**.
  - Select Sample tray in the dropdown menu under **Rack size:**.
- Select sample vial size from the dropdown menu Vial size (mL):. The software ad-justs the dead volume accordingly. Optionally you can adjust the dead volume at Dead volume (mL):.
- Click the 🗹 button to save the device configuration.
- Click on **Set default** to activate the device configuration as standard configuration.

#### Adjusting the autosampler

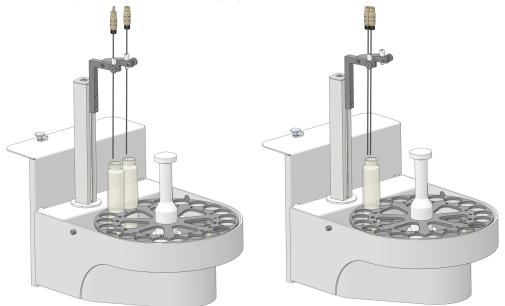
During adjustment, you adjust the immersion depth of the canulas so that the needles are optimally immersed in the sample vessels. Adjust the autosampler during commissioning and after each conversion, transportation or storage.

- ▶ Start the software
- Place the sample vessel in position 1.
- Place magnetic stirring rod in the sample vessel in the AS 21hp autosampler.
- Use the Instrument | Sampler alignment menu option to open the Sampler alignment window.
- Select adjustment position **Position 1** from the list box in the **Sampler position** sec-tion.
- Click on the Request current values button to retrieve the current offset values.
- Adjust the immersion depth of the canulas using the up-down control higher / + lower in 0.1 mm steps.
- After each change, click **Move** to check the immersion depth.
- With the AS 21hp autosampler, maintain a distance of about 0.5 cm from the mag-netic stirring rod so that the rod can move freely and does not damage the canulas.
- After adjustment save offset values by clicking on **Confirm**. Close the window.
  - ✓ The autosampler is ready for operation.

Checking and extending the configuration

### 4.3.1.2 Conversion for parallel purging (AS 21hp)

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



### Fig. 28 Parallel purging (left) and sequential purging (right)

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

### See also

Installing and commissioning the sampler [▶ 43]

### 4.3.2 AS vario autosampler



## CAUTION

### Risk of injury from moving parts

There is a risk of injury in the movement range of the sampler arm. For example, hands or fingers may be crushed. The canula can cause puncture injuries.

• Maintain a safety distance from the autosampler during operation.



## NOTICE

### Risk of device damage by commissioning with transport locks

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

• Remove the transport locks before commissioning.



## NOTICE

### Risk of device damage

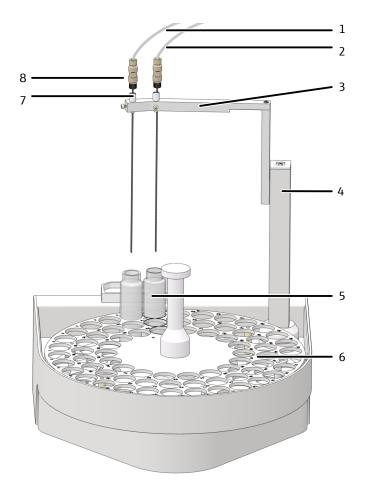
If the sampler arm is obstructed during operation, the drives can be destroyed.

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

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

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

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

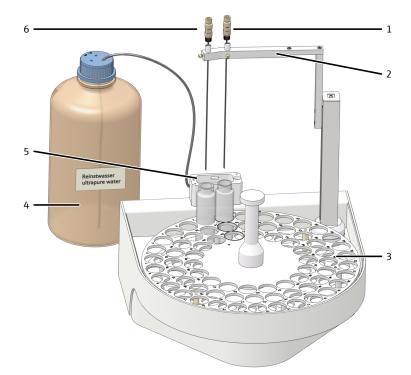


### Fig. 29 Layout of the AS vario autosampler

- 1 Connection hose to the analyzer (purging hose for NPOC measurement)
- 3 Canula holder
- 5 Sample vessel
- 7 Sleeve

- 2 Connection hose to the analyzer (sample intake hose)
- 4 Autosampler arm
- 6 Sample tray
- 8 Canula

The AS vario ER model is particularly suited for analyzing liquid samples with high solid particle content. The model is equipped with an additional canula flush that flushes the canula(s) with ultrapure water from the outside. When commissioning the autosampler, the ultrapure water supply for the canula flush must be installed additionally. It can be used for all measurement methods, and in particular for NPOC analysis with parallel purging. There is a suitable block with wash cups for each sample tray. When using different sample trays, simply unscrew the block with the wash cups from the autosampler and exchange it.



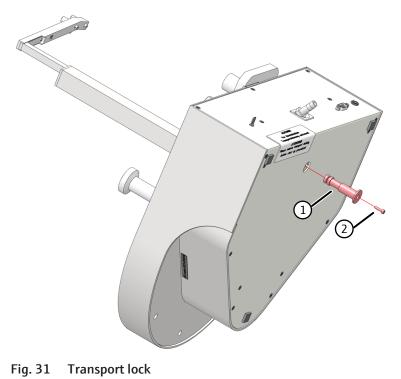
### Fig. 30 Layout of the AS vario ER autosampler

- 1 Canula for connection to the sample intake hose
- 3 Sample tray for 72
- 5 Canula flush

- 2 Canula holder (here with no. 72)
- 4 Ultrapure water bottle
- 6 Canula for connection with the purge hose for NPOC measurement

Removing the transport locks

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



1 Transport lock

2 M3x12 screw

- Turn the autosampler on its side and put it down safely.
- Remove the screw with the supplied hexagon socket screwdriver. Remove the transport lock (red plastic part).
- Place the autosampler on the bottom plate again.

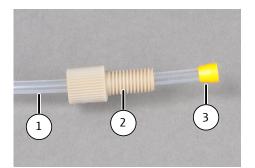
Commissioning the autosampler

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



#### Fig. 32 Sleeve with two canulas for non-parallel purging

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



### Fig. 33 Fingertight connection

1 Hose

2 Banjo bolt

3 Conical nipple

Z Banjo bo

• Connect the power supply unit to the mains network.

• Switch on the components of the analysis system. Start the software.

- Check the device configuration with the **Instrument** | **Manage instruments** menu option in the **Manage instruments** window.
- If necessary, change the device configuration or create a new device configuration:
  - Click on the **Add** button to create a new device configuration.
  - Edit the device configuration in the detail view Instrument configuration.
  - Select autosampler in the dropdown menu under Sampler type.
  - Select Sample tray in the dropdown menu under **Rack size:**.
- Select sample vial size from the dropdown menu Vial size (mL):. The software adjusts the dead volume accordingly. Optionally you can adjust the dead volume at Dead volume (mL):.
- Click the  $\boxdot$  button to save the device configuration.
- Click on **Set default** to activate the device configuration as standard configuration.
- Installing the canula flush

Checking and extending the

configuration

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



#### Fig. 34 Canula flush on the AS vario ER model

- 1 Ultrapure water connection
- 2 Waste connection
- 3 Removable block with wash cups
- Place the suitable block with wash cups on the autosampler.
  - For simpler installation, wet the O-ring on the bottom of the block with water.
  - Fasten the block to the autosampler with the two hexagon socket screws.
- Screw the ultrapure water connection into connection (1) and place the hose end in the ultrapure water bottle.
- Insert the waste hose in connection (2). Place the hose end in the waste container.
   NOTICE! Route the outlet tube at a constant downward incline. If necessary, shorten the hose. The hose must not dip into the liquid.
- Adjust the autosampler before the first start.

Activating the canula flush for measurements

- Create a new method.
- Set the number of flushing cycles on the Step properties tab at Reverse rinse. One flushing process is usually sufficient.

#### See also

Adjusting the AS vario autosampler [▶ 75]

### 4.3.3 EPA Sampler



## CAUTION

### Risk of injury from moving parts

There is a risk of injury in the movement range of the sampler arm. For example, hands or fingers may be crushed. The canula can cause puncture injuries.

• Maintain a safety distance from the autosampler during operation.



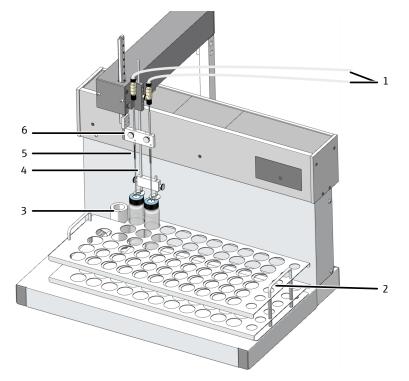
## NOTICE

### Risk of device damage

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

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

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

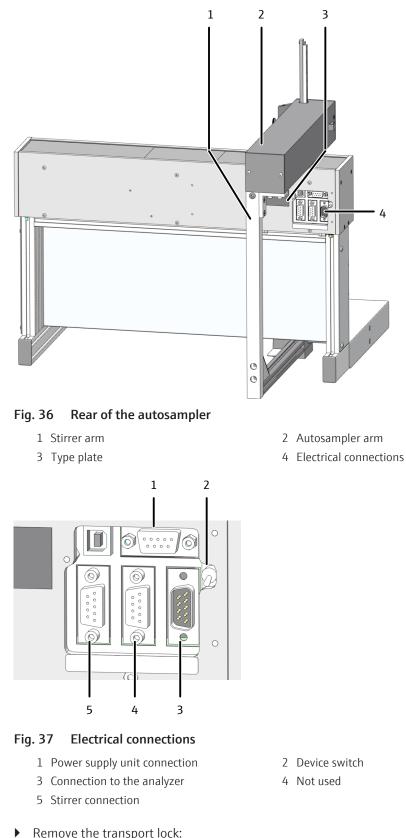


### Fig. 35 EPA Sampler autosampler

- 1 Connection hoses to the analyzer
- 3 Wash cup
- 5 Special canula

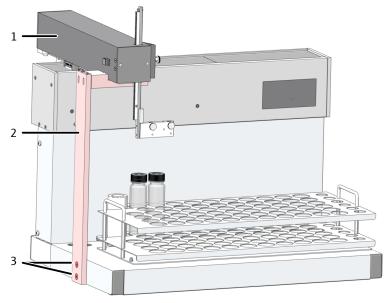
- 2 Sample tray
- 4 Holding-down clamp
- 6 Autosampler arm with canula holder

Design



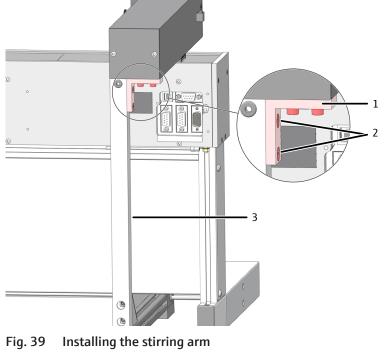
Commissioning the autosampler

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



### Fig. 38 Transport lock

- 1 Autosampler arm2 Transport retaining clip3 Screws
- ▶ Install the stirring arm:
  - Install the stirring arm on the bracket on the rear of the autosampler arm.
- Screw on the arm with the supplied countersunk screws (M4x10) using the hexagon head wrench (A/F2.5).
  - Tighten the screws evenly to aligned the arm.
  - Connect the stirrer cable to the "Stirrer" connection on the rear of the autosampler.



- 1 Bracket on the autosampler arm
- 2 Countersunk screws

3 Stirrer arm

- Place the autosampler next to the analyzer. Position the autosampler so that enough space is provided behind the device for the motion range of the autosampler arm as well.
- Connect the low voltage side cable of the table power supply unit to the rear of the autosampler. Do not connect the power supply unit to the mains power supply yet.
- Connect the supplied serial data cable to the "sampler" interface on the rear of the analyzer. Connect the other end of the data cable to the interface on the autosampler.
- Plug the grounding conductor into the connection on the rear of the analyzer.
- Connect the waste hose to the wash cup of the autosampler and to a suitable waste container or drain.

**I** NOTICE! Route the outlet hose at a constant downward incline. If necessary, shorten the hose. The hose must not dip into the liquid.

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

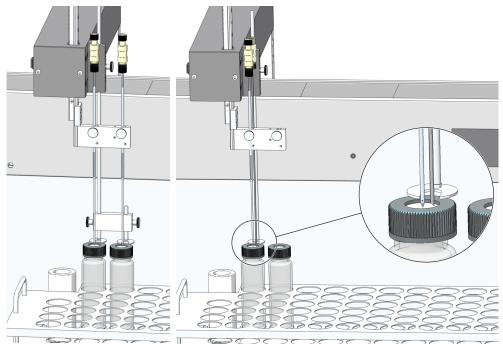
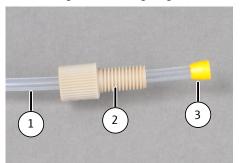


Fig. 40 Canula position for NPOC measurement with parallel (left) and non-parallel (right) purging.

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

- Retighten the Fingertight connections.



### Fig. 41 Fingertight connection

1 Hose

2 Banjo bolt

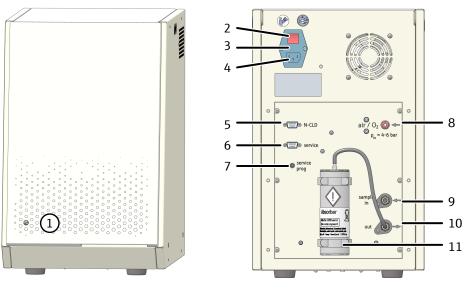
- 3 Conical nipple
- Connect the power supply unit to the mains network. Switch on the autosampler.
- Switch on the components of the analysis system. Start the software.
- Check the device configuration with the **Instrument** | **Manage instruments** menu option in the **Manage instruments** window.
- If necessary, change the device configuration or create a new device configuration:
  - Click on the **Add** button to create a new device configuration.
  - Edit the device configuration in the detail view Instrument configuration.
  - Select autosampler in the dropdown menu under **Sampler type**.
  - Select Sample tray in the dropdown menu under **Rack size:**.
- Select sample vial size from the dropdown menu Vial size (mL):. The software adjusts the dead volume accordingly. Optionally you can adjust the dead volume at Dead volume (mL):.
- ▶ Click the 🗹 button to save the device configuration.
- Click on **Set default** to activate the device configuration as standard configuration.
- Adjust the autosampler before the first start.

### See also

Adjusting the EPA Sampler [▶ 77]

Checking and extending the configuration

### 4.3.4 Chemiluminescence detector (CLD)



### Fig. 42 Chemiluminescence detector (CLD)

- 1 Status LED
- 3 Fuse holder
- 5 RS 232 connection to the analyzer
- 7 Programming switch (service only)
- 9 "sample in" analyzer gas connection
- 11 Adsorber cartridge (removes  $NO_x$  from the waste air)
- 2 Mains switch
- 4 Power connection
- 6 Service connection
- 8 Carrier gas connection (O<sub>2</sub>, synthetic/ purified air)
- 10 "out" sample outlet (gas)



## CAUTION

### Risk of poisoning due to ozone

The ozone generator contained in the device produces ozone  $(O_3)$ . When used in accordance with the intended use, the downstream ozone decomposer decomposes the poisonous gas. Various safety measures result in the automatic shut-down of the ozone generator. The following nevertheless applies:

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

Installation on the analyzer

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

Checking and extending the configuration

- Switch on the components of the analysis system. Start the software.
- Check the device configuration with the Instrument | Manage instruments menu option in the Manage instruments window.
- If necessary, change the device configuration or create a new device configuration for TN<sub>b</sub> determination with chemiluminescence detector (CLD):
  - Click on the Add button to create a new device configuration.
  - Edit the device configuration in the detail view Instrument configuration.
  - In the dropdown menu under N sensor: select option CLD.
- ▶ Click the button to save the device configuration.
- Click on **Set default** to activate the device configuration as standard configuration.

### 4.3.5 Integrated solids module

An integrated solids module, the Swab Test module, can be added to the combustion system of the analyzer. Small amounts of solid samples can be examined with the solids module, e.g., during cleaning validation.

The module achieves digestion temperatures of up to 950 °C. Sample digestion is performed by catalysts.

Up to 950 °C

0 to 500 mg

kPa

CeO<sub>2</sub> (special catalyst)

Manual, in boats via the lock

Oxygen ( $\geq$ 4.5), inlet pressure 400 to 600

Technical of	lata
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Layout

The integrated solids module consists of the following main components:

- Sample supply system
- Combustion system

Digestion temperature

Catalyst

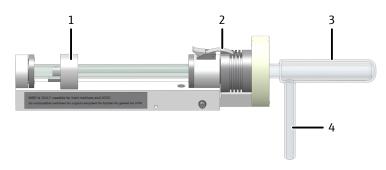
Sample volume

Carrier gas supply

Sample feed

Accessories

The module is connected to the combustion furnace of the analyzer with an adapter. The combustion tube for solids is inserted into the furnace for this.



### Fig. 43 Layout of the integrated solids module

1 Sample feed

- 2 Furnace lock with interlock
- 4 Gas outlet (for measuring gas)
- 3 Combustion tube, filled with catalyst

Sample supply	The solids module has a furnace lock with interlock. The furnace lock is mounted on the side opening of the combustion tube. The solid samples are weighed into boats and pushed into the combustion tube with the aid of the manual sample feed. The furnace lock can be opened and closed manually via the interlock.
Combustion system	The integrated solids module can only be used together with a combustion furnace for vertical and horizontal operation. The combined combustion furnace has two openings. The furnace can be operated with both a vertically installed combustion tube and the horizontally installed combustion tube.
	The combustion tube for solids consists of quartz glass. The furnace lock with manual feed is mounted on the side opening of the combustion tube. The gas hose is connected to the gas outlet. The gas hose is connected to the condensation coil in the analyzer with a fork clamp.
	The double-walled combustion tube is filled with catalyst and auxiliary material. As a catalyst, the special catalyst for multi N/C ( $CeO_2$ ) is used with a reaction temperature of up to 950 °C. The standard temperature setting is 900 °C.
Accessories	<ul><li>The following accessories are part of the supply scope:</li><li>Connection hoses</li><li>Tools</li></ul>

### 4.3.5.1 Installing the solids module



## CAUTION

### Risk of burns from the hot furnace, furnace head and combustion tube

• Switch off the device and allow it to cool down before installation and maintenance.



# CAUTION

### Skin and respiratory system irritation due to dust

Quartz wool and  $CeO_2$  special catalyst tend to form dust. Irritation can occur after breathing in or skin contact with this dust.

- Avoid the formation of dust.
- Wear protective clothing and gloves.
- Work under an extractor or wear a respiratory mask.



## NOTICE

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

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

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

1

Assemble the module as follows:

- Turn the gas outlet (4) of the combustion tube upward to fill.
- Fill quartz wool into the combustion tube via the large opening (1). Carefully push the quartz wool down with a glass rod and press it in place.
- ► Carefully fill approximately 60 g CeO<sub>2</sub> of the special catalyst into the sleeve of the combustion tube (2) via the gas outlet.
- Close the gas outlet with some quartz wool (3). The quartz wool is used to hold back the catalyst. Close the gas outlet so that no catalyst can enter the gas pathway. Do not pack the quartz glass wool in too tightly.
- Open the furnace lock. To do this, push the interlock (1) upwards.
- Pull the clip (2) out of the anchoring.
- Pull the furnace lock open to the left.



2

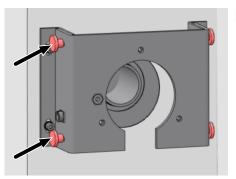
• 3

2

- Unscrew the three hexagon socket screws by half of a rotation with the right-angle screwdriver. Do not completely unscrew the screws.
- Push the filled combustion tube into the module until it hits the stop on the inner ring. The gas outlet must then point downward.
- Tighten the screws.
- Close the furnace lock again.

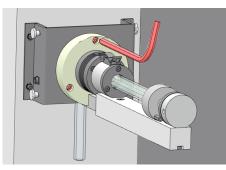
Mount the module on the analyzer as follows:

- Remove the combustion tube for vertical operation.
- Remove the sealing plug from the horizontal opening of the combustion furnace. Place the plug on the vertical opening of the furnace (see image).

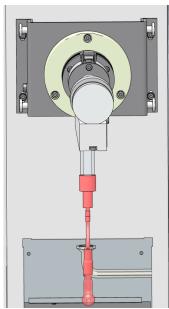


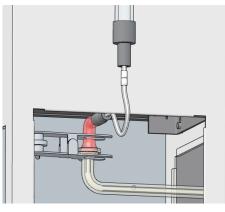
ی ک

• Attach the holding plate to the angular profiles in front of the horizontal opening of the combustion furnace with the four knurled head screws.

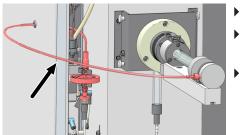


- Insert the module into the horizontal opening of the furnace. The gas outlet of the combustion tube points downward.
- Fasten the module to the holding plate with three hexagon socket screws.
- Fasten the gas hose to the gas outlet of the combustion tube.

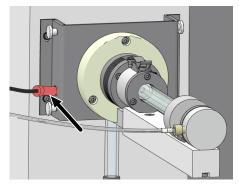




- Connect the gas hose and the inlet of the condensation coil.
- Secure the spherical joint connection with the forked clamp. Tighten the knurled head screw on the fork clamp hand-tight.



- Route the carrier gas hose through the opening in the rear wall.
- Connect the carrier gas hose to the top outlet of the water traps on the gas box with the FAST connector.
- Screw the other hose end to the module with the Fingertight connection.



• Connect the plug-in connector of the analyzer to the left side of the module.

The software detects that an integrated solids module is connected to the analyzer via the interface and sets the gas flows accordingly, for example.

- Close the side wall of the analyzer again.
  - Unscrew the knurled head screws and open the middle recess in the side wall.
  - Carefully guide the side wall over the solids module up to the basic device.
  - Apply the grounding conductor.

### See also

■ Removing the combustion tube [▶ 86]

# 5 Operation

## 5.1 General notes



## WARNING

### Risk of chemical burns from concentrated acids

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

- Wear safety goggles and protective clothing when handling concentrated acids. Work under an extractor.
- Observe all instructions and specifications in the safety data sheets.
- When analyzing samples with high acidic or saline content, aerosols can form in the TIC condensation vessel. The capacity of the halogen trap is then depleted relatively quickly. The water trap also clogs up quickly. Both components have to be replaced frequently if this is the case. If possible, dilute such samples before measurement, for example 1:10. Alternatively, use a smaller volume of sample.
- When significant aerosol formation occurs, the analyzer is immediately protected by the integrated aerosol trap (water trap) and the carrier gas supply is automatically interrupted. Additionally, to protect the analyzer, remove the hose of the water trap on the front side.
- To acidify samples, use analytically pure acid (HCI (2 mol/l)) and make it out of concentrated acid and TOC water.
- For TIC detection, only use orthophosphoric acid (H<sub>3</sub>PO<sub>4</sub>, 10 %) made from concentrated acid (p.a.) and TOC water.
- Solutions made from the following are suitable as standard solutions: Potassium hydrogen phthalate, sodium carbonate/sodium hydrogen carbonate, sucrose.
- Only clean, particle-free glass containers (volumetric flasks, sample vessels) may be used for the preparation and storage of the solutions.
- When preparing and storing solutions with very low concentrations (<1 mg/l), observe that the laboratory air components (CO<sub>2</sub>, organic vapors) can change the solution concentrations. The following measures can remedy this:
  - Keep the free space above liquids, the so-called headspace, as small as possible.
  - During autosampler operation, cover the vessels on the sample tray with foil.
     This is important in particular with differential mode, as the samples remain on the sample tray for a longer time.
  - Eliminate the source of organic vapors.
  - Optionally: Fill the headspace above the samples with inert gas.

## 5.2 Switching on the analyzer



## NOTICE

### Risk of device damage due to depleted copper wool

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

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

The software can support you with a checklist for the daily start of the analysis system. To do this create the checklist under **Program** | **Settings** in the **Instrument initialization** section.

Before switching on the analyzer, check the following:

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

Prepare the sample and switch on the analyzer as follows:

- Open the valve on the pressure reducer of the gas supply.
- Switch on the PC.
- Switch on the components of the analysis system.
- Finally, switch on the analyzer at the main switch. The analyzer is ready for operation when the status LED on the left front door lights up green.
- Start the software using the Windows start command Start | multiWinPro or by double clicking on the software icon on the desktop.
- ▶ In the login window enter the user name and password. Click **OK** to confirm the entered data.
- Initialize the analysis system by clicking on the Initialize instrument button in the Instrument controls panel.

If you activate the **Auto-Initialization on start** option under **Program** | **Settings** the software will automatically initialize the analysis system when the software starts.

- ✓ The software initializes the analysis system and activates the standard configuration.
- Use the Instrument | Manage instruments menu option to change the device configuration if needed. Activate the desired device configuration by clicking on the Set default button or by double-clicking.
- Wait for the end of the warming-up phase (30 min).
- The analysis system is not ready for measurement after the warm-up phase if components in the **Instrument status** panel are displayed in color. If so, start troubleshooting. First check the hoses for tight fit.

Set the purge flow for NPOC measurements. To do this, use the menu option Instrument | Single control steps | Purge to activate the purge flow.

Set the gas flow at the "NPOC" needle valve.

- Adjust the autosampler after each modification. To do this, open the Sampler alignment window using the menu option Instrument | Sampler alignment.
  - ✓ The analysis system is ready for measurement.

#### See also

■ Troubleshooting [▶ 102]

### 5.3 Switching off the analyzer

#### Standby

Switch the analyzer system to standby for measurement breaks of  $\geq$ 30 min, for example while you are evaluating measurement results or overnight.

In standby mode, the software switches off the gas flow and lowers the oven temperature to the standby temperature.

- Select the **Instrument** | **Standby** menu option.
- Or: In the Instrument controls panel click on the Instrument standby or switch off button.
  - In Standby select the option Standby .
  - Set the standby temperature in [°C].
- Exit the dialog with **OK**.
  - ✓ The software stays open. The analysis system will be put in standby mode.

### Switching off Switch off the analysis system before long periods of inactivity, e.g. at weekends or during vacations.

The software switches off the gas flow and pumps out the TIC condensate container. The oven cools down to room temperature.

- Select the **Program** | **Close** menu option.
- Or: Shut down the software using the imes icon (top right).
- Or: Select the **Instrument** | **Switch off** menu option.
- Or: In the Instrument controls panel click on the Instrument standby or switch off button.
- In Standby select the option Switch off.
- Exit the dialog with **OK**.
  - ✓ The software shuts down. The analysis system shuts down. You can now switch off the components of the analysis system at their main switches.

Standby / switch off at end of measurement

At the end of a sequence, you can automatically shut down the analysis system or put it into standby. For example, they can save gas and energy when measuring overnight.

- Use the **Measurement** | **Add new sequence** menu option to create a new sequence.
- Standby: At the end of the sequence use the Add control step button to set the Standby instrument control step. Set the standby temperature in the Step properties panel.

- If necessary, use the **Wake up** control step to make the analysis system ready for operation again at the desired time.
- Switching off: Set the control step **Turn off instrument** at the end of the sequence.

### 5.4 Performing measurements

### 5.4.1 Create sequence and measure with manual sample feed

Preliminary considerations:

- Blank values change over time. You should therefore decide whether to re-measure blank values at the start of the sequence.
- If necessary, you can correct the calibration with a daily factor. To do this, measure one or more standard solutions at the beginning of the sequence to determine the daily factor(s). The software automatically transfers the daily factors to the calibration.
- Prepare one or more methods for manual sample feed. To do this, activate the Manual measurement checkbox in method parameters.
   A sequence can contain sample steps with different methods. However, liquids and solids cannot be measured in a sequence.
- Alternatively: Wait to activate the Manual measurement checkbox until the sequence was created in method parameters.
- Use the **Measurement** | **Add new sequence** menu option to create a new sequence.
- In the Sequence properties panel activate the Is a solids measurement checkbox for manual solids measurement.
- By default, the software assigns a new sequence to the active device configuration. If

necessary, click on **C** to assign the empty sequence to another device configuration. To do this, select a device configuration in the **Select instrument configuration** window. Confirm the selection by clicking on the **OK** button.

- Alternatively, open an already prepared sequence. Open the Manage sequences window using the menu option Sequences | Sequences. Select prepared sequence from the Overview table. Open the sequence by double-click or with Load.
- Create measurement steps in sequence with **Add by method**.
- Select the method from the dropdown menu or in the **Add by method** window.
- Enter sample name in sequence table by double-click on measurement step or in the Step properties panel, Tab Step.
   The default name is: method type + step number.
   Optionally add a comment.
- If necessary, create several sample steps using the option Add multiple steps (in context menu).
  - Select the method in the window **Add multiple steps to sequence**.
  - Set the number of measurement steps under Count of steps:.
  - Choose a common base word for the designation of the steps under Base name:.
     The default name is: sample + method type.
  - Enter the start number in the **Use numbers:** input field to number the measurement steps.
  - Transfer the measurement steps to the sequence by clicking on **Create steps**.

In case of manually diluted samples, enter the dilution ratio under Dilution ratio numerator and Dilution ratio denominator: Parts of the primary sample in the total parts.

The software takes the dilution into account when calculating the results.

- If required, select one or more measurement steps in the sequence table and adjust the method settings in the **Step properties** panel to the measurement task.
- For each measuring channel, select the calibration for calculating the measurement results from the drop-down menu in the **Step properties** panel, Tab **Calibration**.
- View blank values for each measuring channel on the **Blanks** tab. Edit blank values if required.

The software automatically corrects the measurement results for any blank values. Unless you redefine the blank values at the start of the sequence, the software uses the last blank values.

- The software creates measurement steps with sample type **Sample**. Select measurement step and after clicking on the **Sample type** button, select other sample type, such as **Daily factor**, from the dropdown menu.
- Optionally specify lower and upper limit value for the measurement result in the Step type properties panel. Select actions from the dropdown menu if the limit is exceeded, such as **cancel** for measuring stop.
- Select result table from dropdown menu after clicking on **Result table**. Or: Create a new result table with Create new result table. Unless you select a result table, the software saves the results in the default result table. For default setting see: Program | Settings | Result table

**1** NOTICE! It is not possible to start the measurement without a results table.

- ▶ Check the finished sequence for plausibility by clicking on 🐱. The software checks whether the created measuring steps can be measured.
- If necessary, save the sequence with 🛃. Set the name for the sequence in the Save **as** window and confirm with **OK**. The software names the window accordingly.
- Provide samples. For liquid measurements, dip the sample intake canula into the sample. For NPOC measurements, also insert a purge canula into the sample.
- Before starting the measurement: Check device readiness in the Instrument status panel.
- ▶ Start the measurement by clicking on ▶. Follow the instructions on the screen.
  - ✓ The analysis system processes the sequence. You can add further steps to the sequence during the measurement.

The software displays the current measurement results during recording graphically in the lower window area and in a result table.

In the Step results panel you can view the results of already measured samples. When the sequence was processed, you can see the results in the **Results** menu.

#### 5.4.2 Creating a sequence and measuring with automatic sample feed

Preliminary considerations:

- Blank values change over time. You should therefore decide whether to re-measure blank values at the start of the sequence.
- If necessary, you can correct the calibration with a daily factor. To do this, measure one or more standard solutions at the beginning of the sequence to determine the daily factor(s). The software automatically transfers the daily factors to the calibration.

- Prepare one or more methods for the measurement. A sequence can contain measurement step with different methods. However, liquids and solids methods cannot be measured in a sequence.
- Provide samples on sample tray.
- Use the **Measurement** | Add new sequence menu option to create a new sequence.
- Make cross-sequence settings in the Sequence properties panel: Solids measurement, automatic or intelligent dilution, intelligent reduction of the sample volume and parallel purging in NPOC methods. To do this, activate the corresponding checkbox. The available options depend on the device configuration.
- By default, the software assigns a new sequence to the active device configuration. If

necessary, click on **C** to assign the empty sequence to another device configuration. To do this, select a device configuration in the **Select instrument configuration** window. Confirm the selection by clicking on the **OK** button.

- Alternatively, open an already prepared sequence. Open the Manage sequences window using the menu option Sequences | Sequences. Select prepared sequence from the Overview table. Open the sequence by double-click or with Load.
- Create measurement steps in sequence with **Add by method**.
- Select the method from the dropdown menu or in the **Add by method** window.
- Enter sample name in sequence table by double-click on measurement step or in the Step properties panel, Tab Step.
   The default name is: method type + step number.
   Optionally add a comment.
- If necessary, create several sample steps using the option Add multiple steps (in context menu).
  - Select the method in the window Add multiple steps to sequence.
  - Set the number of measurement steps under **Count of steps**.
  - Choose a common base word for the designation of the steps under Base name:.
     The default name is: sample + method type.
  - Enter the start number in the Use numbers: input field to number the measurement steps.
  - Transfer the measurement steps to the sequence by clicking on **Create steps**.
- The software creates measurement steps with sample type Sample. Select measurement step and after clicking on the Sample type button, select other sample type, such as Daily factor, from the dropdown menu.
- Determine position on sample tray under Step properties | Tab Step under Sample position.

You can occupy positions on the autosampler tray more than once in a sequence.

- If required, select one or more measurement steps in the sequence table and adjust the method settings in the **Step properties** panel to the measurement task.
- In case of manually diluted samples, enter the dilution ratio under Dilution ratio numerator and Dilution ratio denominator: Parts of the primary sample in the total parts.

The software takes the dilution into account when calculating the results.

• For each measuring channel, select the calibration for calculating the measurement results from the drop-down menu in the **Step properties** panel, Tab **Calibration**.

View blank values for each measuring channel on the Blanks tab. Edit blank values if required.

The software automatically corrects the measurement results for any blank values. Unless you redefine the blank values at the start of the sequence, the software uses the last blank values.

- Optionally specify lower and upper limit value for the measurement result in the Step type properties panel. Select actions from the dropdown menu if the limit is exceeded, such as cancel for measuring stop.
- Click on the Add control step button to add control steps such as pauses or additional rinsing steps to the sequence.
- Add the control steps **Reverse rinse**, **Standby** or **Turn off instrument** at the end of the sequence in order to shut the analysis system down after sequence processing.
- Select result table from dropdown menu after clicking on Result table. Or: Create a new result table with Create new result table.
   Unless you select a result table, the software saves the results in the default result table. For default setting see: Program | Settings | Result table

**1** NOTICE! It is not possible to start the measurement without a results table.

- Check the finished sequence for plausibility by clicking on to . The software checks whether the created measuring steps can be measured.
- ► If necessary, save the sequence with <sup>L</sup>. Set the name for the sequence in the Save as window and confirm with OK. The software names the window accordingly.
- Before starting the measurement: Check device readiness in the **Instrument status** panel.
- ▶ Start the measurement by clicking on ▶.
  - $\checkmark$  The analysis system processes the sequence. You can add further measurement or control steps to the sequence during the measurement.

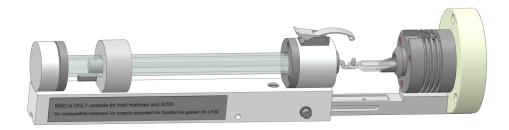
The software displays the current measurement results during recording graphically in the lower window area and in a result table.

In the **Step results** panel you can view the results of already measured samples. When the sequence was processed, you can see the results in the **Results** menu.

### 5.5 Operating the integrated solids module

Preparing for the measurement	•	Check that the solids module has been properly installed before switching on the analyzer. Check that the correct carrier gas (oxygen, $\geq$ 4.5) is connected.
	•	Switch on the analyzer. The carrier gas flow is automatically set to 390 to 410 ml/min as soon as a solids method is loaded.
		Check the system for leaks.
Preparing the sample boats	The sample boats may be contaminated. Temper the sample boats prior to analyz- ing standard solutions and samples. Tempering is performed by an "empty measure- ment".	
	•	After tempering, do not touch the sample boat by hand any more. Store the boat in a clean vessel, e.g., a petri dish. Transport the boat with a clean pair of tweezers.

- The sample material can have a blank value as well. The pharmaceutical industry tests the effectiveness of cleaning processes by using swabs. The swab can be tempered on a boat before wiping. The blank value of the swab material can also be determined and taken into account. Fold the swab using a pair of tweezers until it can be placed on the boat. The swab must only protrude slightly from the boat. Performing the analysis Only measurements with manual sample feed are possible with the integrated solids module. Create device configuration for the measurement with integrated solids module: Select the option Internal horizontal from the dropdown menu at Furnace type:. Save and activate the device configuration as standard configuration by clicking on the Set default button. Create method for solids analysis in the Manage methods window with Add TC method. In the Method detail view activate the Method is for solid measurement and Manual measurement checkboxes. • Adjust the furnace temperature under **Furnace temperature** to 900 °C. • Use the **Measurement** | **Add new sequence** menu option to create a new sequence. In the Sequence properties panel activate the checkbox Is a solids measurement. • Create measurement steps by clicking on Add by method. For each measurement step enter the sample name in the Step properties | Step panel under Name. • Enter the sample mass [µg] under **Sample mass**. You can add further measurement steps to the sequence and edit the sample mass during the measurement.
  - Select the result table to save the results after clicking on the **Result table** button.
  - Start the measurement by clicking on  $\blacktriangleright$  .
  - After the prompt from the software, insert the sample boat in the furnace lock.
    - Open the furnace lock.
    - Insert the sample boat in the furnace lock. Hook the eye of the boat into the hook on the feed (see image).
  - Acknowledge sample feed.
  - Follow the software instructions and close the lock again.
  - Push the boat into the combustion furnace with the feed.



### Fig. 44 Inserting the sample boat in the solids module

 For multiple determinations: Start a second measurement with new sample material by clicking on ▶.

- ✓ At the end of the measurement, you can view the measurement results in the result table and generate a report.
- Open the Manage result tables window with the Result details | Manage result tables menu option.
- Select result table and load using the **Load** menu option or by double-clicking.

### See also

B Checking the system for leaks [▶ 85]

# 6 Maintenance and care

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

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

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

### 6.1 Maintenance overview

#### Analyzer

Maintenance interval	Maintenance task	
Weekly	<ul><li>Clean and service the device.</li><li>Clean the reagent bottle and the drip tray.</li><li>Check the fastening screws for proper fit.</li></ul>	

### Sample supply system and autosampler

Maintenance interval	Maintenance task	
Quarterly	<ul> <li>Check the syringe pump for leaks.</li> </ul>	
Every 12 months	<ul> <li>Clean the dosing syringe (earlier if required).</li> </ul>	
As required	<ul> <li>After initial start, change of the sample tray or recommis- sioning after transport and storage: Adjust the autosampler.</li> </ul>	

### Hose system

Maintenance interval	Maintenance task	
Daily	<ul> <li>Check the gas flow display in the <b>Instrument status</b> panel.</li> </ul>	
Weekly	<ul> <li>Check the hose connections for proper fit.</li> </ul>	
Quarterly	Check the condensate and phosphoric acid pumps for leaks.	
Every 12 months	<ul> <li>Replace the pump hose.</li> </ul>	

### **Combustion system**

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

### Measuring gas drying and cleaning

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

### Integrated Swab Test solids module

Maintenance interval	Maintenance task	
Quarterly	<ul><li>Check the combustion tube for cracks and damage.</li><li>Check the furnace lock for leaks.</li></ul>	
Every 12 months	<ul> <li>Clean the combustion tube (earlier if required)</li> </ul>	
When necessary	<ul> <li>After software notification at the latest: Check the catalyst. Replace as necessary.</li> <li>Replace a worn sealing ring of the lock.</li> </ul>	

### Chemiluminescence detector (CLD)

Maintenance interval	Maintenance task	
Every 12 months	<ul> <li>Replace the adsorber cartridge.</li> </ul>	

## 6.2 Adjustment and setting

### 6.2.1 General notes for adjusting the autosampler

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

An adjustment of the autosampler is necessary:

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

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

#### See also

Installing and commissioning the sampler [▶ 43]

### 6.2.2 Adjusting the AS vario autosampler

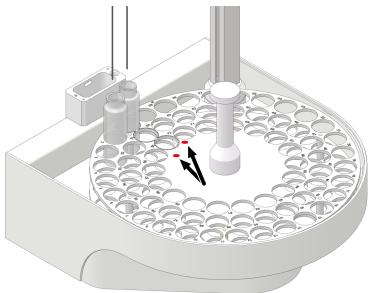


## NOTICE

### **Risk of bending**

The canula may bend during adjustment.

- Unscrew the screw connections on the canulas before adjustment.
- Start the software
- Use the Instrument | Sampler alignment menu option to open the Sampler alignment window.
- Aligning the canulas:
- Select adjustment position **Needle** from the list box in the **Sampler position** section.
- Click on the **Request current values** button to retrieve the current offset values.
- Adjust the canulas with higher / + lower until they stand about 2 cm above the adjustment points.
- After each change, click **Move** to check the adjustment.
- Align the canulas to the two adjustment points by carefully bending them.



### Fig. 45 Adjustment points on the sample tray

• Adjust the immersion depth of the sample intake canula into the wash cup and into a sample vessel in position 1 of the sample tray:

Sampler adj	ustment	Samp	oler adjustment				AS Vario[72] at Mu	ıltiNC 3300
Sampler Lo	cations			Move to positio	n			
Position	Offset X	Offset Y	Offset Z	Select position :	1	÷ 🛛	Move	
Position 1 Rinse	0 mm 0 mm	0 mm 0 mm	139 mm 132 mm	Waste position :	0		Move	
Canula	0 mm	0 mm	132 mm	Acid position :			Move	
position1	Reque	st current offsets						
- higher / + lo	ower 0	\$	139					
Move		Commit	Cancel					

#### Fig. 46 Sampler alignment window

- At first, select the adjustment position **Rinse** from the list box in the **Sampler posi**tion section.
- Modify the canula immersion depth via up-down control higher / + lower until the canula dips at least 1 cm into the wash cup. Click on Move after each modification.
- For the AS vario ER autosampler: Lower the canula as far as possible into the wash cup so that the canula is sufficiently flushed with ultrapure water.
- After adjustment save offset values by clicking on **Confirm**. Close the window.
- Select adjustment position **Position 1** from the list box in the **Sampler position** section.
- Place a sample vessel with magnetic stirring rod on position 1 of the sample tray.
- Lower the cannulas to position 1 using the higher / + lower up-down control into the sample vessel until the stirring rod can still rotate unhindered (distance approx. 5 mm).
- After adjustment save offset values by clicking on **Confirm**. Close the window.
- Select positions Position 1 and Waste position: in the Move to position section and approach by clicking on Move.
  - ✓ The autosampler is adjusted.

#### Adjusting the autosampler for automatic acidification

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

- Open Sampler alignment window.
- Adjust the adjustment position **Position 1**.
- Place a sample vessel on the acid position. For the acid position see: Acid position in the Move to position section.
- Approach position by clicking on **Move** and check it.
- Apply offset values by clicking on **Confirm**.
- Check offset values by NPOC test measurement with automatic acidification.
- Ensure the canula goes through the sample lid but does not immerse in the sample liquid during acidification.

### 6.2.3 Adjusting the EPA Sampler



## NOTICE

### **Risk of bending**

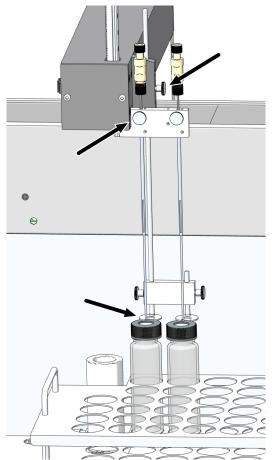
The canula may bend during adjustment.

• Unscrew the screw connections on the canulas before adjustment.

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

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

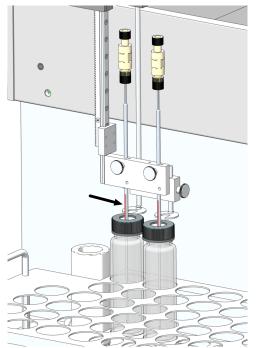
For sample vessels with septum caps, special sample intake and purging canulas with piercing function are required: Piercing needles with ventilation slot.



#### Fig. 47 Install canulas (here: 2 canulas for parallel purging)

- Install the hold-down clamp and the sample intake canulas in the canula holder. Unscrew the retaining screws of the canulas before adjustment. Clamp the canulas into the holder so that the canula tip does not immerse in the sample vessel.
- Use the Instrument | Sampler alignment menu option to open the Sampler alignment window.
- Adjust the immersion depth of the sample intake canula into the wash cup and into a sample vessel in position 1 of the sample tray.

- Select adjustment position **Position 1** from the list box in the **Sampler position** section.
- Click on the **Request current values** button to retrieve the current offset values.
- Change offset values in 0.1 mm steps using the up-down control backwards / + forwards , left / + right and higher / + lower.



#### Fig. 48 Adjusting position 1

- First adjust position 1 without sample vessel. Place the magnetic stirring rod in position 1 on the sample tray.
- Align canula with up-down control backwards / + forwards and left / + right to make it stand centrally over Position 1.
- Insert a sample vessel with a screw cap and a septum cap, e.g., an EPA sample vessel, on Position 1 in the sample tray.
- Adjust the immersion depth of the special needle using up-down control higher / + lower until approximately 2 cm of the ventilation slot can be seen above the septum. The ventilation slot must be located above and below the septum. No pressure compensation is otherwise possible in the sample vessel.
- After adjustment save offset values by clicking on **Confirm**. Close the window.
- Select adjustment position **Rinse** from the list box in the **Sampler position** section.
- Adjust the canula at the rinse position so that the canula is immersed in the center of the wash cup.
- Adjust the immersion depth of the special canula so that the ventilation slot can be seen at the upper edge of the wash cup.
- After each change, click **Move** to check the adjustment.
- After adjustment save offset values by clicking on **Confirm**. Close the window.
- Select positions Position 1 and Rinse in the Move to position section and approach by clicking on Move.
  - ✓ The autosampler is adjusted.

### Adjusting the autosampler for automatic acidification

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

- Open Sampler alignment window.
- Adjust the adjustment position **Position 1**.
- Place a sample vessel on the acid position. For the acid position see: Acid position in the Move to position section.
- Approach position by clicking on **Move** and check it.
- Apply offset values by clicking on **Confirm**.
- Check offset values by NPOC test measurement with automatic acidification.
- Ensure the canula goes through the sample lid but does not immerse in the sample liquid during acidification.

### 6.2.4 Setting the NPOC purge flow



## CAUTION

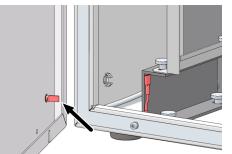
### Risk of burns from the furnace

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

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

The NPOC purge flow is preset to approx. 170 to 190 ml/min. Depending on the measurement task, you can increase or decrease the NPOC purge flow via the NPOC needle valve. The NPOC needle valve is located behind the left side wall, to the left of the combustion furnace.

Set the NPOC purge flow as follows:



#### Fig. 49 Grounding conductor connection on the side wall

- Open the left side wall of the analyzer. Push the accessory modules to the side if necessary. Do not kink any connection hoses.
  - Unscrew the four attachment screws. The screws are captive and remain attached to the wall.
  - Remove the protective grounding. Set the side wall aside safely.
- Open the **Single control steps** window with the **Instrument** | **Single control steps** menu option.
- For sample feed with autosampler: Select a random position on the sample tray in the **Sample purge** section at **Sample position** on which to observe the purge flow.

- Place a sample vessel with ultrapure water at this position.
- For manual sample supply: Insert purge hose 7 in a sample vessel filled with ultrapure water.
- Set the purge time at **Purge time**: 1 to 900 s.
- Click on **Purge**.
- Unscrew the adjustment screw on the NPOC needle valve.
- Set the desired NPOC purge flow:
  - Increase the NPOC purge flow: Turn the needle valve to the left.
  - Decrease the NPOC purge flow: Turn the needle valve to the right.
- Check the flow display in the **Instrument status** panel while doing this. The current NPOC purge flow is displayed with **Purge:**.
- Screw the adjustment screw on the needle valve back in.
- Close the side wall.
  - Connect the protective grounding to the left side wall.
  - Slightly tighten the screws first on the bottom side and then on the top side. Tighten the screws in turns.

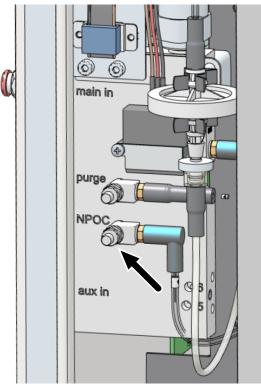
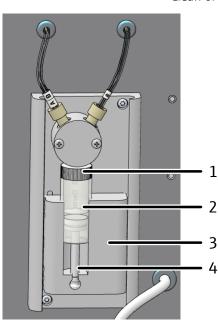


Fig. 50 Setting the NPOC purge flow

## 6.3 Syringe pump maintenance



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

- Open the doors of the analyzer.
- Open the window of the same name with the Instrument | Single control steps menu option.
- In the Move syringe to change position section click on the Syringe change position button.
  - $\checkmark$  The syringe is emptied and moved to the replacement position.
- Unscrew the dosing syringe on the valve (1) and remove it from the drive (3).
- Dismantle and clean the glass cylinder (2) and piston (4).
- Insert the piston rod of the new dosing syringe into the drive.
- Screw the glass cylinder onto the valve.
  - ✓ The analyzer is ready for operation again.

## 6.4 Replacing the pump hose



## CAUTION

### Risk of chemical burns during hose replacement

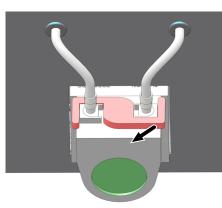
Small quantities if acidic solutions can still be in the hoses.

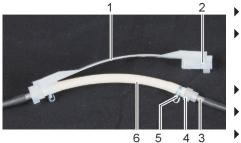
- Wear protective gloves and clothing when replacing the hoses.
- Collect any leaking liquids with an absorbent sheet.

Check the pump hoses every 3 months for leaks and replace them after12 months at the latest.

### Condensate pump

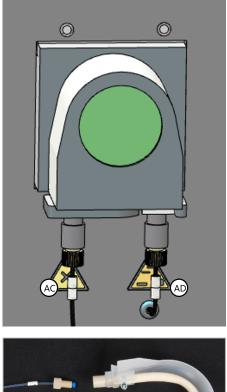
- Shut down control and evaluation software or turn off the gas flow with the Instrument | Gas flow off menu option.
- Open the doors of the analyzer.
- Press the bracket on the condensate pump to the left.
- Pull hoses 10 and 11 off of their connections.





- 1 Guide piece
- 2 Groove
- 3 Metal connection
- 4 Hose guide
- 5 Hose clamp
- 6 Pump hose
- (11)(10) $\bigcirc$

- Remove the guide piece with the pump hose from the pump body.
- Check the pump hose and the connections for excessive wear and cracks. If moisture escapes the pump hose or the connections, replace the pump hose.
- Wipe the pump body and the roller carrier with ultrapure water.
- Check the pump body and roller carrier for wear.
- Press the still-intact or new pump hose back into the guide piece. Align the hose clamps downward during installation.
- Insert the hose guide in the groove of the guide piece.
- Position the guide piece around the pump body.
- Press the guide piece upward with one hand. Turn the clip to the right until it engages with the other hand.
- Push hoses 10 and 11 back onto their adapters.
- Switch on the gas supply again and check the system for leaks.
  - ✓ The pump is once again ready for operation.



### Phosphoric acid pump

- Shut down control and evaluation software or turn off the gas flow with the Instrument | Gas flow off menu option.
- Remove the pump hose as with the condensate pump.



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

## 6.5 Replacing the hose connections

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



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



Thread the hose into the canula of the threading aid.



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

#### Angled FAST connectors

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

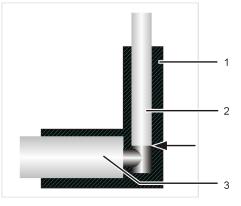


Fig. 51 FAST connector, angled

Angled FAST connector
 Glass connection

Fingertight connections

• When replacing Fingertight connections, only use straight cut, round, uncrimped hose ends.

2 Hose

- Slide the conical nipple onto the hose with the conical side towards the banjo bolt. The conical nipple and hose end must be flush.
- Do not jam the banjo bolt during insertion and only tighten it hand-tight.

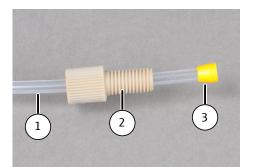


Fig. 52 Replacing the Fingertight connection

1 Hose

2 Banjo bolt

3 Conical nipple

## 6.6 Checking the system for leaks



## NOTICE

### Risk of gas leakage

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

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

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

- Switch on the analyzer.
- Open the carrier gas supply on the pressure reducer.
- Start the control and analysis software.
- Check the flow display in the **Instrument status** panel:
  - In: (inlet flow) 115 to 125 ml/min
  - Out: (outlet flow) 115 to 125 ml/min

## 6.7 Replacing the catalyst

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

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

Dispose of the catalyst in accordance with disposal regulations.

### See also

B Disposal [▶ 115]

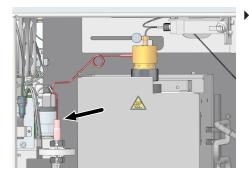
### 6.7.1 Removing the combustion tube



## CAUTION

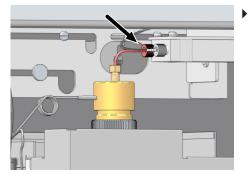
### Risk of burns from the hot furnace, furnace head and combustion tube

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



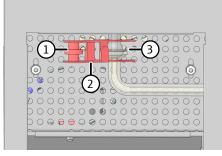
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Pull the carrier gas canula out of the FAST connector on the water trap on the gas box.



Unscrew the Fingertight connection of the furnace canula from the switching valve.

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



- Release the joint connection (3) on the bottom of the combustion furnace connecting the combustion tube with the condensation coil.
- To do this, unscrew the knurled head screw (1) and remove the fork clamp (2).
- Carefully pull the combustion tube out of the combustion furnace toward the top.
- Unscrew the furnace head from the combustion tube. Remove the union nut, the pressure ring and the three sealing rings.
- Remove the used catalyst filling. Check the combustion tube for heavy crystallization, cracks and burst spots. Only re-use intact combustion tubes.
- Thoroughly rinse the empty combustion tube with ultrapure water and dry it well.

### 6.7.2 Filling the combustion tube



## CAUTION

### Skin and respiratory system irritation due to dust

Quartz wool, HT mat and catalyst tend to form dust. Irritation can occur after breathing in or skin contact with this dust.

- Avoid the formation of dust.
- Wear protective clothing and gloves.
- Work under an extractor or wear a respiratory mask.



## NOTICE

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

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

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



## NOTICE

### Risk of damage due to emission of gas

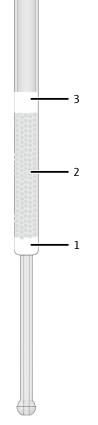
The catalyst can emit gas during initial heating, this can be seen as mist formation in the TIC condensate container.

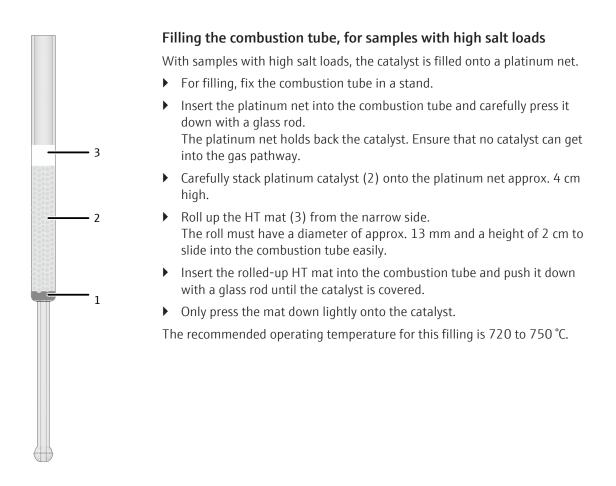
- Allow the catalyst to burn out during initial heating for approximately 30 min at operating temperature.
- During this, interrupt the gas flow at the water traps on the front side to protect the water traps from becoming full.

### Filling the combustion tube, for normal samples

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

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





### 6.7.3 Installing the combustion tube



## NOTICE

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

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

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



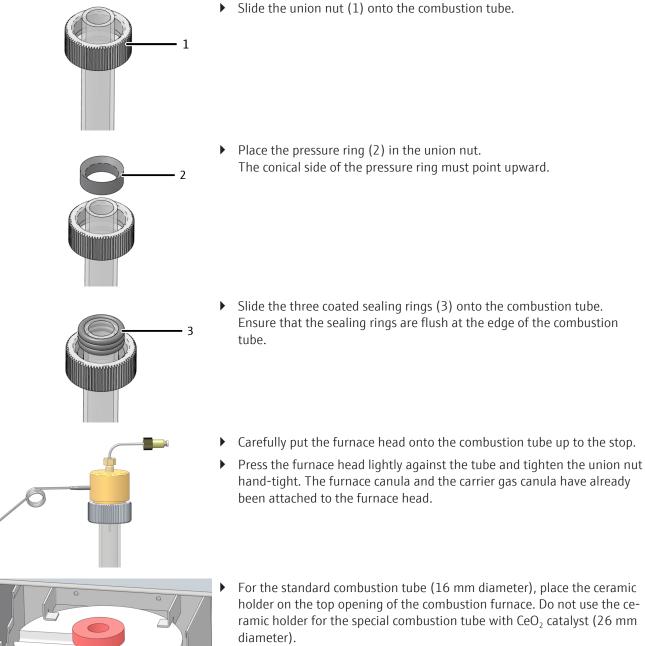
## NOTICE

### Preventing tightness problems

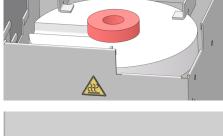
Due to slight variation in the outer diameter of the combustion tubes, a new combustion tube may not be able to be installed tightly with previously used O-rings.

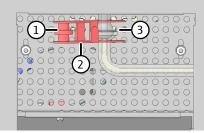
• When installing a new combustion tube, always use new O-rings (402-815.102).

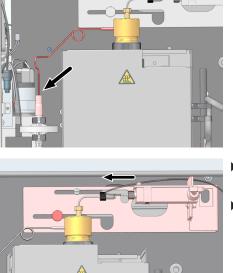
Install the furnace head on the combustion tube before inserting the combustion tube into the furnace.



- For the standard combustion tube (16 mm diameter), place the ceramic holder on the top opening of the combustion furnace. Do not use the ce-
- Insert the combustion tube with the furnace head into the combustion furnace.
- Connect the lower end of the combustion tube and the inlet of the con-densation coil via the spherical joint connection (3).
- Secure the spherical joint connection with the forked clamp (2). Tighten ▶ the knurled head screw (1) hand-tight.

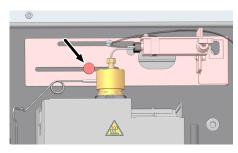




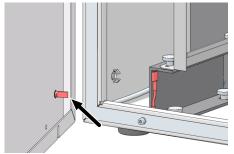


Connect the carrier gas connection with the connection on the gas box with the FAST connector.

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



- Fix the valve in this position. Tighten the knurled head screw on the holder finger-tight to do so.
- Place the top cover on top of the analyzer.



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

## 6.8 Removing and installing the combustion furnace

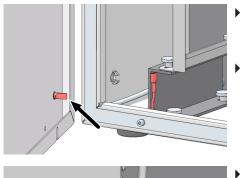
### 6.8.1 Removing the combustion furnace

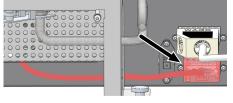


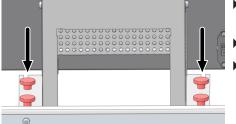
## CAUTION

### Risk of burns from the hot furnace, furnace head and combustion tube

• Switch off the device and allow it to cool down before installation and maintenance.





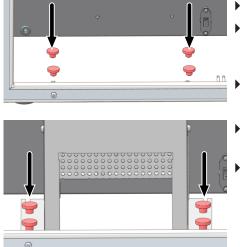


- Switch off the analyzer via the main switch. Disconnect the power plug from the socket. Shut off the gas supply on the pressure reducer in the laboratory.
- Open the left side wall of the analyzer. Push the accessory modules to the side if necessary. Do not kink any connection hoses.
  - Unscrew the four attachment screws. The screws are captive and remain attached to the wall.
  - Remove the protective grounding. Set the side wall aside safely.
- Remove the top cover.
- Remove the combustion tube. Slide the change-over valve to the right to prevent it from interfering with removal.
- Remove the condensation coil.
- Pull the plug-in connector for the combustion furnace out of its socket.
- Unscrew the knurled head screws the furnace is fastened to the device bottom with.
- Lift the furnace out of the analyzer.
- Reattach the knurled head screws to the device bottom so they are not lost.

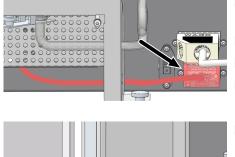
### See also

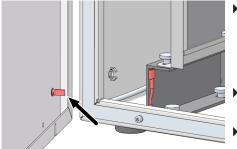
- Removing the combustion tube [▶ 86]
- B Condensation coil maintenance [▶ 94]

### 6.8.2 Installing the combustion furnace



- Open the left side wall of the analyzer. Remove the top cover.
- Loosen the knurled head screw on the holder of the change-over valve.
   Slide the change-over valve to the right to prevent it from interfering with installation.
- Unscrew the knurled head screws on the device bottom the furnace is fastened with.
- Insert the furnace in the middle. Align the front side of the furnace to be parallel with the device wall.
- ▶ Fasten the furnace with the knurled head screws. Tighten the knurled head screws finger-tight.





- Plug the plug-in connector for the combustion furnace into the socket at the bottom right of the rear device wall.
  - Install the combustion furnace.
- Install the condensation coil.
- Push the sample intake hose and the purging hose through the top opening. Attach the top cover. Connect the hoses to the autosampler.
- Close the side wall.
  - Connect the protective grounding to the left side wall.
  - Slightly tighten the screws first on the bottom side and then on the top side. Tighten the screws in turns.
- Open the gas supply. Connect the power plug with the socket and switch on the analyzer via the main switch.
- Check the system for leaks.
  - ✓ The analyzer is ready for operation again.

## 6.9 Cleaning the TIC condensate vessel



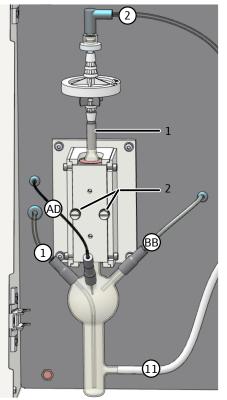
## WARNING

### Risk of chemical burns from phosphoric acid

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

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

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



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

## 6.10 Condensation coil maintenance

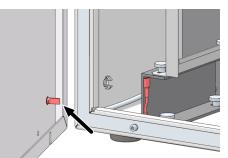
### Removal and cleaning



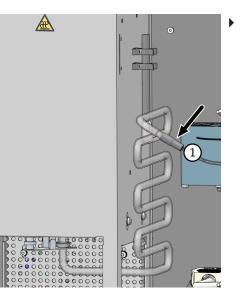
## CAUTION

### Risk of burns from the hot furnace, furnace head and combustion tube

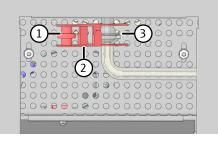
• Switch off the device and allow it to cool down before installation and maintenance.



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

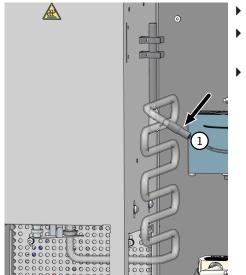


Pull hose 1 out of the FAST connector of the condensation coil.

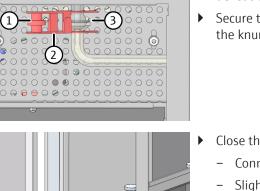


- Release the joint connection (3) on the bottom of the combustion furnace connecting the combustion tube with the condensation coil.
- To do this, unscrew the knurled head screw (1) and remove the fork clamp (2).
- Carefully remove the condensation coil from the analyzer. Pull the bottom part out of the opening of the combustion furnace when doing this.
- Remove the FAST connector from the glass connection of the condensation coil.
- Inspect the condensation coil for deposits and cracks.
- Rinse the condensation coil with ultrapure water and dry it well.

### Installation



- Slide hose 1 into the FAST connector.
- Slide the FAST connector onto the glass connection of the condensation coil.
- Mount the condensation coil in the holder on the side of the furnace. The spherical joint of the coil points toward the lower opening of the furnace.



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

Close the side wall.

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

## 6.11 Replacing the water traps

െ

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

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

Check the system for leaks after replacing the water traps.

Water traps on the front side

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

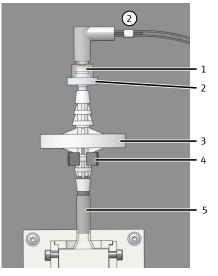


Fig. 53 Replacing the water traps on the front side

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

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

Water traps on the gas box

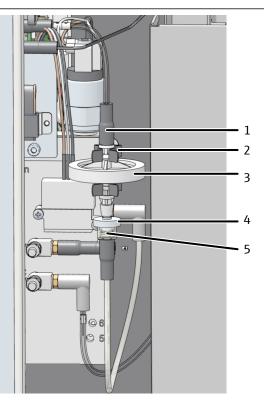
Two water traps are installed in front of the gas box (prefilter and disposable retention filter). They protect the gas box from aerosols and rising water in case of incorrect gas pressures. The left side wall of the analyzer must be opened to replace the water traps.



## CAUTION

### Risk of burns from the hot furnace

• Switch off the device and allow it to cool down before installation and maintenance.



#### Fig. 54 Replacing the water traps on the gas box

- 1 FAST connector
- 3 Prefilter (aerosol trap)
- 5 Luer connection

- 2 Clamp on the gas box
- 4 Disposable retention filter
- Exit the control and analysis software.

- Switch off the analyzer using the power switch. Disconnect the power plug from the socket. Allow the analyzer to cool down.
- Open the left side wall of the analyzer. Push the accessory modules to the side if necessary. Do not kink any connection hoses.
  - Unscrew the four attachment screws. The screws are captive and remain attached to the wall.
  - Remove the protective grounding. Set the side wall aside safely.
- Pull the water traps out of the two clamps on the gas box.
- Pull the upper FAST connector off of the water traps.
- Remove the water traps from the Luer connection.
- Assemble the new water traps:
  - The "INLET" marking on the large water trap (aerosol trap) must face upward.
  - The labeling on the small water trap (disposable retention filter) must face downward.
- Connect the large water trap with the upper FAST connector.
- Connect the small water trap to the Luer connection on the bottom.
- Press the water traps into the clamps on the gas box.
- Close the side wall.
  - Connect the protective grounding to the left side wall.
  - Slightly tighten the screws first on the bottom side and then on the top side. Tighten the screws in turns.
- Connect the power plug with the socket and switch on the analyzer again via the main switch.
- Check the system for leaks.
  - $\checkmark$  The water traps on the front side and the gas box are replaced.

#### See also

B Checking the system for leaks [▶ 85]

### 6.12 Replacing the halogen trap



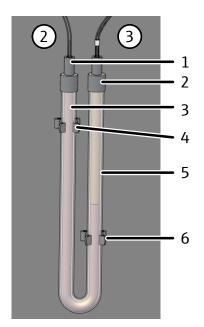
## NOTICE

### Risk of device damage due to depleted copper wool

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

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

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



### Fig. 55 Replacing the halogen trap

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

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

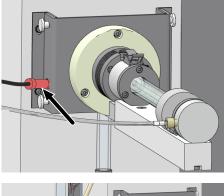
### 6.13 Removing the integrated solids module



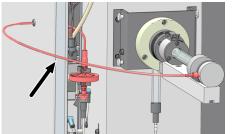
## CAUTION

### Risk of burns from the hot furnace and combustion tube

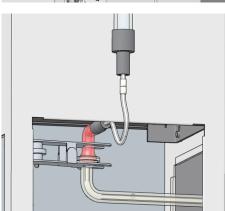
• Switch off the device and allow it to cool down before installation and maintenance.



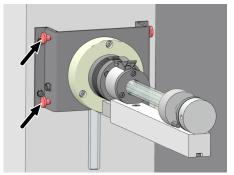
- Exit the software.
- Switch off the analyzer via the main switch and disconnect the power plug from the socket. Cut the gas supply.
- Disconnect the plug-in connector on the left side of the module.



- Disconnect the carrier gas hose from the FAST connector on the water trap on the gas box.
- Unscrew the other hose end from the solids module.

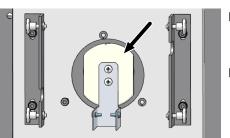


• Remove the fork clamp at the spherical joint between the measuring gas hose and the condensation coil inlet.



Loosen the four knurled head screws on the holding plate and pull the module out of the combustion furnace.
 The measuring gas hose and the holding plate can remain on the module. This makes the next installation easier.
 NOTICE! Do not remove the angled profiles from the furnace. These

**i** NOTICE! Do not remove the angled profiles from the furnace. These profile have been preadjusted and ensure the correct installation position.



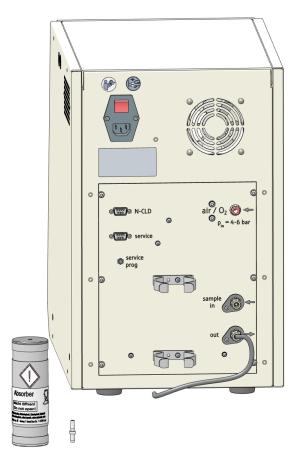
- Remove the sealing plug from the vertical opening of the combustion furnace. Insert the plug into the horizontal opening of the combustion furnace.
- Reinstall the combustion tube for vertical operation.

See also ■ Installing the combustion tube [▶ 89]

## 6.14 Maintaining the chemiluminescence detector (CLD)

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

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



### Fig. 56 Replacing the adsorber cartridge

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

# 7 Troubleshooting



## NOTICE

### Risk of device damage

Contact customer service in the following cases:

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

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

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

The software records log files. Make the log files available to customer service after consultation in the event of a fault.

- Use the Help | Logs | Application log folder and Traffic log folder menu options to open the log file folders.
- Send the current log files to customer service by e-mail. Use Help | Contact service to do this.

## 7.1 Software error messages

Error code: Error message	<ol> <li>Incomplete command from the PC</li> <li>PC command without STX</li> <li>PC command without *</li> <li>PC command CRC error</li> <li>PC command invalid command</li> <li>PC command invalid MESS command</li> <li>Remedy</li> </ol>
<ul> <li>Faulty connection between the internal</li> </ul>	<ul> <li>Initialize the analyzer.</li> </ul>
and external program	
Error code: Error message	7: COM 2 not found
	8: COM 3 not found
	9: COM 4 not found
Cause	Remedy
<ul> <li>Internal hardware problems</li> </ul>	Switch the analyzer off/on.
<ul> <li>The water trap is clogged.</li> </ul>	<ul> <li>Reinitialize the analyzer.</li> <li>Check if the gas pressure error occurs again. If not, replace the water traps.</li> </ul>
<ul> <li>No gas flow at the measuring outlet due to kinked hose for sample gas supply</li> </ul>	<ul> <li>Check the hose. If necessary, eliminate any kinks.</li> </ul>
<ul> <li>The condensation coil is clogged with catalyst balls.</li> </ul>	<ul> <li>Interrupt the measuring gas flow between the combustion tube and condensation coil. Check if the gas pressure error occurs again. If not, rinse the condensation coil with ultrapure water.</li> <li>When replacing the catalyst, always fill in enough quartz glass wool as the first layer.</li> </ul>
<ul> <li>Heavy salt deposits in the combustion tube. (During analysis of highly saline samples, salt deposits can form in the combustion tube.)</li> <li>HT mat used up by analysis of highly saline samples.</li> </ul>	<ul> <li>Replace the HT mat in the combustion tube, or replace the catalyst. Select the measure depending on the number of measurements with the current catalyst filling and the activity of the catalyst.</li> </ul>
<ul> <li>The gas supply to the furnace head is clogged.</li> </ul>	<ul> <li>Clean the gas supply to the furnace head.</li> </ul>
Error code: Error message	12: Incorrect version number
Cause	Remedy
<ul> <li>The version of the control software and the software of the internal computer do not match.</li> </ul>	<ul> <li>Perform a software update.</li> </ul>
Error code: Error message	13: No connection to sampler
Cause	Remedy
<ul> <li>The autosampler is not switched on.</li> <li>The connection cable is not connected or is faulty.</li> </ul>	<ul><li>Switch on the autosampler and initialize the analyzer.</li><li>Check the connection cables.</li></ul>

Error code: Error message	15: Flow-error / no carrier gas			
Cause	Remedy			
<ul> <li>Gas connection not present or faulty.</li> </ul>	Connect the carrier gas. Check the inlet			
Gas connection not present of faulty.	pressure.			
Error code: Error message	20: No connection to optics (NDIR)			
	21: CRC error optics			
	22: Status error optics			
	26: Optics error; incorrect command return			
Cause	Remedy			
Communication error.	<ul> <li>Initialize the analyzer.</li> </ul>			
<ul> <li>NDIR detector faulty.</li> </ul>	<ul> <li>Inform the service.</li> </ul>			
Emerandes Emerandes	24. Ontine entry and a surface surface former			
Error code: Error message	24: Optics error. analog values out of range			
Cause	Remedy			
<ul> <li>The analog values of the detector are outside of the working range.</li> </ul>	<ul><li>Check the quality of the carrier gas.</li><li>Initialize the analyzer and check the</li></ul>			
outside of the working range.	analog values via the component test.			
Error code: Error message	30: No connection to N sensor			
Cause	Remedy			
<ul> <li>The nitrogen detector is not switched on.</li> </ul>	<ul><li>Switch on the detector.</li><li>Check the connection cables.</li></ul>			
<ul> <li>The connection cable is not connected or</li> </ul>				
is faulty.				
<ul> <li>Incorrect connection.</li> </ul>				
Error code: Error message	40: No connection to the syringe pump			
-				
Cause	Remedy			
	<ul> <li>Initialize the analyzer.</li> </ul>			
Cause	<ul><li>Initialize the analyzer.</li><li>Switch the PC off and back on again and</li></ul>			
Cause No communication between the ana-	<ul> <li>Initialize the analyzer.</li> </ul>			
Cause <ul> <li>No communication between the ana-</li> </ul>	<ul><li>Initialize the analyzer.</li><li>Switch the PC off and back on again and</li></ul>			
Cause No communication between the analyzer and the syringe pump.	<ul> <li>Initialize the analyzer.</li> <li>Switch the PC off and back on again and initialize the analyzer.</li> </ul>			
Cause No communication between the analyzer and the syringe pump. Error code: Error message Cause The drive is incorrectly positioned, e.g.	<ul> <li>Initialize the analyzer.</li> <li>Switch the PC off and back on again and initialize the analyzer.</li> <li>111: Rotator error Remedy Initialize the analyzer.</li> </ul>			
Cause          No communication between the analyzer and the syringe pump.         Error code: Error message         Cause         The drive is incorrectly positioned, e.g. jammed.	<ul> <li>Initialize the analyzer.</li> <li>Switch the PC off and back on again and initialize the analyzer.</li> <li>111: Rotator error Remedy Initialize the analyzer. Initialize the analyzer. If the error cannot be corrected, contact</li> </ul>			
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<ul> <li>Cause</li> <li>No communication between the analyzer and the syringe pump.</li> <li>Error code: Error message</li> <li>Cause</li> <li>The drive is incorrectly positioned, e.g. jammed.</li> <li>The drive is faulty.</li> <li>Error code: Error message</li> <li>Cause</li> <li>The drive is incorrectly positioned, e.g. jammed.</li> </ul>	<ul> <li>Initialize the analyzer.</li> <li>Switch the PC off and back on again and initialize the analyzer.</li> <li>111: Rotator error         Remedy         <ul> <li>Initialize the analyzer.</li> <li>If the error cannot be corrected, contact the service.</li> </ul> </li> <li>112: Swivel drive error         <ul> <li>Remedy</li> <li>Initialize the analyzer.</li> <li>If the error cannot be corrected, contact the service.</li> </ul> </li> </ul>			
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CauseRemedy• The sample tray is not positioned correctly.• Position the sample tray again and make sure it clicks into place. • Initialize the analyzer.Error code: Error message115: Wrong rackCauseRemedy• The wrong sample tray is set in the software.• Check the device configuration settings. • If necessary, set a different sample tray.Error code: Error message116: Unknown sampler commandCauseRemedy• Communication error.• Inform the service.Error code: Error message201: Restart the internal programCauseRemedy• Internal program error.• Initialize the analyzer. • For repeated occurrences, monitor precisely at which time the error occurs.CauseRemedy• Communication error.• Initialize the analyzer. • For repeated occurrences, monitor precisely at which time the error occurs.CauseRemedy• Communication error. • Syringe pump faulty.• Initialize the analyzer. • Inform the service.Error code: Error message409: Syringe pump sluggishCauseRemedy• A hose line is clogged. • A hose line is clogged.• Search for the cause and remedy the fault. • Clean or replace the hose line. • Initialize the analyzer.• Syringe pump faulty.• Inform the service.	Error code: Error message	114: Rack detection error			
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<ul> <li>Internal program error.</li> <li>Initialize the analyzer.</li> <li>For repeated occurrences, monitor precisely at which time the error occurs.</li> <li>Cause</li> <li>Communication error.</li> <li>Syringe pump faulty.</li> <li>Inform the service.</li> </ul> Error code: Error message <ul> <li>409: Syringe pump: pump sluggish</li> <li>Cause</li> <li>Remedy</li> </ul> A hose line is clogged. <ul> <li>Search for the cause and remedy the fault.</li> <li>Clean or replace the hose line.</li> <li>Initialize the analyzer.</li> </ul> Syringe pump faulty. <ul> <li>Inform the service.</li> </ul>	Error code: Error message	201: Restart the internal program			
<ul> <li>For repeated occurrences, monitor precisely at which time the error occurs.</li> <li>Cause</li> <li>Communication error.</li> <li>Syringe pump faulty.</li> <li>Initialize the analyzer.</li> <li>Inform the service.</li> </ul> Error code: Error message 409: Syringe pump: pump sluggish Cause <ul> <li>A hose line is clogged.</li> <li>Search for the cause and remedy the fault.</li> <li>Clean or replace the hose line.</li> <li>Initialize the analyzer.</li> </ul> Syringe pump faulty. <ul> <li>Inform the service.</li> </ul>	Cause	Remedy			
<ul> <li>Communication error.</li> <li>Syringe pump faulty.</li> <li>Inform the service.</li> <li>Error code: Error message</li> <li>409: Syringe pump: pump sluggish</li> <li>Cause</li> <li>A hose line is clogged.</li> <li>Search for the cause and remedy the fault.</li> <li>Clean or replace the hose line.</li> <li>Initialize the analyzer.</li> <li>Syringe pump faulty.</li> <li>Inform the service.</li> </ul>	<ul> <li>Internal program error.</li> </ul>	<ul> <li>For repeated occurrences, monitor pre-</li> </ul>			
<ul> <li>Syringe pump faulty.</li> <li>Inform the service.</li> <li>Error code: Error message</li> <li>409: Syringe pump: pump sluggish</li> <li>Cause</li> <li>Remedy</li> <li>A hose line is clogged.</li> <li>Search for the cause and remedy the fault.</li> <li>Clean or replace the hose line.</li> <li>Initialize the analyzer.</li> <li>Syringe pump faulty.</li> <li>Inform the service.</li> </ul>	Cause	Remedy			
Cause       Remedy         A hose line is clogged.       Search for the cause and remedy the fault.         Clean or replace the hose line.       Initialize the analyzer.         Syringe pump faulty.       Inform the service.					
<ul> <li>A hose line is clogged.</li> <li>Search for the cause and remedy the fault.</li> <li>Clean or replace the hose line.</li> <li>Initialize the analyzer.</li> <li>Syringe pump faulty.</li> <li>Inform the service.</li> </ul>	Error code: Error message	409: Syringe pump: pump sluggish			
fault.         Clean or replace the hose line.         Initialize the analyzer.         Syringe pump faulty.         Inform the service.	Cause	Remedy			
	<ul> <li>A hose line is clogged.</li> </ul>	<ul><li>fault.</li><li>Clean or replace the hose line.</li></ul>			
	<ul> <li>Syringe pump faulty.</li> </ul>	<ul> <li>Inform the service.</li> </ul>			
Error code: Error message 410: Syringe pump: valve sluggish	Error code: Error message	410: Syringe pump: valve sluggish			
Cause Remedy	-				
<ul> <li>Syringe pump faulty.</li> <li>Inform the service.</li> </ul>		<ul> <li>Inform the service.</li> </ul>			
Error code: Error message 415: Syringe pump: invalid command	Error code: Error message	415: Syringe pump: invalid command			
Cause Remedy	Cause	Remedy			
Communication error.     Initialize the analyzer.	Communication error.	<ul> <li>Initialize the analyzer.</li> </ul>			
Syringe pump faulty.         Inform the service.	Syringe pump faulty.	<ul> <li>Inform the service.</li> </ul>			

## 7.2 Status errors

Error indication	In 120 ml/min; Out < 110 ml/min	
Cause	Remedy	
<ul> <li>The union nut on the combustion tube is not tightened correctly (after catalyst replacement).</li> <li>The carrier gas supply to the furnace head or lock is not connected properly (after catalyst replacement).</li> <li>The sealing rings on the combustion tube are defective (heavily deformed) or not attached (after catalyst replacement).</li> <li>The FAST connector on the TIC condensate container is leaky.</li> <li>The connections on the water trap systems are loose (after water trap replacement or halogen trap maintenance).</li> </ul>	<ul> <li>Check the screw connections for completeness and deformations. Tighten as necessary.</li> <li>Check the carrier gas supply, in particular the FAST connector on the analyzer wall and the screw connection on the furnace head.</li> <li>Check all connection points on the water traps. Replace the FAST connector as necessary.</li> </ul>	
<ul> <li>The connection between the combustion tube and the condensation coil or the screw connections are leaky.</li> </ul>	<ul> <li>Check the connection of the combustion tube to the condensation coil, in particu- lar the fork clamp position.</li> </ul>	
<ul> <li>The combustion tube is faulty (cracks, fractures at the edge).</li> <li>TIC condensate container faulty (fractures on the connections).</li> </ul>	<ul> <li>Check the glass components. Replace as necessary.</li> </ul>	
<ul> <li>Water traps clogged.</li> </ul>	<ul> <li>Replace the water traps.</li> </ul>	
The condensate pump hose is leaky.	<ul> <li>Check the condensate pump. Replace the hose as necessary.</li> </ul>	
Error indication	In 120 ml/min; Out < 110 ml/min; Out > 130 ml/min	
Cause	Remedy	
<ul> <li>The MFM (mass flow sensor) is faulty.</li> </ul>	<ul> <li>Check the flow with an external mass flow sensor if possible to confirm the er- ror.</li> <li>Inform the service.</li> </ul>	
<ul> <li>The filling of the halogen trap is used up.</li> </ul>	<ul> <li>Check the halogen trap.</li> </ul>	
Error indication	In < 120 ml/min; Out < 110 ml/min	
Cause	Remedy	
<ul><li>No carrier gas.</li><li>The hose line is leaking.</li></ul>	<ul><li>Turn on the carrier gas on the pressure reducer.</li><li>Search for and remedy the leak.</li></ul>	
<ul> <li>The inlet pressure of the carrier gas sup- ply is too low.</li> </ul>	<ul> <li>Set the carrier gas inlet pressure cor- rectly.</li> </ul>	
<ul> <li>The pressure switch in the analyzer was triggered simultaneously with error 10: Gas pressure error.</li> </ul>	<ul> <li>See the remedy for 10: Gas pressure error.</li> </ul>	
<ul> <li>The MFC is faulty.</li> </ul>	<ul> <li>Inform the service.</li> </ul>	
Error indication	In < 120 ml/min;Out:115 125 ml/min	
<ul> <li>No carrier gas.</li> </ul>	• Turn on the carrier gas on the pressure reducer.	

• The inlet pressure of the carrier gas supply is too low.	<ul> <li>Set the carrier gas inlet pressure cor- rectly.</li> </ul>	
The MFM is faulty.	<ul> <li>Inform the service.</li> </ul>	
Error indication	In 120 ml/min; Out > 130 ml/min	
Cause	Remedy	
<ul> <li>Peltier cooling insufficient.</li> </ul>	<ul> <li>Check the cooling from above on the TIC condensate container. The formation of condensation water on the cooling block indicates that the cooling is working.</li> </ul>	
The MFC is faulty.	<ul> <li>Inform the service.</li> </ul>	
Error indication	In; Out = 0 ml/min	
Cause	Remedy	
<ul> <li>A hose line is clogged.</li> </ul>	<ul> <li>Remove and rinse the clogged hose line.</li> <li>Reinstall it again afterward.</li> <li>Replace the clogged hose line.</li> </ul>	
<ul> <li>No method loaded.</li> </ul>	<ul> <li>Load a method.</li> </ul>	
Error indication	NDIR detector values highlighted in color in the <b>Instrument status</b> panel	
<ul> <li>The analog values of the detector are at the edge of the working range.</li> </ul>	<ul> <li>Check the halogen trap. Replace the fill- ing if necessary.</li> <li>Contact the application team and get tips on application regulations for diffi- cult sample matrices.</li> </ul>	

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

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

### 7.3 Device errors

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

Error Cause		Water traps clogged Remedy	
Error		Scattering measurements	
Cai	use	Remedy	
•	The combustion tube filling is used up.	<ul> <li>Replace the catalyst.</li> </ul>	
•	The dosing is faulty.	<ul> <li>Check the dosing.</li> </ul>	
•	The canula is damaged.	<ul> <li>Replace the canula.</li> </ul>	
•	Inhomogeneous samples.	<ul><li>Warm up cold samples before analysis.</li><li>Filter samples prior to analysis.</li></ul>	
-	Sensitive samples can be affected by ambient air.	<ul> <li>Prevent the addition of CO<sub>2</sub> or organic vapors from the ambient air.</li> <li>Check the ambient conditions and remedy the source of the fault.</li> <li>Cover the sample vessels on the autosampler with aluminum foil.</li> <li>Treat the headspace of the sample with gas.</li> </ul>	
•	NDIR-based drift: Unsuitable integration criteria. The software ends measurement too early.	<ul> <li>Check the method settings.</li> <li>If necessary, increase the maximum integration time.</li> </ul>	
Err	or	Autosampler does not draw in sample without air bubbles	
Cai	use	Remedy	
•	The sample intake path is leaky.	<ul> <li>Check the hose connections.</li> <li>If necessary, tighten loose hose connections to the canula or to the valve of the syringe pump.</li> </ul>	
•	The sample intake canula is clogged.	<ul> <li>Remove the canula and clean it in an ul trasonic bath.</li> <li>Replace the canula.</li> </ul>	
•	The dosing syringe is leaky. The PTFE sealing lips of the plunger are damaged.	<ul><li>Remove and check the dosing syringe.</li><li>Replace the dosing syringe.</li></ul>	
Error		Incomplete dosing in the reactors	
Cai	use	Remedy	
•	The dosing path is leaky.	<ul> <li>Check the hose connections. If neces- sary, tighten loose connections: Syringe pump to change-over valve, change- over valve to injection canula, change- over valve to TIC condensate container.</li> </ul>	
Err	or	Carry-over	
Cai	use	Remedy	
	Insufficient syringe flushing.	Flush the dosing syringe with sample before the next injection. To do this, ed the method in the Manage methods window and enter for measurement 1 "3" on the Replicates tab, all additional measurements do not require flushing. Here enter "0".	

Error	Low results (general)		
Cause	Remedy		
<ul> <li>The catalyst is used up.</li> </ul>	<ul> <li>Replace the catalyst.</li> </ul>		
<ul> <li>The system is leaking.</li> </ul>	<ul> <li>Check the system for leaks.</li> </ul>		
<ul> <li>Faulty dosing.</li> </ul>	Check the dosing.		
Error	Low results for TC, TOC, NPOC and TNb analyses (TIC analyses OK)		
Cause	Remedy		
<ul> <li>The catalyst is used up.</li> </ul>	<ul> <li>When using the platinum catalyst and measuring in differential mode (neutral to slightly alkaline samples): The catalyst can be regenerated.</li> <li>Inject ultrapure water that has been acidified six times (pH &lt;2). Recommendation: Measure one or two sample vessels with acidified ultrapure water per analysis series.</li> <li>Replace the catalyst.</li> <li>After replacing the catalyst, perform calibration.</li> </ul>		
Error	Low results for TIC analyses (TC, TOC, NPOC analyses OK)		
Cause	Remedy		
<ul> <li>No phosphoric acid in the reagent bot- tle.</li> </ul>	<ul> <li>Refill the bottle.</li> </ul>		
<ul> <li>Incorrect sample dosing.</li> </ul>	Check the dosing.		
Error	Low results for TNb analyses		
Cause	Remedy		
<ul> <li>The catalyst is used up.</li> </ul>	<ul> <li>Replace the catalyst.</li> </ul>		
<ul> <li>The sample concentration is above the calibrated range.</li> </ul>	<ul> <li>Observe the calibrated range.</li> <li>Use quadratic regression.</li> <li>If possible, calibrate dependent on the matrix.</li> <li>For analysis of unknown substances, use low concentrations if possible. If possible, dilute the sample.</li> <li>Use synthetic air as a carrier gas.</li> </ul>		
Error	Unusual peak shape during TC and TNb analyses		
Cause	Remedy		
<ul> <li>The catalyst is used up.</li> </ul>	<ul> <li>Note: Low results also occur at the same time. Regenerate or replace the catalyst.</li> </ul>		
<ul> <li>Unsuitable integration criteria.</li> </ul>	<ul> <li>Check the integration criteria in the method.</li> </ul>		
<ul> <li>Measuring range for CLD exceeded.</li> </ul>	Dilute the sample.		
<ul> <li>Faulty dosing.</li> </ul>	<ul> <li>For manual sample supply: Ensure even injection.</li> </ul>		
Error	Incorrect TNb analyses with CLD (TC analy- ses OK)		
Cause	Remedy		
<ul><li>Faulty hose connection between the an- alyzer and the detector.</li><li>Ozone generator faulty.</li></ul>	<ul><li>Check the hose connections.</li><li>Inform the service.</li></ul>		

Error	Condensate pump or phosphoric acid pump leaky
Cause	Remedy
<ul><li>Leaking hose connections.</li><li>Defective pump hose.</li></ul>	<ul><li>Check the connections.</li><li>Replace the hose.</li></ul>
Error	5 V, 24 V indicator lamps on LED strip not lighting
Cause	Remedy
<ul> <li>Power supply or electronics fault.</li> </ul>	<ul><li>Check the electrical connections.</li><li>Check the laboratory power supply.</li></ul>
<ul> <li>Device fuse faulty.</li> </ul>	<ul> <li>Inform the service.</li> </ul>
Error	Status LED on the analyzer not lighting
Cause	Remedy
<ul> <li>The internal program has not been started.</li> </ul>	<ul> <li>Switch the analyzer off and on again.</li> </ul>
Error	Heating monitoring lamp on LED strip not lighting
Cause	Remedy
<ul> <li>Device in standby with standby tempera- ture = room temperature</li> </ul>	<ul> <li>Initializing the device</li> </ul>
<ul> <li>Faulty thermocouple (furnace). The "Broken Thermocouple" lamp is lit on the LED strip.</li> </ul>	<ul> <li>Inform the service.</li> </ul>
Faulty electronics component.	<ul> <li>Inform the service.</li> </ul>
<ul> <li>The combustion furnace is not con- nected correctly.</li> </ul>	<ul> <li>Check the connection of the combustion furnace.</li> </ul>
needed contectiy.	

# 8 Transport and storage

## 8.1 Transport

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

Avoid the following during transport:

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

## 8.1.1 Preparing the analyzer for transport



## CAUTION

### Risk of burns from the furnace, furnace head and combustion tube

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

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



# CAUTION

### **Risk of injury**

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

- Handle glass parts with extreme caution.
- Wear non-slip glass handling gloves that allow a firm and secure grip.



## NOTICE

### Risk of device damage due to unsuitable packaging material

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

Prepare the analyzer for transport as follows:

- Shut down the analyzer via the software.
- Switch off the analyzer via the main switch. Allow the device to cool down.
- Cut the gas supply. Disconnect the power plug from the power socket.
- Disconnect all cables and gas hoses on the rear of the analyzer.
- Open the doors of the analyzer.

• Remove the reagent bottle, the drip tray and other loose accessories. Wipe off the hose(s) with a clean paper towel.

▲ CAUTION! The hoses contain acid residue.

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

#### See also

■ Maintenance and care [▶ 73]

### 8.1.2 Preparing the AS vario autosampler for transport

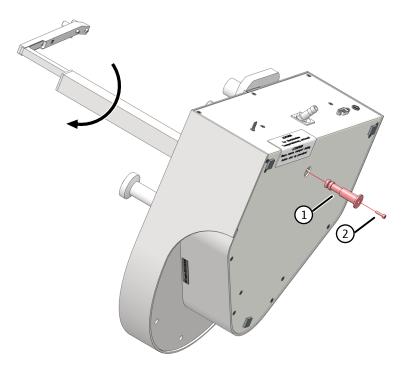


## NOTICE

#### Risk of device damage when transporting without transport locks

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

• Always apply a transport lock before transport.



#### Fig. 57 Securing the autosampler for transport

1 Transport lock

2 M3x12 screw

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

### 8.1.3 Moving the device in the laboratory



## CAUTION

### Risk of injury during transport

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

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

Observe the following when moving the device within the laboratory:

- Insufficiently secured components pose a risk of injury! Before moving the device, remove all loose parts and disconnect all connections from the device.
- For safety reasons, two persons are required to transport the device, one person on each side of the device.
- As the device does not have carrying handles, grip the device firmly with both hands at the lower end. Lift the device simultaneously.

- Observe the guide values and adhere to the legally mandated limits for lifting and carrying loads without auxiliary means.
- Observe the installation conditions at the new location.

## 8.2 Storage



## NOTICE

### Risk of device damage due to environmental conditions

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

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

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

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

# 9 Disposal

Waste water containing acids and samples occurs during device operation. Dispose of the neutralized waste in accordance with the legal requirements.
The halogen trap contains copper and brass. Contact the responsible institution (author- ity or waste disposal company). There you will receive the information regarding recy- cling or disposal.
The special catalysts contain $Pt(Al_2O_3)$ or $CeO_2$ .
Dispose of the used catalyst properly in accordance with the legal disposal requirements.
Analytik Jena will accept the special catalyst back for disposal. Please contact the cus- tomer service department. For the customer service address, see the inside front cover.
At the end of its service life, the device and its electronic components must be disposed of as electronic waste in accordance with the applicable regulations.

# CAUTION

### Skin and respiratory system irritation due to dust

The insulation of the furnace contains alkaline earth silicate wool (AES wool). Dust may form when working with AES wool.

- Avoid the formation of dust.
- Wear personal protective equipment: Respiratory mask, safety goggles, gloves and coat.
- Dispose of properly.

# 10 Specifications

# 10.1 Technical data of the basic device

General characteristics	Designation/type		multi N/C 3300 HS		
	Item number	Item number		11-0118-201-62	
	Basic device dimer	Basic device dimensions (W x D x H)		513 x 547 x 464 mm	
	Basic device mass		21 kg		
	Sound pressure lev	vel	<70 dB(	(A)	
Methods data	Digestion principle	2	Thermo	catalytic oxidation	
	Digestion tempera	Digestion temperature		50 °C, depending on catalyst	
	Sample feed			Flow injection	
	Sample volume		50 to 30	μι 000	
	Particle handling c	capacity	In accord	dance with DIN EN 1484	
	Carbon detection p	principle	NDIR (co	oupled with the VITA method)	
	TC, TOC, NPOC, TI	C measurement range	0 to 300	000 mg/l	
	TC, TOC in solid m (with the HT 1300	easurement range	0 to 500	) mg	
Nitrogen detection	Nitrogen detection	n principle (optional)	CLD		
5	TN <sub>b</sub> measurement		0 to 20000 mg/l		
Process control		Control and analysis software		multiWin pro	
	Software function	scope	analysis	ne graphics, status indication during , graphical display of the measured result print-out	
				An optional FDA software upgrade that pro- vides data integrity and ensures compliance with pharmaceutical guidelines 21 CFR Part 11 and EudraLex Volume 4 Annex 11	
Gas supply	Option 1	Oxygen		≥4.5	
	Option 2	Synthetic air (from a compressed gas cy	/linder)	Hydrocarbon and $CO_2$ -free	
	Option 3	Purified compressed air (provided by a TOC gas ge	nerator)	$CO_2 < 1 \text{ ppm}$ Hydrocarbons (as $CH_4$ ) <0.5 ppm	
	Inlet pressure	400 to 600 kPa			
	Flow rate	15 l/h, depending on mea	suring mod	de	
	Analyte gas flow	115 to 125 ml/min	-		
	NPOC purge flow	50 to 160 ml/min			

Electrical variables	Voltage	115/230 V
	Frequency	50/60 Hz
	Fuses	2 T6.3 A H
	Typical average power consumption	400 VA
	Maximum power consumption	500 VA
	PC interface	USB 2.0
	Module/accessory interface	RS 232
	Only use original fuses from Analytik Jena!	
Ambient conditions	Operating temperature	+10 to 35 °C (air-conditioning recom- mended)
	Maximum humidity	90 % at 30 °C
	Air pressure	0.7 to 1.06 bar
	Storage temperature	5 to 55 ℃
	Humidity during storage	10 to 30 % (use desiccant)
	Operating altitude (max.)	2000 m
Control computer minimum re- quirements	Processor	Min. 3.2 GHz
	Disk drive	Min. 64 GB
	RAM	Min. 8 GB
	Screen resolution	Min. 1920 x 1080 px
	Graphic card	compatible with DirectX 12 or higher, with WDDM 2.0 driver
	USB port	Min. 1 USB 2.0 interface, to connect the basic device
	CD/DVD drive	For software installation
	Operating system	Windows 10/11, 32 or 64 bit
		÷

## 10.2 Technical data of the accessories

AS 21hp, AS 10e autosamplers	Order number (designation)	11-0513-001-26 ( AS 21hp)
		11-0516-003-26 ( AS 10e)
	Dimensions (W x D x H), without holder	260 x 320 x 390 mm
	Mass	4.5 kg
	Operating voltage	24 V DC, 2.5 A via external power supply
	Power supply of power supply unit	110 to 240 V +10/-5 %, 50/60 Hz:
	Power consumption	60 VA
AS 21hp autosampler	Sample positions	21
	Tube size	50 ml
	Purging NPOC samples	Parallel and sequential
	Magnetic stirrer (integrated)	Homogenization of samples containing par- ticles

AS 10e autosampler	Sample positions		10		
F -	Tube size		50 ml		
	Purging NPOC samples		Sequential or	าโง	
				5	
AS vario autosampler	Order number (designation)		11-0514-00	3-26 ( AS vario)	
·	Dimensions (W x D x H)		350 x 400 x		
	Mass		15 kg		
	Operating voltage		24 V DC via external power supply		
	Power supply, external powe	r supply unit	100 to 240 V, 50/60 Hz (autosensing)		
	Power consumption		50 VA		
	Sample tray with sample	Tube size		AS vario	AS vario ER
	positions 20	100 ml		Yes	No
	47 (dilut)	12 ml + 50 m		Yes	Yes
	52	100 ml		Yes	No
	72	40 ml + 50 m	l (ontional)	Yes	Yes
	100	20 ml		Yes	Yes
	146	12 ml		Yes	Yes
				-	
EPA Sampler	Order number (designation)		11-126.693 ( EPA Sampler)		
	Dimensions (W x D x H)		500 x 540 x 550 mm		
	Mass		15 kg		
	Operating voltage		24 V DC via external power supply		
	Power supply, external power supply unit		100 to 240 V, 50/60 Hz (autosensing)		
	Power consumption		30 VA		
	Sample positions		64		
	Sample vessels		40 ml		
Chemiluminescence detector	Order number (designation)		11-0401-002-62 (CLD-300)		
(CLD)	Detection principle		Chemiluminescence detector		
	Parameter		$TN_{b}$ (total bound nitrogen)		
	Measurement range		0 to 20000 mg/l TN <sub>b</sub>		
	Detection limit		0,005 mg/l TN $_{\rm b}$		
	Analysis time		3 to 5 min		
	Gas for ozone generation		Gas supply as for basic device		
			60 ml/min, 400 to 600 kPa:		
	Dimensions (W x D x H)		296 x 581 x 462 mm		
	Mass		12.5 kg		
	Operating voltage		110 to 240 V, 50/60 Hz:		
	Fuses		2 T4.0 A H		
	Typical average power consumption		200 VA		
	Analyzer interface		RS 232		

The ambient conditions for operation and storage of the accessories correspond to the ambient conditions of the basic device.

The technical data for other accessories can be found in their separate operating instructions.

## 10.3 Standards and directives

Protection class and protection type	The device is protection class I and protection type IP 20.
Device safety	<ul> <li>The device complies with the following safety standards</li> <li>EN 61010-1</li> <li>EN 61010-2-081</li> <li>EN 61010-2-010</li> <li>EN 61010-2-051 (for operation with autosampler)</li> </ul>
EMC compatibility	<ul> <li>The device has been checked for transient emissions and noise immunity.</li> <li>In terms of transient emissions, the device complies with Group 1 / Class A according to EN IEC 61326-1 Section 7 and is not suitable for use in residential areas.</li> <li>The device meets the requirements for interference immunity according to EN IEC 61326-1 Section 6 Classification I (requirements for use in industrial electromagnetic environments).</li> </ul>
Environmental and ambient in- fluences	<ul> <li>This device has been tested in environmental simulations under operation and transport conditions and is in accordance with the requirements in:</li> <li>ISO 9022-2</li> <li>ISO 9022-3</li> </ul>
EU directives	The device meets the requirements of the directive 2011/65/EU. The device is designed and tested in accordance with standards meeting the require- ments of EU directives 2014/35/EU and 2014/30/EU. The device leaves the factory in a sound condition with regard to technical safety. To maintain this condition and to en- sure safe operation, the user must strictly observe the safety and operating instructions contained in this operating manual. For accessories delivered with the device and sys- tem components from other manufacturers, the information provided in their respective operating manuals has priority.
Guidelines for China	The device contains substances subject to regulation (according to the directive GB/T 26572-2011). Analytik Jena guarantees that, if the device is used as intended, these substances will not leak within the next 25 years and therefore will not pose a threat to the environment or health within this time period.

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