

Operating Manual

multi N/C 3300 (duo) TOC/TNb Analyzers



Manufacturer

Analytik Jena GmbH+Co. KG Konrad-Zuse-Straße 1 07745 Jena / Germany Phone: +49 3641 77 70 Fax: +49 3641 77 9279 Email: info@analytik-jena.com

Technical Service

Analytik Jena GmbH+Co. KG Konrad-Zuse-Straße 1 07745 Jena / Germany Phone: +49 3641 77 7407 Fax: +49 3641 77 9279 Email: service@analytik-jena.com



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

General Information	http://www.analytik-jena.com
Documentation Number	11-0118-004-23
Edition	D (10/2024)
Technical Documentation	Analytik Jena GmbH+Co. KG
	© Copyright 2024, Analytik Jena GmbH+Co. KG

Table of contents

1 Basic information		formation	7
	1.1	About this user manual	7
	1.2	Analyzer area of application	8
	1.3	Intended use	8
2	Safety.		
-	2.1	Safety labeling on the device	
	2.2	Requirements for the operating personnel	
	2.3	Safety instructions, transport and commissioning	
	2.4 2.4.1 2.4.2 2.4.3 2.4.4 2.4.5 2.4.6 2.5	Safety instructions: during operation Summary of safety instructions	11 11 11 12 12 12 13
3	Functio	n and design	15
	3.1 3.1.1 3.1.2 3.1.3 3.1.4 3.1.5 3.1.6 3.1.7	LayoutSample supply system Hose system Combustion system Measuring gas drying and cleaning Detection Indicator and control elements, connections Accessories	16 17 20 21 24 25
	3.2	Additional options for the analyzer	
	3.3	Function and measuring principle	
	3.4 3.4.1 3.4.2 3.4.3 3.4.4 3.4.5 3.4.6 3.4.7 3.4.8	Measuring methods TC analysis TOC analysis TIC analysis NPOC analysis DOC analysis POC analysis TN _b analysis Additional sum parameters	29 29 29 29 30 30 31 31
	3.5	Catalysts	
	3.6 3.6.1 3.6.2 3.6.3 3.6.4 3.6.5	Calibration Calibration strategies Daily factor Calibration method Method characteristics Other calculations	32 32 33 34
	3.7 3.7.1 3.7.2	Blank values Water blank values Eluate blank value	35 35

	3.7.3	Boat blank value	. 36
	3.8	System suitability test	. 36
4 Installa		tion and commissioning	
	4.1	Installation conditions	. 38
	4.1.1	Ambient conditions	. 38
	4.1.2	Device layout and space requirements	
	4.1.3	Power supply	
	4.1.4	Gas supply	
	4.2	Unpacking and setting up the device	
	4.2.1	Installing and commissioning the analyzer	
	4.3	Connecting accessories	
	4.3.1	The AS 10e and AS 21hp autosamplers	
	4.3.2 4.3.3	AS vario autosampler EPA Sampler	
	4.3.4	POC module	
	4.3.5	Chemiluminescence detector (CLD)	
	4.3.6	External solids module	. 65
	4.4	Using the salt kit	. 66
5	Operati	on	. 71
	5.1	General notes	
	5.2	Switching on the analyzer	. 72
	5.3	Switching off the analyzer	. 73
	5.4	Performing measurements	
	5.4.1	Create sequence and measure with manual sample feed	
	5.4.2	Creating a sequence and measuring with automatic sample feed	. 76
6	Mainte	nance and care	. 78
	6.1	Maintenance overview	. 78
	6.2	Adjustment and setting	
	6.2.1	General notes for adjusting the autosampler	
	6.2.2 6.2.3	Adjusting the AS vario autosampler	
	6.2.5 6.2.4	Adjusting the EPA Sampler Setting the NPOC purge flow	
	6.3	Syringe pump maintenance	
	6.4	Replacing the pump hose	
	6.5	Replacing the hose connections	
	6.6	Checking the system for leaks	
	6.7	Replacing the catalyst	
	6.7.1	Removing the combustion tube	
	6.7.2	Filling the combustion tube	
	6.7.3	Installing the combustion tube	. 94
	6.8	Optional salt kit maintenance	. 96
	6.9	Removing and installing the combustion furnace	
	6.9.1	Removing the combustion furnace	
	6.9.2	Installing the combustion furnace	
	6.10	Cleaning the TIC condensate vessel	
	6.11	Condensation coil maintenance	100

	6.12	Replacing the water traps	102
	6.13	Replacing the halogen trap	104
	6.14	POC module maintenance	105
	6.15	Maintaining the chemiluminescence detector (CLD)	106
7	Trouble	shooting	108
	7.1	Software error messages	108
	7.2	Status errors	112
	7.3	Device errors	114
8	Transpo	ort and storage	118
	8.1	Transport	118
	8.1.1	Preparing the analyzer for transport	
	8.1.2	Preparing the AS vario autosampler for transport	
	8.1.3	Moving the device in the laboratory	120
	8.2	Storage	121
9	Disposa	1	122
10	Specific	ations	123
	10.1	Technical data of the basic device	123
	10.2	Technical data of the accessories	124
	10.3	Standards and directives	126

1 Basic information

1.1 About this user manual

Content

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

- multi N/C 3300
- multi N/C 3300 duo

In this manual, these models are collectively referred to as the multi N/C 3300. Any differences between the models are explained in the relevant section.

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

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

The multi N/C 3300 duo modular measuring system enables automated analysis of liquid and solid samples. The layout, installation and operation of the modular measuring system are described in the user manual for the HT 1300 solids module. Pay particular attention to the information on switching between liquid and solid operation given there.

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.

The user manual uses the following symbols and signal words to indicate hazards or in-

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

Program terms are in bold (e.g., the **System** menu).

structions. These warnings are always placed before an action.

Menu items are separated by vertical lines (e.g., System | Device).

Symbols and signal words used in this manual



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

Use in water treatment

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

Use in environmental monitoring

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

Use in power plants and laboratories

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

Analysis of waste and soil samples

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

Use in research and education

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

Use in pharmacy, medicine, biotechnology

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.

Analyzers with upgraded software are suitable, for example, for use in cleaning validation, as well as for the analysis of water for injection purposes. Extractable organic compounds in pharmaceutical plastic packaging can also be summarily analyzed.

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	 On the furnace, on the furnace cover: On the left side wall: Risk of burns from the hot furnace
	Warning against corro- sive substances	 On the front side, next to the phosphoric acid bottle: Warning against phosphoric acid
	Warning against harm- ful or irritating sub- stances	 On the front side: Warning against phosphoric acid
	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 (O₃). When used in accordance with the intended use, the downstream ozone decomposer destroys the toxic gas. Various safety measures result in the automatic shut-down of the ozone generator. However, the following still applies: if you notice a strong smell of ozone, switch the device off immediately and inform customer service. In order to guarantee perfect and safe operation, Analytik Jena recommends an annual inspection and maintenance of the detector by customer service.

2.4.2 Safety instructions – protection against explosion and fire

The device may not be operated in an explosive environment.

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

2.4.3 Electrical system safety instructions

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

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

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

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

2.4.5 Handling of auxiliary and operating materials

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

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

 Special care must be taken when handling concentrated acids. The regulations and notes in the safety data sheets for the handling of orthophosphoric acid (H₃PO₄) 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 [▶ 78]

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

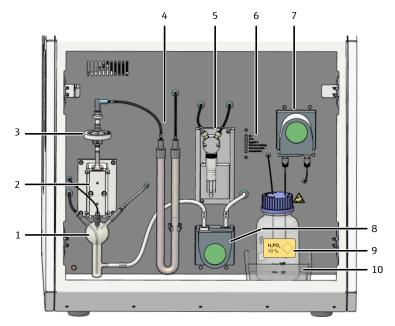
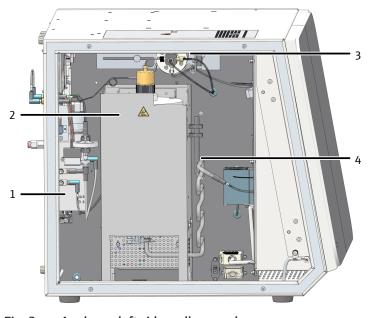
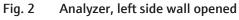


Fig. 1 Analyzer, front doors opened

- 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





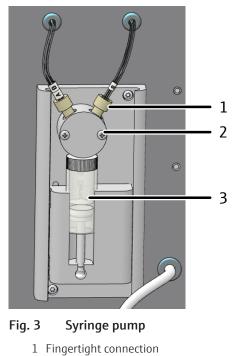
Gas box
 5-way valve

- 2 Combustion system
- 4 Condensation coil

3.1.1 Sample supply system

Sample supply is carried out as flow injection via syringe pump with 2-port valve. The injection volume is 50 to 1000 $\mu l.$

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



- 2 2-port valve
- The hoses on the 2-port valve are connected to the following components:
- Hose 8 to the ultrapure water bottle

3 Dosing syringe

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.

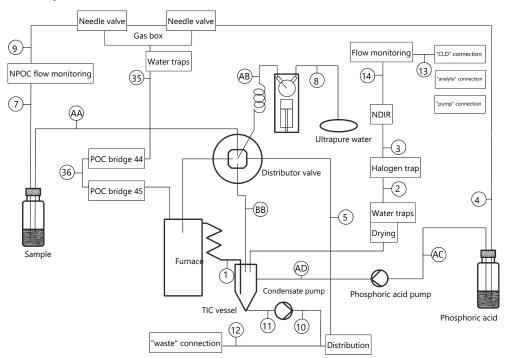


Fig. 4 Hose diagram

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.

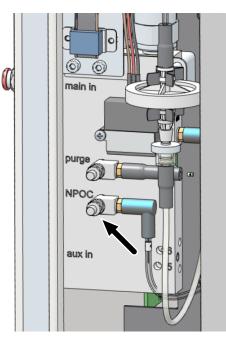


Fig. 5 Setting the NPOC purge flow

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.

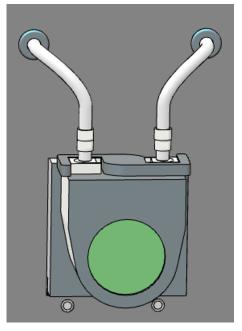


Fig. 6 Condensate pump

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.

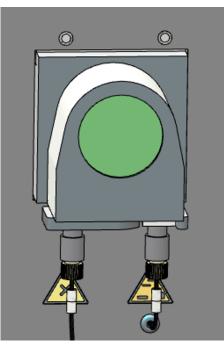


Fig. 7 Phosphoric acid pump

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.

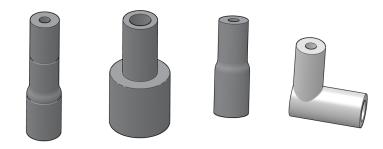
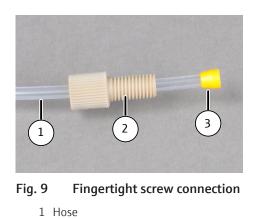


Fig. 8 FAST connector

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.



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 $^\circ\!\text{C}.$

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

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

For solutions with a high salt content, Analytik Jena recommends the optional salt kit, which significantly reduces wear. The kit contains a special furnace head and a combustion tube into which a salt vessel can be inserted.

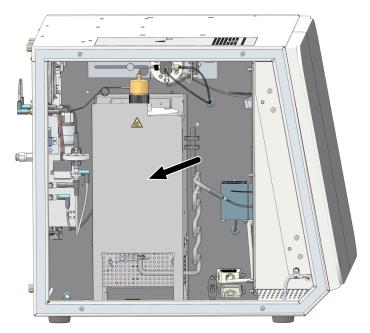


Fig. 10 Combustion furnace

3.1.4 Measuring gas drying and cleaning

Condensation coil

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

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

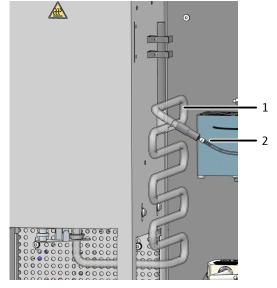


Fig. 11 Condensation coil

1 Condensation coil

2 Hose 1 to the TIC condensate container

```
TIC condensation module
```

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

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

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

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

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

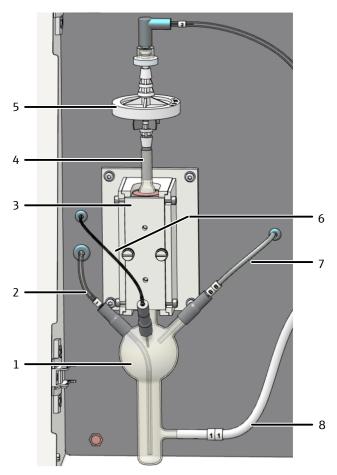


Fig. 12 TIC condensation module

- 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.

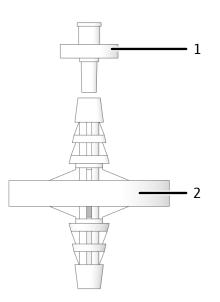


Fig. 13 Water traps

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.

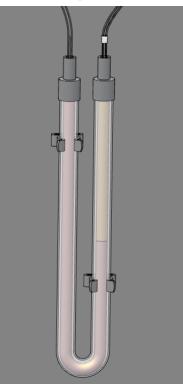


Fig. 14 Halogen trap

3.1.5 Detection

NDIR detector	The NDIR detector (non-dispersive infrared absorption detector) is behind the right side wall of the analyzer.
	Gases with molecules from different atoms have specific absorption bands in the in- frared wavelength range. When a light beam is sent through an arrangement of cells which contains IR-active gases, these gas components absorb a proportional share of the total radiation on their characteristic wavelengths according to their concentration in the gas mixture.
	The radiation receiver used in the NDIR detector is selective for CO_2 .
Measurements using the VITA method	The CO_2 molecules are detected metrologically as long as they remain in the cell of the NDIR detector. The measuring gas flow can fluctuate during CO_2 measurement, because, for example, liquid samples evaporate or condense during dosing. For this reason, the CO_2 molecules are sometimes detected spectrometrically for a longer time (at lower gas flows) or a shorter time (at higher gas flows).
	The VITA method is formally the residence-time-coupled integration for TOC analyses. The measuring gas flow is determined in parallel with the NDIR signal in the VITA method. The NDIR signal is normalized via computer control. This compensates for oc- curring flow fluctuations, ensuring constant gas flow. Integration is only carried out after this.
	A highly precise digital flowmeter detects the gas flow in the immediate area of the NDIR detector.
Electrochemical NO detector (ChD, optional)	For TN_b detection, the electrochemical NO detector can be used. The NO detector is behind the right side wall of the analyzer. It analyzes the nitrogen oxide (NO) content in the measuring gas.
	After thermal oxidation of the sample, the measuring gas enters the detector. In the de- tector, the nitrogen oxides diffuse via a highly selective membrane in the electrochemi- cal measuring cell.
	The nitrogen oxides are oxidized at the anode. This alters the current flow between the electrodes in proportion to the concentration of nitrogen oxide. The change of the current flow is evaluated as a signal and the nitrogen content of the analyzed sample is determined from this. The electrolyte in the measuring cell only serves as catalyst and is not used up.
	A supply voltage is required for the operation of the electrochemical NO detector (ChD). Even if the analyzer is switched off, a support voltage must maintain the electrochemical equilibrium in the ChD. A battery (U9VL) is installed in the right side part of the analyzer for this.
Chemiluminescence detector CLD (optional)	The optional addition of a chemiluminescence detector (CLD) to the analyzer enables $TN_{\rm b}$ determination. The CLD-300 must be positioned next to the analyzer as an external device.
	The measuring gas formed by the thermal oxidation of the sample is dried and then en- ters the reaction chamber of the chemiluminescence detector. There, the nitrogen monoxide present in the measuring gas is oxidized with ozone into activated nitrogen dioxide. Emission of light photons (luminescence) returns the molecules of the nitrogen dioxide to their original state. The luminescence is recorded. The signal is proportional to the nitrogen monoxide concentration. The total nitrogen content of the sample can be determined in this manner.

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

3.1.6 Indicator and control elements, connections

LED display

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

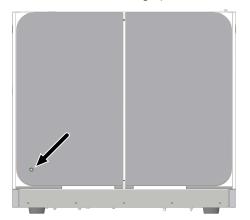


Fig. 15 Status LED

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

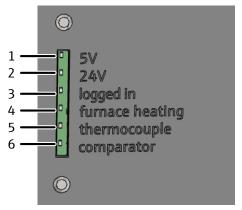


Fig. 16 LED strip (right front door open)

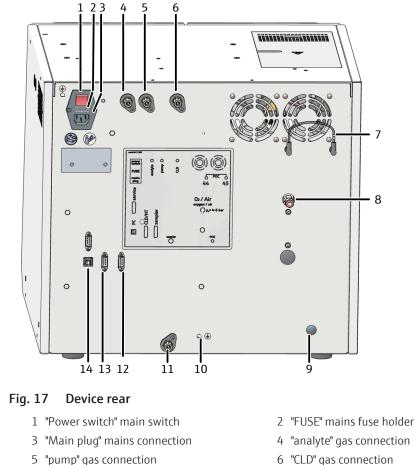
- 1 Voltage of the internal firmware controller
- 3 Internal computer start
- 5 Thermocouple (is lit if a thermocouple is broken)
- 2 Device voltage
- 4 Furnace heating on/off
- 6 Furnace comparator (is lit at excessive temperatures)

Main switch and connections

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

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

A diagram in the center details the different connections.



- 7 Bridge for the gas connection of the POC module
- 9 POC module connection (optional)
- 11 "waste" connection
- 13 RS 232 interface for "CLD/HT" CLD and solids modules

- 6 "CLD" gas connection
- 8 "O₂/Air" carrier gas connection
- 10 Connection of the neutral conductor on the autosampler
- 12 RS 232 interface for "sampler" autosampler
- 14 USB 2.0 "PC" interface

Type plate

- The type plate is attached to the device rear.
- The type plate contains the following information:
- manufacturer address, trademark
- Designation of the device, serial number
- Electrical connection data
- Conformity markings
- WEEE marking

3.1.7 Accessories

The following accessories are required for measurements with the analyzer:

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

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

3.2 Additional options for the analyzer

Autosampler	 The following autosamplers are available for the analyzer: AS vario with various tray sizes AS vario ER with various tray sizes and canula flushing AS 10e for 10 samples AS 21hp for 21 samples EPA Sampler with piercing function
POC module	The addition of a POC module to the analyzer enables direct detection of POC in aque- ous samples.
External solids module	The addition of the external HT 1300 solids module to the analyzer enables the cata- lyst-free digestion of solid samples at temperatures of up to 1300 $^{\circ}$ C in the ceramic com- bustion furnace. The ceramic boats allow input of large sample sizes (up to 3000 mg). This can compensate for sample inhomogeneities.
Manual TIC solids module	The TIC determination in solid samples can be performed by equipping the analyzer with a TIC solids module. Large sample amounts can be weighed in an Erlenmeyer flask. The sample is acidified and magnetically stirred on a heating plate to digest carbonates and hydrogen carbonates to CO_2 .

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.

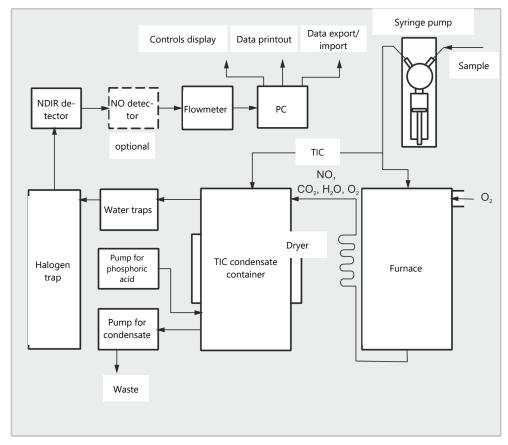


Fig. 18 Principle of operation

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.

 $\mathsf{R} + \mathsf{O}_2 \rightarrow \mathsf{CO}_2 + \mathsf{H}_2\mathsf{O} (1)$

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

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

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 μ m.

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

3.4.6 POC analysis

POC: Purgeable Organic Carbon

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

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

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

3.4.7 TN_b analysis

TN_b: Total Nitrogen bound

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

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

In the control and analysis software, you can activate the calculation of additional sum

3.4.8 Additional sum parameters

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 BOD₅: 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 BOD₅, default setting: A = 3.000, B = 0.000. CO2 For TIC methods and liquid measurements, you can activate the calculation of the carbon dioxide concentration based on the TIC. Formula: $c(CO_2) = 2.833 \times c(TIC)$ ΤP **TP:** Total Protein For TN methods you can activate the calculation of the total protein content based on the TN. Formula: $c(Total Protein) = A \times c(TN)$ You can set the factor for calculating the total protein content between 0 and 10, default 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 $^\circ$ 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 CeO₂ 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: $c = (k_2 \times I_{Net}^2 + k_1 \times I_{Net} + k_0)/V$
	c: target concentration of the standards
	V: Sample volume
	I _{Net} : Net integral
	k_0, k_1, k_2 : calibration coefficient
	The net integral is the raw integral corrected by the blank value of the preparation wa- ter.
	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.
TC/NPOC	The TC channel is calibrated directly for the TC parameter, and after sample purging for the NPOC parameter.
	The concentration c_{TC} is proportional to integral I_{TC} : $c_{TC} = f(I_{TC})$.
TIC	The TIC channel is calibrated.
	The following applies: $c_{TIC} = f(I_{TIC})$
ТОС	The TOC is determined with the differential method (TOC Diff). Generally, separate cali- bration 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 so- lutions. This is useful if different ranges are to be calibrated for the TC and TIC channels.
NPOC plus	The calibration of the NPOC plus method is the same as the calibration of the TOC (Diff) method. Before analysis, the TIC must be sufficiently purged for the use of the differen- tial 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)
	 Purging of the actumed sample (5 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 mea- suring 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 sepa- rately 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 (μ 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 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 _{DIBV} : Diluent blank value
	V _{Sample} Sample volume
	I _{eff} : Effective integral
	N _P : Number of units of the primary sample
	N_{D} : Number of units of the diluent
	I _{Raw} : Raw integral
	I _{DiBV} : Diluent integral
Diluent indication	Proportions of the primary probe: in total proportions (e.g., 10 parts in 100 parts)
	This means that 10 ml of the primary sample is filled to a total volume of 100 ml with dilution water.
	A 1:1 dilution ratio equals $I_{DiBV} = 0$.
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.

 $|_{eff} = |_{Raw} - |_{Eluate blank value}$

 I_{eff} : Effective integral

I_{Raw}: Raw integral

I_{Eluate blank value}: Eluate blank value

3.7.3 Boat blank value

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

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

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

3.8 System suitability test

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

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

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

Procedure:

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

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

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

E: System efficiency in %

r_s: TOC of the reference solution (sucrose)

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

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

4 Installation and commissioning

4.1 Installation conditions

4.1.1 Ambient conditions

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

The following climate requirements apply in the room of operation:

Operating temperature	+10 to 35 °C (air-conditioning recom- mended)
Maximum humidity	90 % at 30 ℃
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.
- The AS vario, AS vario ER and EPA Sampler autosamplers must be positioned to the left of the basic device.
- The CLD-300 nitrogen detector is set up to the right of the basic device.
- The HT 1300 solids module is placed to the right of the basic device. The solids module can be set up with the front side or with its left side facing forward.
- The manual TIC solids module is placed to the right of the basic device.
- The FPG 48 solids autosampler is placed in front of the HT 1300 solids module.
- The ChD (approx. 0.5 kg) nitrogen detector is installed in the basic device.

Component	Dimensions (Width x Depth x Height)	Weight
Basic device	513 x 547 x 464 mm	21 kg
multi N/C 3300 duo modular measuring sys- tem (basic device + AS 60 autosampler + HT 1300 solids module + FPG 48 autosampler)	2215 x 650 x 464 mm (mini- mum)	85 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
AS vario ER autosampler (with canula flush)	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
HT 1300 solids module	510 x 550 x 470 mm	22 kg
FPG 48 autosampler	500 x 550 x 460 mm	20 kg
Manual TIC solids module	300 x 550 x 470 mm	10 kg

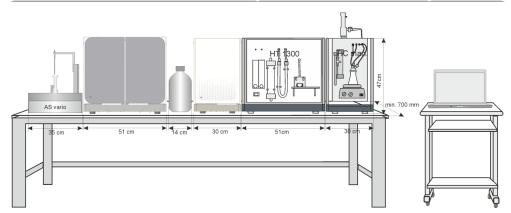


Fig. 19 Space required for multi N/C 3300 with modules

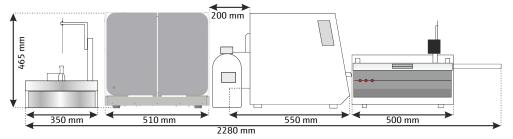


Fig. 20 Space required for multi N/C 3300 duo modular measuring system

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

B Maintenance and care [▶ 78]

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.

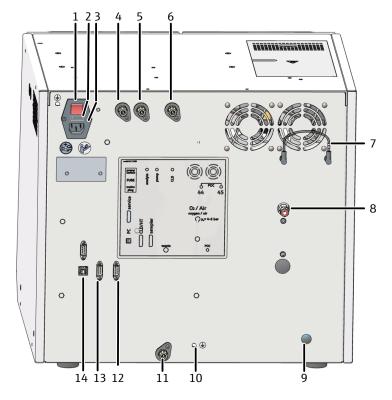


Fig. 21 Device rear

- 1 "Power switch" main switch
- 3 "Main plug" mains connection
- 5 "pump" gas connection
- 7 Bridge for the gas connection of the POC module
- 9 POC module connection (optional)
- 11 "waste" connection
- 13 RS 232 interface for "CLD/HT" CLD and solids modules

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.

- 2 "FUSE" mains fuse holder
- 4 "analyte" gas connection
- 6 "CLD" gas connection
- 8 "O₂/Air" carrier gas connection
- 10 Connection of the neutral conductor on the autosampler
- 12 RS 232 interface for "sampler" autosampler
- 14 USB 2.0 "PC" interface



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.

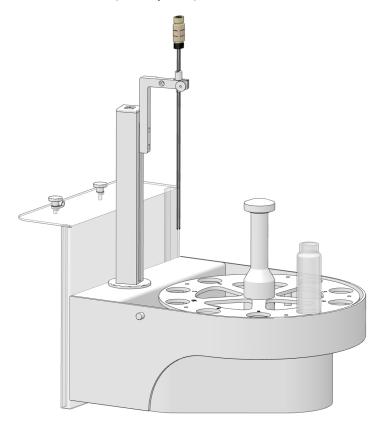


Fig. 22 AS 10e autosampler

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 CO₂ 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.

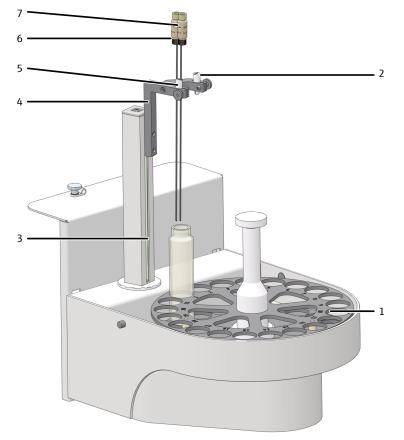


Fig. 23 AS 21hp autosampler

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

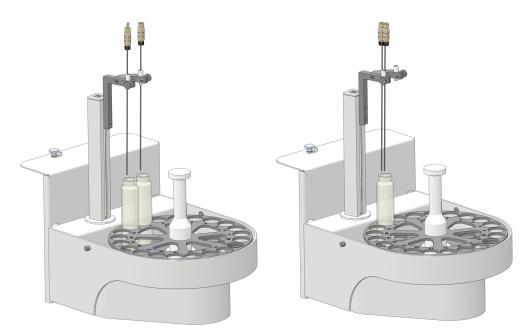


Fig. 24 Parallel purging (left) and sequential purging (right)

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.

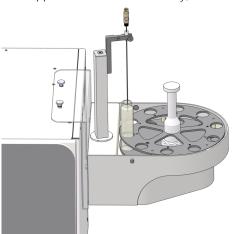


Fig. 25 Autosampler attached to the analyzer by means of the holder

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. 26 Connections on the bottom of the autosampler

- 1 Connection for equipotential bonding cable (grounding cable)
- 2 Power cable connection

3 Analyzer interface

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

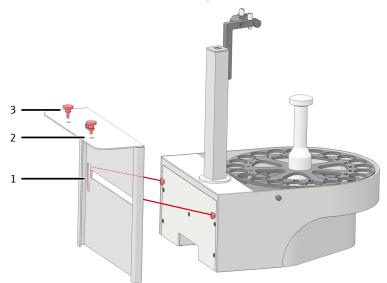


Fig. 27 Attaching the AS 21hp autosampler to the holder

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

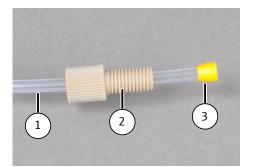


Fig. 28 Fingertight connection

1 Hose

2 Banjo bolt

3 Conical nipple

- Z Daliju Du
- Connect the power supply unit to the mains network.

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:
 - 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 I 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** section.
- 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 magnetic 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.

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".

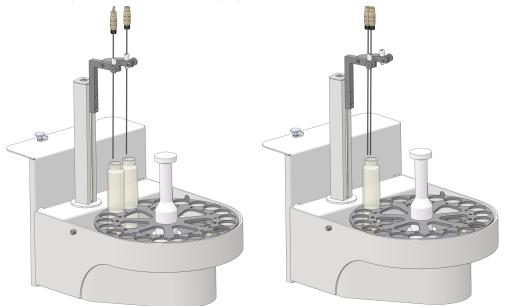


Fig. 29 Parallel purging (left) and sequential purging (right)

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

See also

Installing and commissioning the sampler [▶ 47]

4.3.2 AS vario autosampler



CAUTION

Risk of injury from moving parts

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

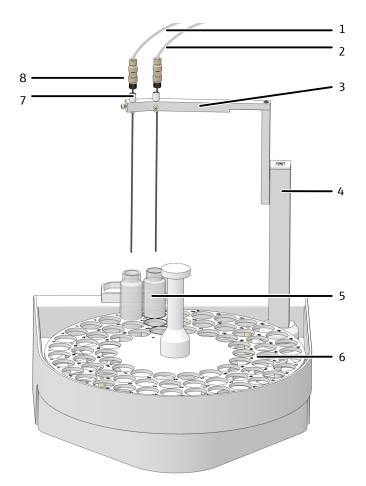


Fig. 30 Layout of the AS vario autosampler

- 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.

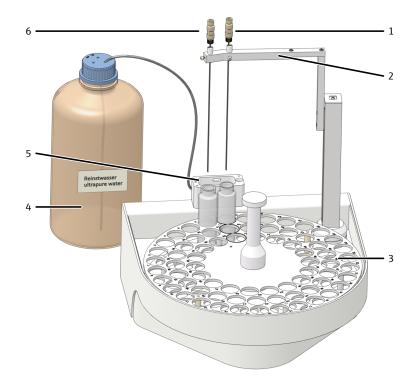


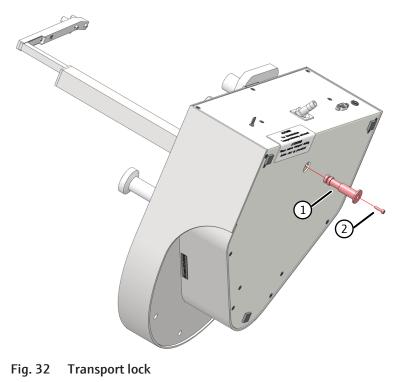
Fig. 31 Layout of the AS vario ER autosampler

- 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 parallel purging: Insert one canula each with sleeve in both positions of the canula holder (Fig. 30 B) 52).
- ▶ For NPOC measurement with non-parallel purging: Insert both canulas in one sleeve with two holes in the position on the right (see below, not suitable for AS vario ER).



Fig. 33 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.

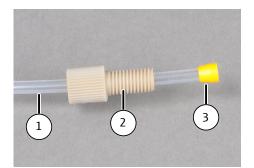


Fig. 34 Fingertight connection

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. 35 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 [▶ 80]

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.

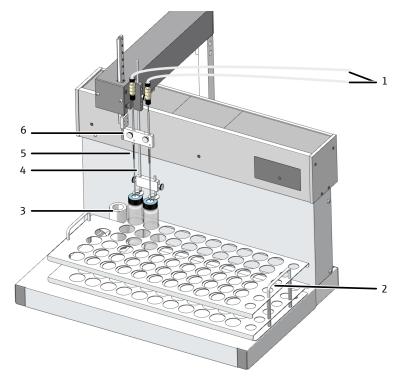
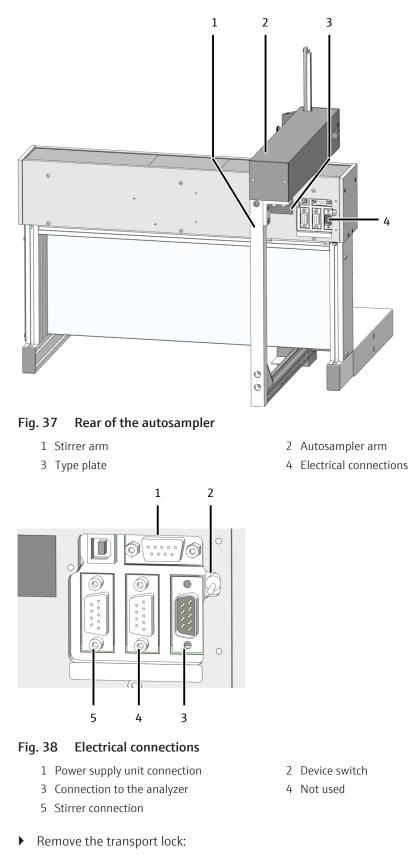


Fig. 36 EPA Sampler autosampler

- 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



 Remove the two countersunk screws with the A/F3 hexagon head wrench supplied.

- Remove the complete transport retaining clip and retain it for later transport.

pler

Commissioning the autosam-

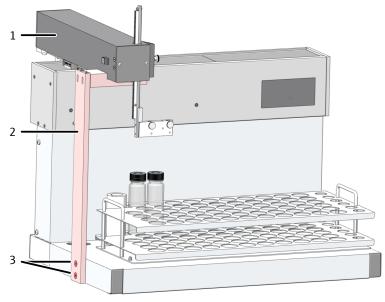
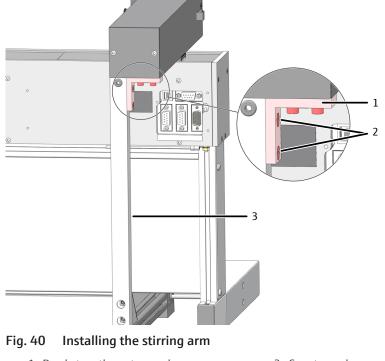


Fig. 39 Transport lock

- 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).

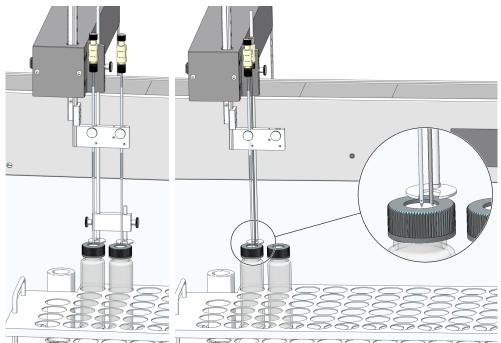


Fig. 41 Canula position for NPOC measurement with parallel (left) and non-parallel (right) purging.

- 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.

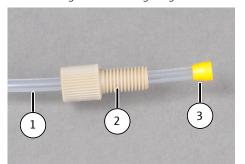


Fig. 42 Fingertight connection

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 [▶ 82]

4.3.4 POC module

The POC module is used for manual operation.

ParameterPOCDigestion principlePurging followed by thermal catalytic oxida-
tionSample volume1 mlSample supplyFlow injection, manual

Design

Technical data

The POC modules consist of the following components:

- POC reactor with septum
- Special sample intake canula for septum vials
- CO₂ adsorber with LiOH

Checking and extending the configuration

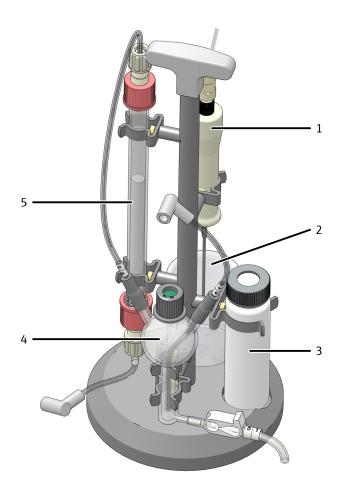


Fig. 43 POC module for manual operation

1 Canula with handle

3 Sample vessel with septum

5 Adsorption tube with LiOH

- 2 Waste container
- 4 POC reactor

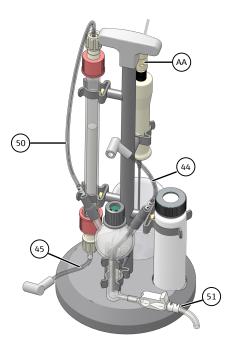
4.3.4.1 Installing the POC module

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

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

Insufficiently secured components pose a risk of injury.

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



After connecting the POC module for manual operation, the carrier gas flows constantly through the POC reactor into the combustion tube in the analyzer.

It is best to remove the reactor when measuring in other operating modes (TC, NPOC, etc.).

- Place the module to the left of the analyzer.
- Install the CO₂ adsorber tube between hose 50 (top end of the adsorber tube) and hose 45 (bottom end).
- Connect hose 50 to the POC reactor.
- Connect the POC module to the analyzer. Remove the hose protection bridge on the gas connections for the POC module on the rear of the analyzer.
- Connect hose 44 with the POC reactor and the 44 connection of the analyzer. The hose will almost reach the bottom of the POC reactor when doing this.
- Connect hose 45 of the POC module with the 45 connection of the analyzer.

\Lambda WARNING! Do not confuse the gas connections.

- Connect waste hose 51 to the bottom end of the TIC reactor. The waste hose is sealed with a hose clamp.
- Replace the standard intake canula on hose AA with the special canula with ventilation slot.

See also

B Checking the system for leaks [▶ 90]

4.3.4.2 POC calibration

Recommendation: Perform calibration with sucrose.

Procedure

- Create a POC method.
- Perform a calibration with an NPOC method and sucrose standards.
- Create a sequence for POC measurement with the Measurement | Add new sequence menu option.
- Use the Add by method menu option to create a measurement step with the prepared POC method.
- Select measurement step in sequence table. In Step properties | Tab Calibration select calibration from the dropdown menu at POC.

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

4.3.5 Chemiluminescence detector (CLD)

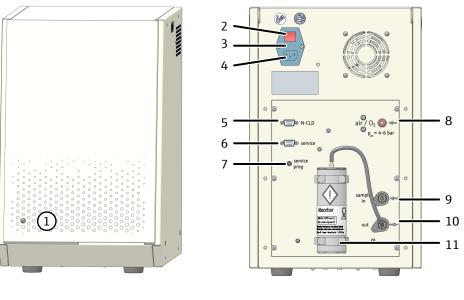


Fig. 44 Chemiluminescence detector (CLD)

- 1 Status LED
- 3 Fuse holder
- 5 RS 232 connection to the analyzer
- 7 Programming switch (service only)
- 9 "sample in" analyzer gas connection
- 11 Adsorber cartridge (removes NO_x from the waste air)
- 2 Mains switch
- 4 Power connection
- 6 Service connection
- 8 Carrier gas connection (O₂, synthetic/ purified air)
- 10 "out" sample outlet (gas)



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/HT" interface on the rear of the analyzer with the RS 232 interface on the detector via the supplied serial data cable.
- Switch on the detector. The status LED indicates operational readiness.

Checking and 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_b 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.

4.3.6 External solids module



NOTICE

Observe accessory instructions

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

• Observe the separate instructions for the accessory during installation.

Installation of the modular multi N/C 3300 duo measurement system for automated solids analysis is described in the separate operating manual for the HT 1300 solids module.

Connection to the analyzer

- Set up the solids module next to the analyzer.
- Connect the "analyte" connection on the solids module to the "analyte" connection on the rear of the analyzer.
- Connect the "pump" connection on the solids module to the "pump" connection on the rear of the analyzer.
- Connect the connection hose for oxygen to the gas supply pressure reducer and to the "oxygen" gas connection on the rear of the solids module. Set an inlet pressure of 400 to 600 kPa on the pressure reducer.
- Connect the supplied serial data cable to the "CLD/HT" connection on the rear of the analyzer. Connect the other end of the data cable to the solids module.
- Switch on the components of the analysis system. Start the software.
- Open the **Instrument** | **Manage instruments** menu option. Create a device configuration for solids analysis by clicking on the **Add** button.
- In Furnace type select the option External horizontal from the dropdown menu. Save the device configuration.
- Click on the Set default button to activate the device configuration as standard configuration.

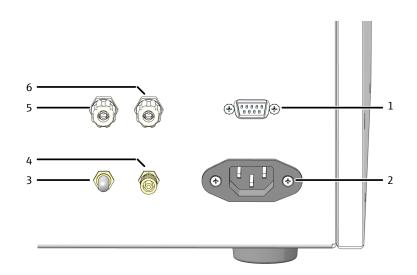


Fig. 45 Connections on the backplate of the solids module

- 1 Analyzer interface
- 3 Measuring gas outlet "OUT"
- 5 Pump connection "pump"
- 2 Power connection
- 4 Oxygen inlet "O₂"
- 6 Measuring gas connection "analyte"

4.4 Using the salt kit

For solutions with a high salt content, Analytik Jena recommends the optional salt kit, which significantly reduces wear. The kit contains a special furnace head and a combustion tube into which a salt vessel can be inserted.

The liquid sample is dosed into the salt vessel. When the sample evaporates, the salt remains in the salt vessel.



CAUTION

Skin and respiratory system irritation due to dust

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

4

3

2

1

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 filling, fix the combustion tube in a stand.
- Insert the platinum net (1) into the combustion tube and place it at the bottom center with a glass rod.
 The platinum net holds back the catalyst. Ensure that no catalyst can get into the gas pathway.
- Carefully stack platinum catalyst (2) onto the platinum net approx. 4 cm high.
- Pull the HT mat (3) apart and divide it into two rounds.
- Insert one half of the 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.
- Place the other half of the HT mat in the salt vessel. The HT mat serves as a splash guard.
- Insert the salt vessel (4) into the combustion tube with tweezers and place it on the catalyst filling.

Installing the furnace head

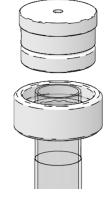
Install the furnace head on the combustion tube before inserting the combustion tube into the furnace.

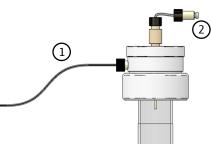
• Insert the O-ring into the upper part of the furnace head from below.



- Slide the union nut onto the filled combustion tube. Slide the recess in the union nut over the glass nose on the combustion tube.
- Secure the combustion tube in the union nut with a quarter turn.







- ▶ Place the furnace head on the combustion tube.
- Press the furnace head lightly against the tube and tighten hand-tight.
 Hold the union nut firmly so that the glass nose does not slip in the lock.

- Screw the connection for carrier gas (1) onto the side of the furnace head using the Fingertight connection.
- Insert the furnace canula (2) into the furnace head from above. Screw the Fingertight connection with the sample hose to the canula.

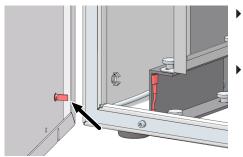
Inserting the salt kit into the furnace



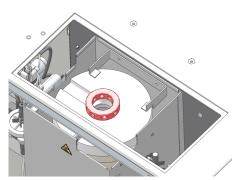
CAUTION

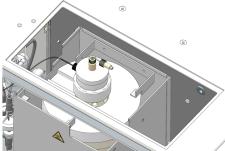
Risk of burns from the hot furnace, furnace head and combustion tube

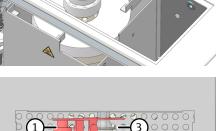
 Switch off the device and allow it to cool down before removing the combustion tube and inserting the salt kit.



- 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 standard combustion tube. Remove the ceramic holder for the standard combustion tube.
- Place the spacer on the combustion furnace.

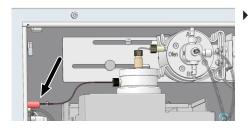




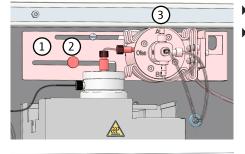


Insert the combustion tube with the furnace head into the combustion furnace.

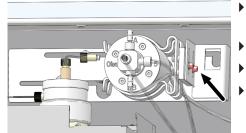
- Connect the lower end of the combustion tube and the inlet of the condensation coil via the spherical joint connection (3).
- Secure the spherical joint connection with the forked clamp (2). Tighten ▶ the knurled head screw (1) hand-tight.



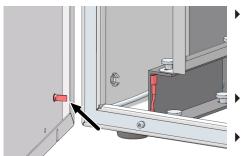
- Connect the carrier gas hose to the FAST connector in the housing wall.
- Loosen the knurled head screw (2) on the holder (1).



Slide the switching valve (3) to the left until the switching valve contacts the connection of the furnace canula.



- If necessary, adjust the height of the switching valve. To do so, undo the two hexagon socket screws on the right side.
- Fix the switching valve in the appropriate position.
- Screw the Fingertight connection hand-tight to the switching valve.
- 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.

I NOTICE! The furnace head of the salt kit is made of temperature-resistant plastic. The furnace head can become deformed when subjected to very high thermal loads.

When working with the salt kit, set a furnace temperature of maximum 720 $^\circ\!\mathrm{C}$ in the method.

See also

■ Removing the combustion tube [▶ 91]

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.
- For solutions with a high salt content, Analytik Jena recommends the optional salt kit, which significantly reduces wear on consumables. The kit is intended to digest solutions containing salt at a maximum of 720 °C.
- 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.
- The autosamplers use the following acid volumes for the automatic acidification of samples:

Capacity of the sample container	Acid volume
12 ml	50 µl
20 ml, 22 ml	100 µl
100 ml	500 µl
All other sample containers	166 µl

- For TIC detection, only use orthophosphoric acid (H₃PO₄, 10 %) made from concentrated acid (p.a.) and TOC water.
- Solutions made from the following are suitable as standard solutions: Potassium hydrogen phthalate, sodium carbonate/sodium hydrogen carbonate, sucrose.
- Only clean, particle-free glass containers (volumetric flasks, sample vessels) may be used for the preparation and storage of the solutions.
- When preparing and storing solutions with very low concentrations (<1 mg/l), observe that the laboratory air components (CO₂, organic vapors) can change the solution concentrations. The following measures can remedy this:
 - Keep the free space above liquids, the so-called headspace, as small as possible.
 - During autosampler operation, cover the vessels on the sample tray with foil.
 This is important in particular with differential mode, as the samples remain on the sample tray for a longer time.
 - Eliminate the source of organic vapors.
 - Optionally: Fill the headspace above the samples with inert gas.

5.2 Switching on the analyzer



NOTICE

Risk of device damage due to depleted copper wool

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

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

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 [▶ 108]

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].
- Flush the analysis device, for measurements without autosampler:
 - Activate the checkbox **Reverse rinse**. Hold the sample intake cannula in the waste canister.
- Flush the analysis device, for measurements with AS vario, EPA Sampler autosampler:
 - Activate the checkbox **Reverse rinse**. The content of the sample intake hose is automatically washed back into the wash cup.
- Flush the analysis device, for measurements with AS 10e, AS 21hp autosampler:
 - At the end of the sequence, measure one sample of ultrapure water.
 (The autosamplers do not have the wash cup required for the backflush process.)
- Exit the dialog with **OK**.
 - \checkmark 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 \times 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 .

- Flush the analysis device, for measurements without autosampler:
 - Activate the checkbox **Reverse rinse**. Hold the sample intake cannula in the waste canister.
- Flush the analysis device, for measurements with AS vario, EPA Sampler autosampler:
 - Activate the checkbox **Reverse rinse**. The content of the sample intake hose is automatically washed back into the wash cup.
- Flush the analysis device, for measurements with AS 10e, AS 21hp autosampler:
 - At the end of the sequence, measure one sample of ultrapure water.
 (The autosamplers do not have the wash cup required for the backflush process.)
- 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
measurementAt 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.

74

- 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
 NOTICE! It is not possible to start the measurement without a results table.
- Check the finished sequence for plausibility by clicking on e. The software checks whether the created measuring steps can be measured.
- ► If necessary, save the sequence with ^L. 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.
- \blacktriangleright Start the measurement by clicking on \blacktriangleright . Follow the instructions on the screen.
 - $\checkmark\,$ 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
 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 ^L. 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 \blacktriangleright .
 - ✓ 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.

6 Maintenance and care

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

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

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

6.1 Maintenance overview

Analyzer

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

Sample supply system and autosampler

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

Hose system

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

Combustion system

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

Optional salt kit

Maintenance interval	Maintenance task	
lf necessary, after approx. 500 injec- tions	Clean the salt vessel.Replace the catalyst.	

Measuring gas drying and cleaning

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

POC module

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

Chemiluminescence detector (CLD)

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

6.2 Adjustment and setting

6.2.1 General notes for adjusting the autosampler

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

An adjustment of the autosampler is necessary:

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

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

See also

Installing and commissioning the sampler [▶ 47]

6.2.2 Adjusting the AS vario autosampler



NOTICE

Risk of bending

The canula may bend during adjustment.

- Unscrew the screw connections on the canulas before adjustment.
- Start the software
- 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.

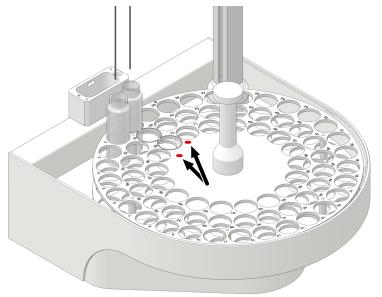


Fig. 46 Adjustment points on the sample tray

• 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:

ampler adj	ustment	-		6			AS Vario[72] at MultiN	C 33
ampler Lo	ocations			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	
	Reque	st current offsets						
osition1	Reque	st current offsets						
osition1 higher / + lo	_		139					

Fig. 47 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.

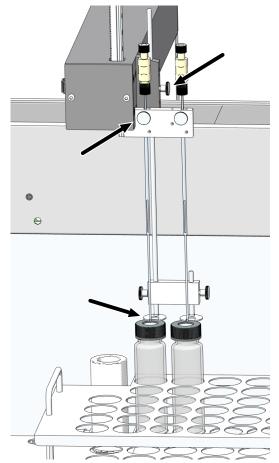


Fig. 48 Install canulas (here: 2 canulas for parallel purging)

- 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.

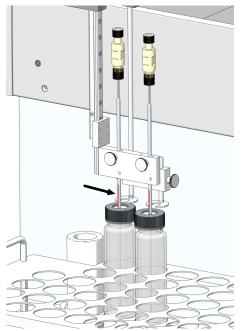


Fig. 49 Adjusting position 1

- 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:

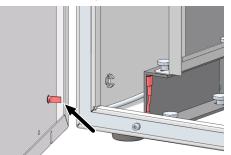


Fig. 50 Grounding conductor connection on the side wall

- 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.

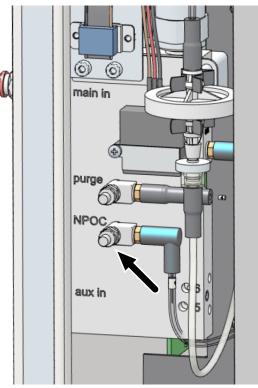
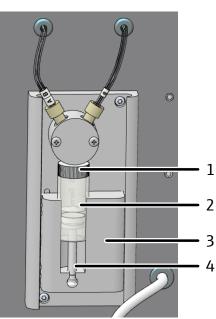


Fig. 51 Setting the NPOC purge flow

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

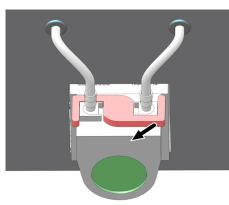
Risk of chemical burns during hose replacement

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

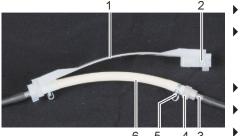
- Wear protective gloves and clothing when replacing the hoses.
- Collect any leaking liquids with an absorbent sheet.

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



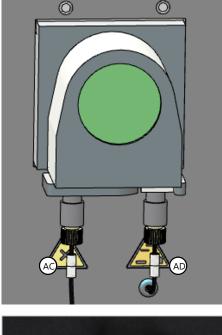
6 5 4 3

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

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

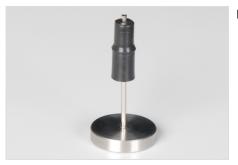
- Shut down control and evaluation software or turn off the gas flow with the Instrument | Gas flow off menu option.
- Remove the pump hose as with the condensate pump.



- 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

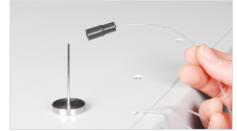
FAST connectors connect hoses with glass components. Use the threading aid to feed thin hoses into the connectors. This is included with the analyzer. Check the system for leaks after hose replacement.



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.

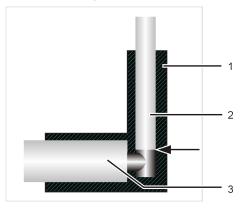


Fig. 52 FAST connector, angled

- 1 Angled FAST connector
- 3 Glass connection

2 Hose

Fingertight connections

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

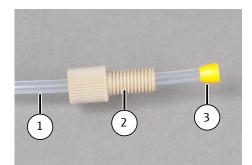


Fig. 53 Replacing the Fingertight connection

- 1 Hose
- 3 Conical nipple

2 Banjo bolt

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) 160 ml/min
 - Out: (outlet flow) 150 to 170 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 [▶ 122]

6.7.1 Removing the combustion tube

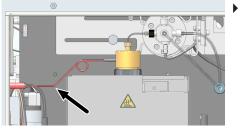


CAUTION

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

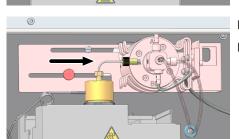
• Switch off the device and allow it to cool down before installation and maintenance.

- 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.

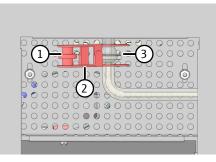


Pull the carrier gas canula out of the FAST connector on the left side wall.

Unscrew the Fingertight connection of the furnace canula from the 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.

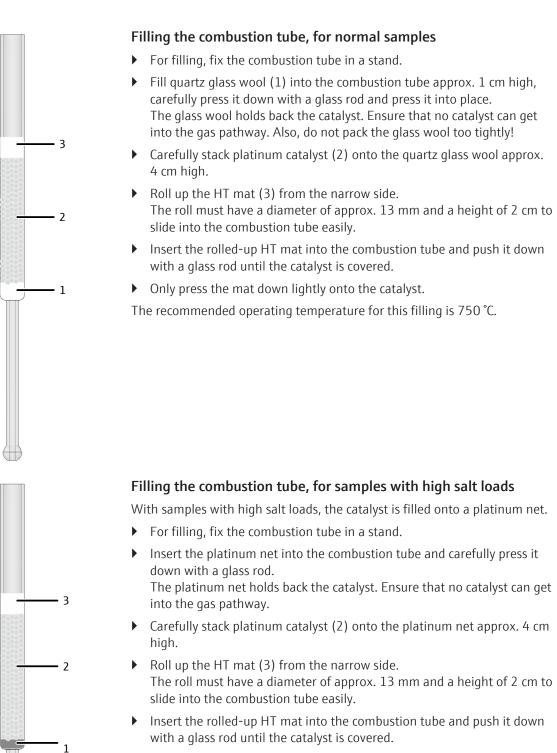


NOTICE

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.

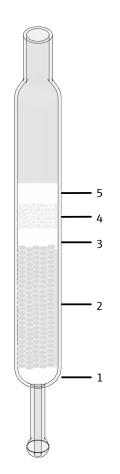


• Only press the mat down lightly onto the catalyst.

The recommended operating temperature for this filling is 720 to 750 $^\circ\!C.$

Using the optional salt kit instead will reduce wear on consumables.





Filling the special combustion tube with CeO₂ catalyst

The special combustion tube has a larger diameter (26 mm).

- 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 CeO₂ catalyst (2) onto the quartz glass wool approx. 4 cm high. Alternatively, use the platinum catalyst.
- Cover the catalyst with a layer of quartz glass wool (3) approx. 1 cm high. Push the glass wool down with a glass rod and only press it lightly onto the catalyst.
- Fill ground quartz glass (4) into the combustion tube approx. 1 cm high.
- Cover the ground quartz glass with a round piece of HT mat (5).

The recommended operating temperature for this filling is 850 °C.

6.7.3 Installing the combustion tube



NOTICE

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

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

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



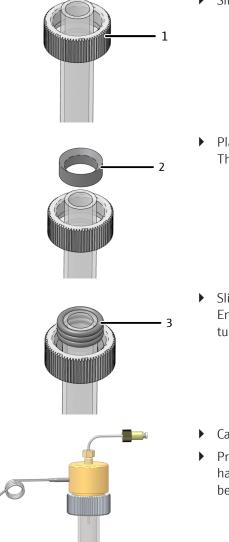
NOTICE

Preventing tightness problems

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

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

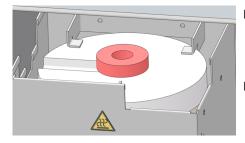
Install the furnace head on the combustion tube before inserting the combustion tube into the furnace.



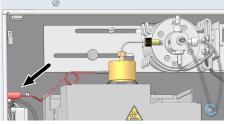
• Slide the union nut (1) onto the combustion tube.

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

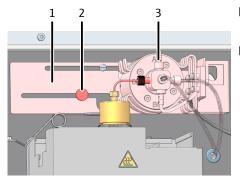
- Slide the three coated sealing rings (3) onto the combustion tube.
 Ensure that the sealing rings are flush at the edge of the combustion tube.
- Carefully put the furnace head onto the combustion tube up to the stop.
- Press the furnace head lightly against the tube and tighten the union nut hand-tight. The furnace canula and the carrier gas canula have already been attached to the furnace head.



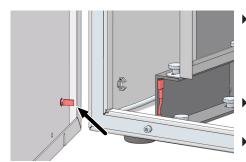
- ▶ For the standard combustion tube (16 mm diameter), place the ceramic holder on the top opening of the combustion furnace. Do not use the ceramic holder for the special combustion tube with CeO₂ catalyst (26 mm diameter).
- Insert the combustion tube with the furnace head into the combustion furnace.
 - Connect the lower end of the combustion tube and the inlet of the condensation coil via the spherical joint connection (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 in the device wall with the FAST connector.
- Slide the change-over valve to the left until the change-over valve contacts the connection of the furnace canula.
- Screw the furnace canula with the Fingertight connection finger-tight to the change-over valve.



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



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

6.8 Optional salt kit maintenance

You will need to clean the salt kit and replace the catalyst if analytical problems occur, such as a change in peak shape or poor recovery of standard solutions. These problems depend to a large extent on the salt composition, the salt load of the samples, and the method settings such as injection volume and furnace temperature.



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.

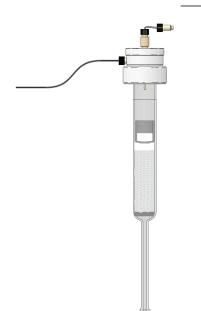


CAUTION

Skin and respiratory system irritation due to dust

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.



- Remove the salt kit from the device.
- Remove the furnace head. Check the O-ring in the furnace head for wear. Replace as necessary.
- Remove the salt vessel with tweezers. Rinse with ultrapure water and allow it to dry well. When analyzing solutions containing heavy metals, dispose of rinsing water in accordance with local regulations.
- 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.
- Fill the combustion tube with catalyst.
- Fit the salt vessel with HT mat and insert it into the combustion tube.
- Mount the salt kit again and insert it into the device.

See also

- Replacing the catalyst [▶ 90]
- Using the salt kit [▶ 66]

6.9 Removing and installing the combustion furnace

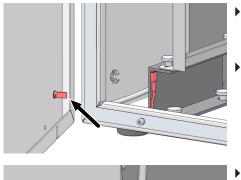
6.9.1 Removing the combustion furnace

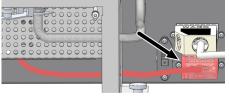


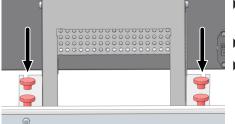
CAUTION

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.





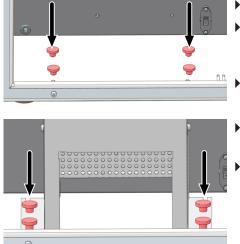


- Switch off the analyzer via the main switch. Disconnect the power plug from the socket. Shut off the gas supply on the pressure reducer in the laboratory.
- Open the left side wall of the analyzer. Push the accessory modules to the side if necessary. Do not kink any connection hoses.
 - Unscrew the 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.

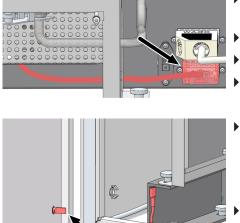
See also

- Removing the combustion tube [> 91]
- Condensation coil maintenance [▶ 100]

6.9.2 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.



0

- 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.

6.10 Cleaning the TIC condensate vessel



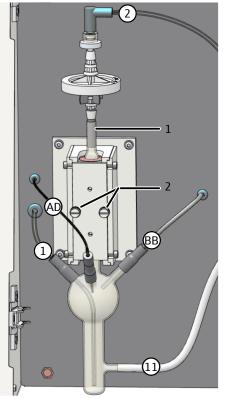
WARNING

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.

6.11 Condensation coil maintenance

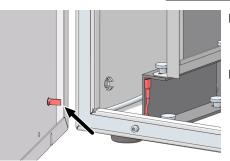
Removal and cleaning



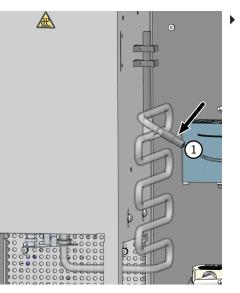
CAUTION

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

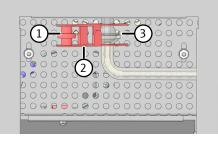
• Switch off the device and allow it to cool down before installation and maintenance.



- 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.



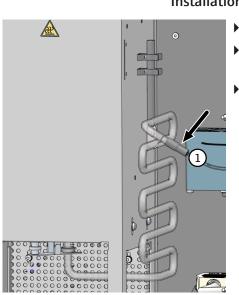
Pull hose 1 out of the FAST connector of the condensation coil.

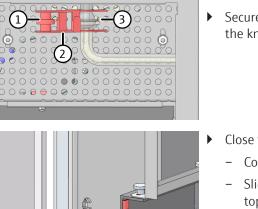


- Release the joint connection (3) on the bottom of the combustion furnace connecting the combustion tube with the condensation coil.
- To do this, unscrew the knurled head screw (1) and remove the fork clamp (2).
- Carefully remove the condensation coil from the analyzer. Pull the bottom part out of 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.
- Hold the condensation coil against the right side of the combustion furnace. The spherical joint of the coil points toward the lower opening of the furnace.





- Connect the lower end of the combustion tube and the inlet of the condensation coil via the spherical joint connection (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.

6.12 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.

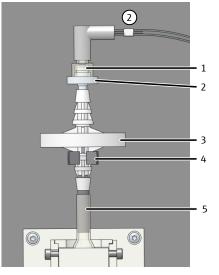


Fig. 54 Replacing the water traps on the front side

- 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.

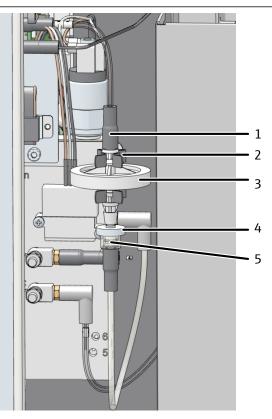


Fig. 55 Replacing the water traps on the gas box

- 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

Checking the system for leaks [▶ 90]

6.13 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.

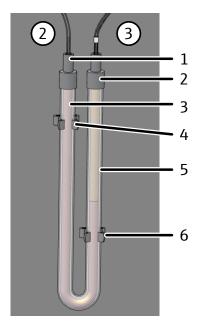


Fig. 56 Replacing the halogen trap

- 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.

6.14 POC module maintenance

Checking the adsorber function

The LiOH adsorber can clump over time.

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

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

Checking the module for leaks



NOTICE

Risk of gas leakage

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

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

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

- Switch on the analyzer.
- Open the carrier gas supply on the pressure reducer.
- Start the control and analysis software.
- Check the flow display in the **Instrument status** panel:
 - In: (inlet flow) 160 ml/min
 - Out: (outlet flow) 150 to 170 ml/min
- If the flow decreases or the outlet flow is significantly below the inlet flow, check all hose connections and septums. Eliminate the leak.
 - ✓ The device system has no leaks.

6.15 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.

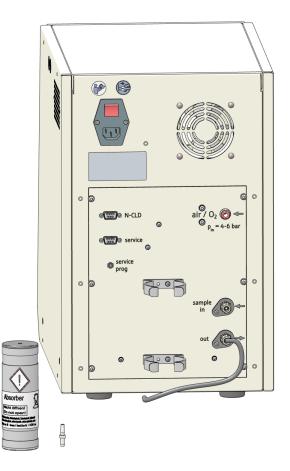


Fig. 57 Replacing the adsorber cartridge

- 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	 Incomplete command from the PC PC command without STX PC command without * PC command CRC error PC command invalid command PC command invalid MESS command
Cause	Remedy
 Faulty connection between the internal and external program 	 Initialize the analyzer.
Error code: Error message	7: COM 2 not found 8: COM 3 not found 9: COM 4 not found
Cause	Remedy
Internal hardware problems	Switch the analyzer off/on.
 The water trap is clogged. 	 Reinitialize the analyzer. Check if the gas pressure error occurs again. If not, replace the water traps.
 No gas flow at the measuring outlet due to kinked hose for sample gas supply 	 Check the hose. If necessary, eliminate any kinks.

7: COM 2 not found 8: COM 3 not found		
ee- onden- re er- on- r. vs fill e first		
tion the r of talyst st.		
2		
tialize		
inlet		
inlet		

_

Error code: Error message	24: Optics error. analog values out of range		
Cause	Remedy		
 The analog values of the detector are outside of the working range. 	 Check the quality of the carrier gas. Initialize the analyzer and check the analog values via the component test. 		
Cause	Remedy		
 The analog values of the detector are outside of the working range. 	 Check the quality of the carrier gas. For solids methods and connection of the HT 1300 module: Set the carrier gas flow higher than the intake flow. Initialize the analyzer and check the analog values via the component test. 		
Error code: Error message	30: No connection to N sensor		
Cause	Remedy		
 The nitrogen detector is not switched on. The connection cable is not connected is faulty. Incorrect connection. 	Switch on the detector.Check the connection cables.orCheck the connection.		
Error code: Error message	40: No connection to the syringe pump		
Cause	Remedy		
 No communication between the ana- lyzer and the syringe pump. 	 Initialize the analyzer. Switch the PC off and back on again and initialize the analyzer. 		
Error code: Error message	80: No connection to temperature con- troller		
Cause	Remedy		
No connection to the solids module.The solids module is not switched on.Incorrect connection.	Check the connection cables.Switch on the optional solids module.Check the connection.		
Error code: Error message	82: Thermocouple HT furnace interruption (HT)		
Cause	Remedy		
Faulty thermocouple.	 Inform the service. 		
 Furnace not connected. 	 Connect the furnace. 		
• The furnace temperature is too high	 Inform the service. 		
Error code: Error message	84: Communication error HT furnace tem- perature controller		
Cause	Remedy		
Communication error.	 Inform the service. 		
Error code: Error message	86: No external furnace found		
Cause	Remedy		
 No connection to the solids module. 	Check the connection cables.		
	· · ·		

Error code: Error message	111: Rotator error	
Cause	Remedy	
 The drive is incorrectly positioned, e.g. jammed. The drive is faulty. 	 Initialize the analyzer. If the error cannot be corrected, contact the service. 	
Error code: Error message	112: Swivel drive error	
Cause	Remedy	
 The drive is incorrectly positioned, e.g. jammed. The drive is faulty. 	 Initialize the analyzer. If the error cannot be corrected, contact the service. 	
Error code: Error message	113: Lifting drive error / Sampler: z drive error (steps lost)	
Cause	Remedy	
The drive is incorrectly positioned, e.g. jammed.The drive is faulty.	 Initialize the analyzer. If the error cannot be corrected, contact the service. 	
Error code: Error message	114: Rack detection error	
Cause	Remedy	
 The sample tray is not positioned cor- rectly. 	Position the sample tray again and make sure it clicks into place.Initialize the analyzer.	
Error code: Error message	115: Wrong rack	
Cause	Remedy	
 The wrong sample tray is set in the soft- ware. 	Check the device configuration settingsIf necessary, set a different sample tray.	
Error code: Error message	116: Unknown sampler command	
Cause	Remedy	
Communication error.	 Inform the service. 	
Error code: Error message	201: Restart the internal program	
Cause	Remedy	
 Internal program error. 	 Initialize the analyzer. For repeated occurrences, monitor precisely at which time the error occurs. 	
Cause	Remedy	
Communication error.Syringe pump faulty.	Initialize the analyzer.Inform the service.	

Error code: Error message	409: Syringe pump: pump sluggish	
Cause	Remedy	
 A hose line is clogged. 	 Search for the cause and remedy the fault. Clean or replace the hose line. Initialize the analyzer. 	
 Syringe pump faulty. 	 Inform the service. 	
Error code: Error message	410: Syringe pump: valve sluggish	
Cause	Remedy	
Syringe pump faulty.The valve is broken.	 Inform the service. 	
Error code: Error message	415: Syringe pump: invalid command	
Cause	Remedy	
Communication error.	 Initialize the analyzer. 	
 Syringe pump faulty. 	Inform the service.	

7.2 Status errors

Status errors are displayed in the **Instrument status** device panel.

Error indication	In 160 ml/min; Out < 150 ml/min Remedy	
Cause		
 The union nut on the combustion tube is not tightened correctly (after catalyst replacement). The carrier gas supply to the furnace head or lock is not connected properly (after catalyst replacement). The sealing rings on the combustion tube are defective (heavily deformed) or not attached (after catalyst replacement). The FAST connector on the TIC condensate container is leaky. The connections on the water trap systems are loose (after water trap replacement or halogen trap maintenance). 	 Check the screw connections for completeness and deformations. Tighten as necessary. Check the carrier gas supply, in particular the FAST connector on the analyzer wall and the screw connection on the furnace head. Check all connection points on the water traps. Replace the FAST connector as necessary. 	
 The connection between the combustion tube and the condensation coil or the screw connections are leaky. 	 Check the connection of the combustion tube to the condensation coil, in particu- lar the fork clamp position. 	
 The combustion tube is faulty (cracks, fractures at the edge). TIC condensate container faulty (fractures on the connections). 	 Check the glass components. Replace as necessary. 	
 Water traps clogged. 	 Replace the water traps. 	
• The condensate pump hose is leaky.	 Check the condensate pump. Replace the hose as necessary. 	

Error indication	In 160 ml/min; Out < 150 ml/min; Out > 170 ml/min		
Cause	Remedy		
 The MFM (mass flow sensor) is faulty. 	 Check the flow with an external mass flow sensor if possible to confirm the er- ror. Inform the service. 		
 The filling of the halogen trap is used up. 	Check the halogen trap.		
Error indication	In < 160 ml/min; Out < 150 ml/min		
Cause	Remedy		
No carrier gas.The hose line is leaking.	Turn on the carrier gas on the pressure reducer.Search for and remedy the leak.		
 The inlet pressure of the carrier gas sup- ply is too low. 	 Set the carrier gas inlet pressure cor- rectly. 		
 The pressure switch in the analyzer was triggered simultaneously with error 10: Gas pressure error. 	 See the remedy for 10: Gas pressure e ror. 		
 The MFC is faulty. 	 Inform the service. 		
Error indication	In < 160 ml/min;Out:155 165 ml/min		
 No carrier gas. 	 Turn on the carrier gas on the pressure reducer. 		
 The inlet pressure of the carrier gas sup- ply is too low. 	 Set the carrier gas inlet pressure cor- rectly. 		
 The MFM is faulty. 	 Inform the service. 		
Error indication	In 160 ml/min; Out > 170 ml/min		
Cause	Remedy		
 Peltier cooling insufficient. 	 Check the cooling from above on the T condensate container. The formation condensation water on the cooling blo indicates that the cooling is working. 		
 The MFC is faulty. 	 Inform the service. 		
Error indication	In; Out = 0 ml/min		
Cause	Remedy		
 A hose line is clogged. 	 Remove and rinse the clogged hose line Reinstall it again afterward. Replace the clogged hose line. 		
No method loaded.	Load a method.		
Error indication	NDIR detector values highlighted in color in the Instrument status panel		
 The analog values of the detector are at the edge of the working range. 	 Check the halogen trap. Replace the fill- ing if necessary. Contact the application team and get tips on application regulations for diffi- cult sample matrices. 		

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.

Water traps clogged Remedy	
Scattering measurements	
Remedy	
 Replace the catalyst. 	
Check the dosing.	
 Replace the canula. 	
Warm up cold samples before analysis.Filter samples prior to analysis.	
 Stir particulate samples. When measur- ing with an autosampler, adjust the stir- ring speed in the method. 	
 Prevent the addition of CO₂ or organic vapors from the ambient air. Check the ambient conditions and remedy the source of the fault. Cover the sample vessels on the autosampler with aluminum foil. Treat the headspace of the sample with gas. 	
 Check the method settings. If necessary, increase the maximum integration time. 	
Canula faulty	
Remedy	
 Misting up of the canula is normal. Replace the canula if the sample is no longer dosed as a cohesive jet but is sprayed. 	

Error	Autosampler does not draw in sample without air bubbles Remedy	
Cause		
 The sample intake path is leaky. 	 Check the hose connections. If necessary, tighten loose hose connections to the canula or to the valve of the syringe pump. 	
 The sample intake canula is clogged. 	 Remove the canula and clean it in an ul trasonic bath. Replace the canula. 	
 The dosing syringe is leaky. The PTFE sealing lips of the plunger are damaged. 	Remove and check the dosing syringe.Replace the dosing syringe.	
Error	Incomplete dosing in the reactors	
Cause	Remedy	
 The dosing path is leaky. 	 Check the hose connections. If neces- sary, tighten loose connections: Syringe pump to change-over valve, change- over valve to injection canula, change- over valve to TIC condensate container. 	
Error	Carry-over	
Cause	Remedy	
 Insufficient syringe flushing. 	 Flush the dosing syringe with sample before the next injection. To do this, edit the method 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	
The catalyst is used up.	 Replace the catalyst. 	
 The system is leaking. 	 Check the system for leaks. 	
 Faulty dosing. 	Check the dosing.	
 Particulate samples are not or are insuf- ficiently stirred. 		
Error	Low results for TC, TOC, NPOC and TNb analyses (TIC analyses OK)	
Cause	Remedy	
 The catalyst is used up. 	 When using the platinum catalyst and measuring in differential mode (neutral to slightly alkaline samples): The catalyst can be regenerated. Inject ultrapure water that has been acidified six times (pH <2). Recommendation: Measure one or two sample vessels with acidified ultrapure water per analysis series. Replace the catalyst. After replacing the catalyst, perform calibration. 	

Error	Low results for TIC analyses (TC, TOC, NPOC analyses OK)		
Cause	Remedy		
 No phosphoric acid in the reagent bot- tle. 	 Refill the bottle. 		
 Incorrect sample dosing. 	Check the dosing.		
Error	Low results for TNb analyses		
Cause	Remedy		
The catalyst is used up.	 Replace the catalyst. 		
 The sample concentration is above the calibrated range. 	 Observe the calibrated range. Use quadratic regression. If possible, calibrate dependent on the matrix. For analysis of unknown substances, use low concentrations if possible. If possible, dilute the sample. Use synthetic air as a carrier gas. 		
Error	Unusual peak shape during TC and TNb analyses		
Cause	Remedy		
 The catalyst is used up. 	 Note: Low results also occur at the same time. Regenerate or replace the catalyst. 		
 Unsuitable integration criteria. 	 Check the integration criteria in the method. 		
 Measuring range for CLD exceeded. 	Dilute the sample.		
 Faulty dosing. 	 For manual sample supply: Ensure even injection. 		
Error	Incorrect TNb analyses with CLD (TC analyses OK)		
Cause	Remedy		
Faulty hose connection between the an- alyzer and the detector.Ozone generator faulty.	Check the hose connections.Inform the service.		
Error	Condensate pump or phosphoric acid pump leaky		
Cause	Remedy		
Leaking hose connections.Defective pump hose.	Check the connections.Replace the hose.		
Error	5 V, 24 V indicator lamps on LED strip not lighting		
Cause	Remedy		
 Power supply or electronics fault. 	Check the electrical connections.Check the laboratory power supply.		
 Device fuse faulty. 	 Inform the service. 		
Error	Status LED on the analyzer not lighting		
Cause	Remedy		
 The internal program has not been started. 	 Switch the analyzer off and on again. 		

Error	Heating monitoring lamp on LED strip not lighting	
Cause	Remedy	
 Device in standby with standby tempera- ture = room temperature 	 Initializing the device 	
 Faulty thermocouple (furnace). The "Broken Thermocouple" lamp is lit on the LED strip. 	 Inform the service. 	
Faulty electronics component.	 Inform the service. 	
 The combustion furnace is not con- nected correctly. 	 Check the connection of the combustion furnace. 	

8 Transport and storage

8.1 Transport

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

Avoid the following during transport:

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

8.1.1 Preparing the analyzer for transport



CAUTION

Risk of burns from the furnace, 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.

A 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 [▶ 78]

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.

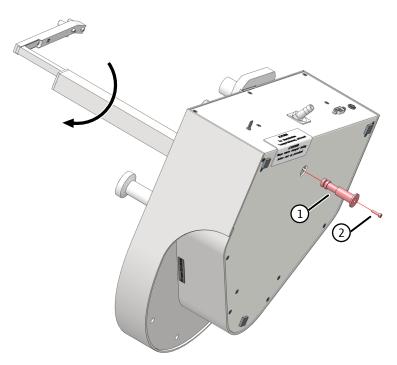


Fig. 58 Securing the autosampler for transport

1 Transport lock

2 M3x12 screw

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

8.1.3 Moving the device in the laboratory



CAUTION

Risk of injury during transport

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

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

Observe the following when moving the device within the laboratory:

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

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

8.2 Storage



NOTICE

Risk of device damage due to environmental conditions

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

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

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

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

9 Disposal

Waste water	Waste water containing acids and samples occurs during device operation. Dispose of the neutralized waste in accordance with the legal requirements.
Halogen trap	The halogen trap contains copper and brass. Contact the responsible institution (author- ity or waste disposal company). There you will receive the information regarding recy- cling or disposal.
Catalyst	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.
Analyzer	At the end of its service life, the device and its electronic components must be disposed of as electronic waste in accordance with the applicable regulations.

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.

Specifications 10

10.1 Technical data of the basic device

General characteristics	Designation/type		multi N/C 3300
		I	multi N/C 3300 duo
	Order numbers		11-0118-101-62 (multi N/C 3300)
			11-0118-102-62 (multi N/C 3300 option- ally with ChD)
	Basic device dim	ensions (W x D x H)	513 x 547 x 464 mm
	Basic device mas	is i	21 kg
	Sound pressure I	evel	<70 dB(A)
Methods data	Disastian princip		
	Digestion principle		Thermocatalytic oxidation
	Digestion tempe	Ť	Up to 950 °C, depending on catalyst
	Sample feed		Flow injection 50 to 1000 μl
	Sample volume		· · · · · · · · · · · · · · · · · · ·
	Particle handling		In accordance with DIN EN 1484
	Carbon detection	· · ·	NDIR (coupled with the VITA method)
			0 to 30000 mg/l 0 to 500 mg
		00 solids module)	
Nitrogen detection			
Nillogen detection	Nitrogen detectio		CLD
			ChD
	TN _b measuremen		0 to 20000 mg/l
	TN _b measuremen		0 to 10000 mg/l
Process control	Control and analysis software Software function scope		multiWin pro
			Real-time graphics, status indication during analysis, graphical display of the measured results, 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 cylin	Hydrocarbon and CO ₂ -free der)
	Option 3	Purified compressed air	CO ₂ <1 ppm

400 to 600 kPa

Inlet pressure

	Flow rate	15 l/h, depending on me	easuring mode	
	Analyte gas flow	160 ml/min		
	NPOC purge flow	50 to 160 ml/min		
Electrical variables	Voltage		115/230 V	
	Frequency		50/60 Hz	
	Fuses		2 T6.3 A H	
	Typical average power consumption		400 VA	
	Maximum power consumption		500 VA	
	PC interface		USB 2.0	
	Module/accessory interface		RS 232	
Ambient conditions	Operating temperature		+10 to 35 °C (air-conditioning recom- mended)	
	Maximum humidity		90 % at 30 ℃	
	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	
unements	Disk drive			
unements			Min. 64 GB	
unements	RAM		Min. 8 GB	
unements	RAM Screen resolution		Min. 8 GB Min. 1920 x 1080 px	
unements	RAM		Min. 8 GB	
unements	RAM Screen resolution		Min. 8 GB Min. 1920 x 1080 px compatible with DirectX 12 or higher, with	
	RAM Screen resolution Graphic card		Min. 8 GB Min. 1920 x 1080 px compatible with DirectX 12 or higher, with WDDM 2.0 driver Min. 1 USB 2.0 interface, to connect the ba-	

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						
AS 2 THP autosampler	Sample positions		x	21		
	Tube size		50 ml			
	Purging NPOC samples		Parallel and sequential			
	Magnetic stirrer (integrated)		Homogenizat ticles	Homogenization of samples containing par- ticles		
AS 10e autosampler	Sample positions		10			
· · · · · · · · · · · · · · · · · · ·	Tube size		50 ml			
	Purging NPOC samples			Sequential only		
AS vario autosampler	Order number (designation)		11-0514-00	11-0514-003-26 (AS vario)		
	Order number (designation)	Order number (designation)		4-26 (AS vario E	R with cannula	
	Dimensions (W x D x H)		350 x 400 x	470 mm		
	Mass		15 kg			
	Operating voltage		24 V DC via e	xternal power s	upply	
AS vario autosampler	Power supply, external powe	r supply unit	100 to 240 V	100 to 240 V, 50/60 Hz (autosensing)		
	Power consumption			50 VA		
	Sample tray with sample positions	Tube size		AS vario	AS vario ER	
	20	100 ml		Yes	No	
	47 (dilut)	12 ml + 50 r	12 ml + 50 ml		Yes	
	52	100 ml		Yes	No	
	72	100 ml 40 ml + 50 ml (optional)	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 _b			
	Detection limit			0,005 mg/l TN _b		
	Analysis time		3 to 5 min			
	Gas for ozone generation		Gas supply as for basic device			
			60 ml/min, 400 to 600 kPa:			

Dimensions (W x 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	 The device complies with the following safety standards EN 61010-1 EN 61010-2-081 EN 61010-2-010 EN 61010-2-051 (for operation with autosampler)
EMC compatibility	 The device has been checked for transient emissions and noise immunity. 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. 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).
Environmental and ambient in- fluences	 This device has been tested in environmental simulations under operation and transport conditions and is in accordance with the requirements in: ISO 9022-2 ISO 9022-3
EU directives	The device meets the requirements of the directive 2011/65/EU. The device is designed and tested in accordance with standards meeting the 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.

List of figures

Fig. 1	Analyzer, front doors opened	15
Fig. 2	Analyzer, left side wall opened	
Fig. 3	Syringe pump	
Fig. 4	Hose diagram	17
Fig. 5	Setting the NPOC purge flow	
Fig. 6	Condensate pump	
Fig. 7	Phosphoric acid pump	19
Fig. 8	FAST connector	19
Fig. 9	Fingertight screw connection	20
Fig. 10	Combustion furnace	20
Fig. 11	Condensation coil	21
Fig. 12	TIC condensation module	22
Fig. 13	Water traps	23
Fig. 14	Halogen trap	23
Fig. 15	Status LED	25
Fig. 16	LED strip (right front door open)	25
Fig. 17	Device rear	
Fig. 18	Principle of operation	
Fig. 19	Space required for multi N/C 3300 with modules	
Fig. 20	Space required for multi N/C 3300 duo modular measuring system	
Fig. 21	Device rear	
Fig. 22	AS 10e autosampler	
Fig. 23	AS 21hp autosampler	
Fig. 24	Parallel purging (left) and sequential purging (right)	
Fig. 25	Autosampler attached to the analyzer by means of the holder	
Fig. 26	Connections on the bottom of the autosampler	
Fig. 27	Attaching the AS 21hp autosampler to the holder	
Fig. 28	Fingertight connection	
Fig. 29	Parallel purging (left) and sequential purging (right)	50
Fig. 30	Layout of the AS vario autosampler	52
Fig. 31	Layout of the AS vario ER autosampler	53
Fig. 32	Transport lock	53
Fig. 33	Sleeve with two canulas for non-parallel purging	
Fig. 34	Fingertight connection	55
Fig. 35	Canula flush on the AS vario ER model	
Fig. 36	EPA Sampler autosampler	57
Fig. 37	Rear of the autosampler	58
Fig. 38	Electrical connections	58
Fig. 39	Transport lock	59

Fig. 40	Installing the stirring arm	59
Fig. 41	Canula position for NPOC measurement with parallel (left) and non-parallel (right) purging	60
Fig. 42	Fingertight connection	61
Fig. 43	POC module for manual operation	62
Fig. 44	Chemiluminescence detector (CLD)	64
Fig. 45	Connections on the backplate of the solids module	66
Fig. 46	Adjustment points on the sample tray	80
Fig. 47	Sampler alignment window	81
Fig. 48	Install canulas (here: 2 canulas for parallel purging)	82
Fig. 49	Adjusting position 1	83
Fig. 50	Grounding conductor connection on the side wall	84
Fig. 51	Setting the NPOC purge flow	
Fig. 52	FAST connector, angled	89
Fig. 53	Replacing the Fingertight connection	90
Fig. 54	Replacing the water traps on the front side	102
Fig. 55	Replacing the water traps on the gas box	103
Fig. 56	Replacing the halogen trap	105
Fig. 57	Replacing the adsorber cartridge	107
Fig. 58	Securing the autosampler for transport	120